FLUTRIAFOL (248)

First draft prepared by Mr M Irie, Ministry of Agriculture, Forestry and Fisheries, Tokyo, Japan

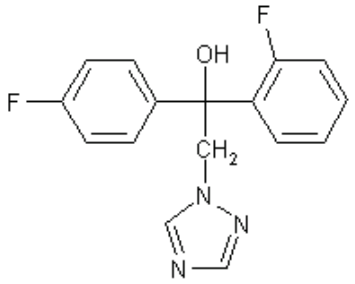
EXPLANATION

Residue and analytical aspects of flutriafol were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2011 JMPR by the Forty-second Session of the CCPR.

Flutriafol is a triazole fungicide used in many crops for control of a broad spectrum of leaf and ear cereal diseases, particularly embryo borne diseases e.g., bunts and smuts. The Meeting received information on identity, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fates of residues in processing.

IDENTITY

Common name Flutriafol
Chemical name
IUPAC: (*RS*)-2,4′-difluoro-α-(1*H*-1,2,4-triazol-1-ylmethyl)benzhydryl alcohol
CAS: (±)-α-(2-fluorophenyl)-α-(4-fluorophenyl)-1*H*-1,2,4-triazole-1-ethanol
CAS Registry No: 76674–21–0
CIPAC No: 436
Synonyms: PP450
Structural formula:



Molecular formula: $C_{16}H_{13}F_2N_3O$
Molecular weight: 301.3

Physical and chemical properties

Pure active ingredient

Property	Results	Reference
Appearance	White solid	Comb, 2006 (1358 FLU)
Melting point	130.5°C (99.0% purity)	Comb, 2006 (1358 FLU)
Relative density	1.17×10^3 kg/m^3 at 19.8 °C (99.0% purity)	Tognucci, 2003 (1097 FLU)
Vapour pressure	$< 0.01 \times 10^{-3}$ Pa at 25 °C (95.1% purity)	Piasentini de Campos, 2007 (1513 FLU)
Volatility (Henry's law constant)	No data submitted	
Solubility in water	95 mg/L in distilled water at 20 °C (99.0% purity)	Tognucci, 2004 (1103 FLU)
Partition coefficient	Log Pow = 2.32 ± 0.004 at 20 °C (95.1% purity)	Piasentini de

Property	Results	Reference
n-octanol/water		Campos, 2007 (1511 FLU)
Hydrolysis	Flutriafol was stable to hydrolysis at pH5, 7 and 9 at 50 °C over 30 days.	Snow and Cavell, 1982 (212 FLU)
	Flutriafol was hydrolytically stable in buffered solution at pH5, 7, and 9 at 25 °C up to 30 days.	Hawkins, Elsom and Jackson, 1987 (213 FLU)
Photolysis	Flutriafol was photolytically stable in aqueous buffer solution (pH7) at 25 °C for periods up to the equivalent of 66 days Florida summer sunlight.	Skidmore, 1987 (214 FLU)
	Photolysis of flutriafol by the direct absorption of sunlight (in the absence of photosensitisers) is not a significant degradation process in the environment.	Moffatt, 1994 (215 FLU)
Dissociation constant	No data submitted	

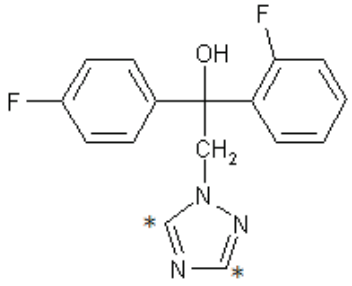
Technical material

Property	Results	Reference
Minimum concentration	93.6%	
Appearance	Fine white powder	Kusk, 2006 (1359 FLU)
Odour	odourless at room temperature (wet paste: 77.2% purity)	Søndergaar, 2006 (1367 FLU)
Solubility in organic solvents	Acetone: 114 - 133 g/L at 21 °C Ethyl acetate: 29 - 33 g/L at 21 °C n-heptane: < 10 g/L at 21 °C Xylene: < 10 g/L at 21 °C 1,2-dichloroethane: 20 - 25 g/L at 21 °C Methanol: 114 -133 g/L at 21 °C	Tognucci, 2004 (1104 FLU)

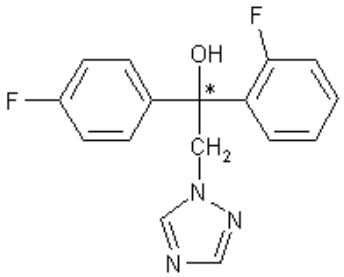
Formulations: Suspension concentrate (SC) 250 g ai/L or 125 g ai/L

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of flutriafol has been investigated in animals and plants. The fate and behaviour of flutriafol in animals, plants and the environment was investigated using the [^{14}C] labelled test materials shown in Figures 1.



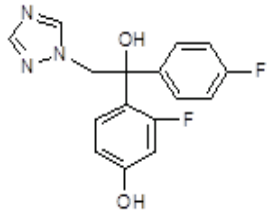
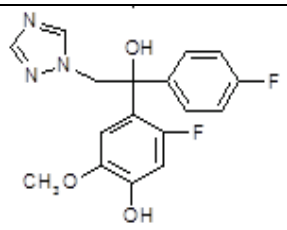
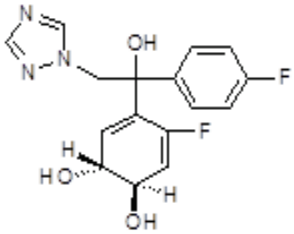
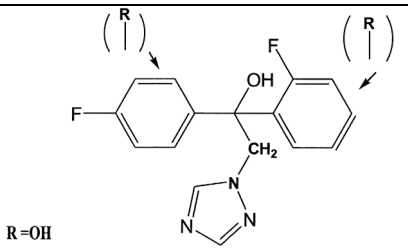
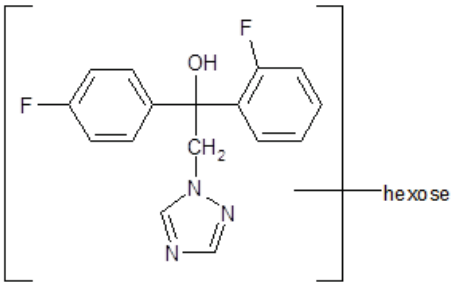
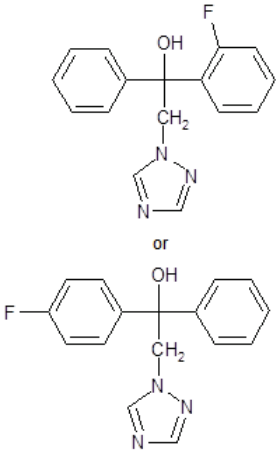
[^{14}C-triazole]flutriafol

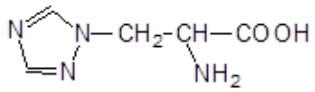
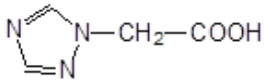
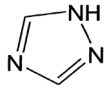
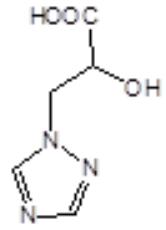
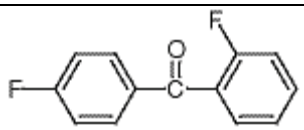


[^{14}C-carbinol]flutriafol

Figure 1 [^{14}C]-Labelled test materials used in animals, plants metabolism studies, and the environmental fate studies

The chemical structures of the major degradation compounds from the metabolism of flutriafol are provided below.

Compound name		Structure	Found in metabolism studies
M1B	4-Hydroxyflutriafol		Livestocks, Rats
M1D	4-Hydroxy-5-methoxyflutriafol		Livestocks, Rats
M2B	Flutriafol-(trans)-dihydrodiol		Livestocks, Rats
M5	Mixture of two isomeric hydroxyflutriafol derivatives		Livestocks
R5a	Hexose conjugated flutriafol		Plants
C6	Defluorinated flutriafol		Plants

Compound name		Structure	Found in metabolism studies
Triazole alanine	1,2,4-triazoyl-3-alanine		Plants
Triazole acetic acid	1*H*-1,2,4-triazol-1-ylacetic acid		Plants
1,2,4-Triazole	1*H*-1,2,4-triazole		Livestocks, Soil
	Triazole lactic acid		Plants (Rotational crops)
	2,4'-difluorobenzophenone		Soil

Animal metabolism

The Meeting received studies on the metabolism of flutriafol in rats, lactating cows and laying hens. The study on rats was evaluated by the WHO Core Assessment Group of the 2011 JMPR. A summary of the rat metabolism is given in this section.

Rats

In rat, flutriafol was extensively absorbed following single oral administration of 5 or 250 mg/kg dose. Bile was shown to be the major route of elimination of administered radioactivity excreted via this route over three days. Excretion of the dose was rapid at both dose levels and urinary excretion was the major route, accounting for 50–68% daily dose in the repeat dose animals and 61–68% for the single dose animals. The remaining radioactivity was excreted in the faeces (29–55%) with less than 6% dose remaining in the tissues 168 hours after the final repeat dose was administered and less than.8% 168 hours after the single dose. Only a minor amount of cleavage of the triazole moiety from the flutriafol molecule occurred. Furthermore, no metabolism was detected in the triazole group or in the 4-fluorophenyl ring of molecule. All identified metabolites were shown to be hydroxylated derivatives of the 2-fluorophenyl ring. Three of the major urinary metabolites were identified as a 3,4-(cis)- and the two M2B isomers of flutriafol. The bulk of remainder of urinary radioactivity was attributable to glucuronide conjugates, the two main aglycones of which were identified as M1B and M1D.

Lactating cow

The metabolism of flutriafol in the lactating cow has been studied using flutriafol labelled with ^{14}C in the triazole positions (Figure 1) (Hignett, 1985: 85 FLU). After a 7 days settling in period, the cow was dosed twice daily, for a period of 7 days, with gelatine capsules containing ^{14}C-triazole flutriafol absorbed onto powdered maize. Capsules were introduced directly into the stomach via a stomach tube. The cow received mean daily doses of 40 mg of flutriafol per day, equivalent to a daily intake of total diet (20 kg) containing flutriafol at a level of approximately 2 ppm in the diet.

Urine and faeces were collected, starting two days before dosing, every 12 hours until the last 36 hours, then four hourly. Milk samples were collected twice daily starting two days before dosing. The cow was sacrificed approximately four hours after administration of the last dose, the following tissues were collected: subcutaneous, omental and perirenal fat, muscle, liver, kidneys and heart. Radioactivity in these compartments as well as in faeces and urine was measured by liquid scintillation counting (LSC) after combustion to determine the total radioactive residues (TRR).

In order to characterise and identify the radioactive residue components, samples of urine, milk, liver and kidney were subjected to extensive extractions and further treatment. Characterisation of radioactive residues covered the fractionation into organosoluble and watersoluble/polar components. Solvent extraction was conducted using water, acetonitrile, methanol, as well as ether, acetone, dichloromethane and hexane. The polar fraction of urine, milk, liver and kidney as well as the unextractable fraction of liver was subjected to enzyme and acid/base hydrolysis. The main method for the identification of metabolites was thin layer chromatography (TLC). The metabolites were characterised by co-chromatography with reference substances in two dissimilar solvent systems. Extracts were analysed using HPLC with subsequent LSC quantification and gas chromatography/mass spectrometry (GC/MS) to determine the metabolic profile by comparison with reference substances.

Most of the administered radioactivity was excreted in the urine and faeces. The total quantity of radioactivity excreted in the urine and faeces was 45% and 33% of the dose, respectively. Radioactive residues in muscle and fat (subcutaneous, omental and perirenal) were insignificant (< 0.01 mg/kg equivalent to flutriafol). The liver, kidney and heart contained residues of 0.291, 0.061 and 0.011 mg/kg equivalent to flutriafol. Table 1 summarizes the results from administration of radiolabelled compound.

Table 1 Excretion and retention of radioactivity by cow after oral administration of ^{14}C-triazole labeled flutriafol at a nominal dietary administration of 40 mg for 7 days

Sample	% of administered dose	TRR (mg flutriafol equivalent/kg)
Urine	45.1	
Faeces	33.4	
Milk	0.1	
Muscle		0.008
Liver		0.291
Kidney		0.061
Heart		0.011
Fat (subcutaneous)		0.002
Fat (omental)		< 0.001
Fat (perirenal)		0.003

A total of 0.1% of the radioactivity administered to the cow was collected in the milk over the 7 day dosing period. The radioactive residue in the milk gradually increased to 0.007 mg/L (flutriafol equivalent) by Day 4 of the study, and maintained this level until the end of the study. The concentration of radioactive residues in milk is summarized in Table 2.

Table 2 Concentration of total radioactive residues (TRR) in milk (12 hours sampling) and amount of milk collected during administration of ^{14}C-flutriafol (40 mg/day)

Time (days)		TRR, mg flutriafol equivalents/L	Volume, L
1	p.m.	-	8.52
	a.m.	0.002	
2	p.m.	0.004	10.13
	a.m.	0.005	
3	p.m.	0.006	9.59
	a.m.	0.006	
4	p.m.	0.007	9.31
	a.m.	0.007	
5	p.m.	0.007	9.93
	a.m.	0.007	

Time (days)		TRR, mg flutriafol equivalents/L	Volume, L
6	p.m.	0.008	9.88
	a.m.	0.007	
7	p.m.	0.008	10.90
	a.m.	0.007	

p.m.: sample of evening milking; a.m.: sample of morning milking

In the urine, no parent flutriafol was observed by TLC analysis. Most of the radioactivity was associated with polar materials, although traces of 4-hydroxyflutriafol (M1B) and flutriafol-(trans)-dihydrodiol (M2B) were seen. Hydrolysis of the polar metabolites with enzyme and acid liberated several organosoluble compounds, including M1B and another compound with very similar chromatographic properties (Compound Y).

A small amount of parent flutriafol and M1B were contained in the ether fraction of the milk. No compound in this fraction accounted for more than 6% of TRR in the milk. Most of the radioactivity in the milk (75%) was associated with polar, water soluble metabolites which were converted to oraganosoluble fractions by extensive hydrolysis conditions. One radioactive compound was present, which cochromatographed with M1B.

Half of the radioactivity extracted from the liver was organosoluble. Parent flutriafol (27%) dominated this fraction. The more polar, water soluble extracted radioactivity was hydrolysed with enzyme, acid and alkali. These combined procedures liberated an organosoluble fraction which contained flutriafol (2%), 4-hydroxy-5-methoxyflutriafol (M1D, 2%), M1B (1%) and at least two other minor radioactive compounds. Most of the radioactivity from a non-extracted fraction was solubilised enzymatically. Two compounds, flutriafol and M1D, were identified and no individual compound accounted for more than 10% of TRR in the liver.

Most of the radioactivity (81%) found in the kidneys was extracted with the aqueous acetonitrile. Only a small amount of the radioactivity in the aqueous acetonitrile extract was partitioned into dichloromethane. This organosoluble fraction contained a mixture of at least six minor radioactive compounds, including flutriafol (1%) and M1B (<1%). Water soluble radioactivity accounted for 66% of the radioactive residue. Hydrolysis with a sequence of enzyme, acid and base rendered most of this fraction organosoluble (38%). M1B (22%) dominated this fraction, which also contained flutriafol (6%).

Table 3 Flutriafol and related residues identified in urine, milk and tissues

Compound	Urine	Milk	Liver	Kidney
	% of sample TRR			
Flutriafol*	-	1	29	7
4-Hydroxyflutriafol* (M1B)	23	3	1	23
4-Hydroxy-5-methoxyflutriafol* (M1D)	-	-	2	-
Compound Y	7	-	-	-
Flutriafol-(trans)-dihydrodiol (M2B)	traces	-	-	-

* including conjugates

This study shows that the residue in ruminants resulting from the use of flutriafol will be low. Traces of metabolite shown to be present are the same compounds identified in rat metabolism studies. The proposed metabolic pathway is shown in Figure 2.

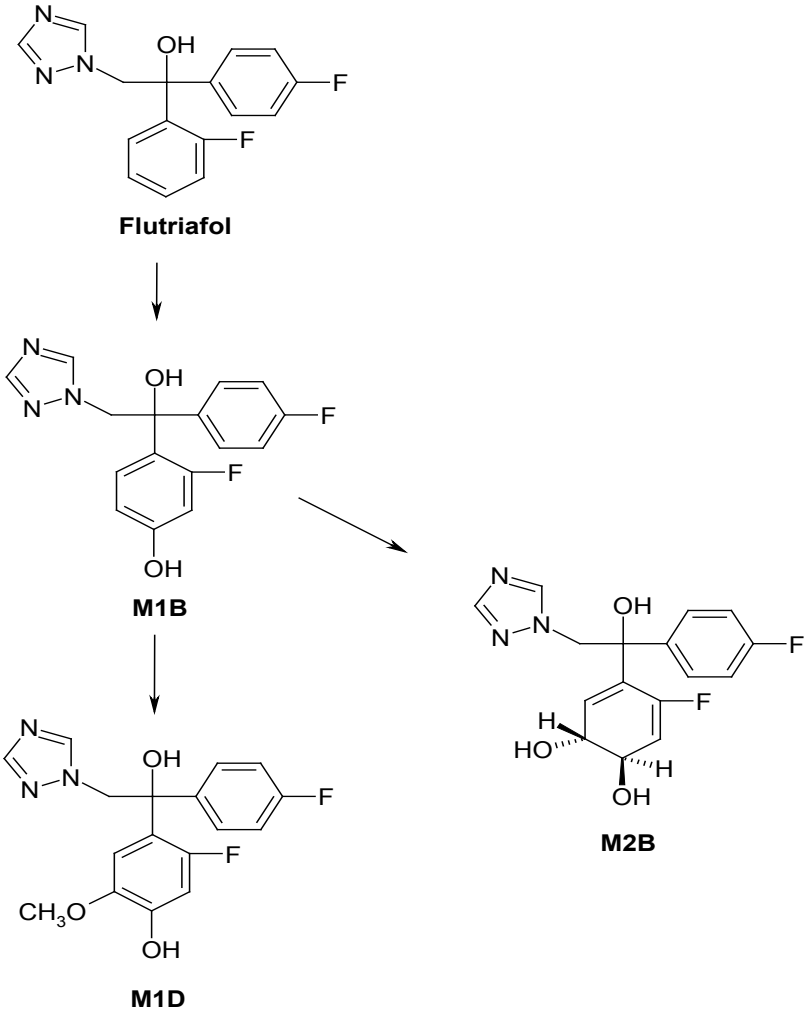


Figure 2 Proposed Metabolic Pathway of Flutriafol in Lactating Cow

Laying hen

The nature of the residue in laying hens (*Gallus gallus*) was conducted with two radiolabeled forms of flutriafol (^{14}C-triazole-flutriafol and ^{14}C-carbinol-flutriafol) (Dohn, 2006: 1464 FLU). Each radiolabelled test material was administered orally in cellulose-filled gelatin capsules to single comb brown laying hens once daily for 7 consecutive days. The average dietary dose was equivalent to 13.9 ppm for the [^{14}C-triazole]-flutriafol, and 11.6 ppm for the [^{14}C-carbinol]-flutriafol. Excreta were collected once daily (in the morning, before dose administration) from the treated hens only. Eggs were collected twice daily (morning and evening) from treated and control hens. The hens were sacrificed approximately 24 hours after the last dose was administered. The following tissues were collected at necropsy from treated and control hens: muscle (composite of breast and thigh), abdominal fat, liver and gastrointestinal tracts.

Portions of each excreta sample and homogenized gastrointestinal tracts were subjected to combustion analysis. The radioactive residue in egg, liver, muscle and fat samples were measured by digestion of subsamples with tissue solubilizer. The ^{14}C contents of the samples were measured by liquid scintillation counting. The characterization of the residues in eggs, liver, muscle and fat was conducted by solvent extraction with acetonitrile/water, followed by HPLC and TLC analysis. The

identification of the metabolites was done with available reference standards, and also by purifying metabolites from excreta followed by identification by LC/MS analysis where possible.

Most of the administered doses (91.6% for triazole label and 92.6% for the carbinol label) were recovered in the excreta and gastrointestinal tracts at sacrifice. Each daily dose was almost completely excreted within 24 hours.

The total radioactive residue (TRR) values were the highest for liver (0.36–0.41 mg/kg), followed by muscle (0.01–0.06 mg/kg) and fat (0.02–0.04 mg/kg). The distribution of the radioactive residues is summarized in Table 4.

Table 4 Recovery of administered ^{14}C in excreta and gastrointestinal tracts and concentration of the TRR in tissues

Matrix	[^{14}C-triazol]flutriafol % Total administered ^{14}C	[^{14}C-carbinol]flutriafol % Total administered ^{14}C
Total excreta	89.7	91.2
Gastrointestinal tract	1.9	1.4
Total	91.6	92.6
Tissue matrix	[^{14}C-triazol]flutriafol, mg/kg	[^{14}C-carbinol]flutriafol, mg/kg
Liver	0.411	0.359
Muscle	0.064	0.011
Fat	0.035	0.016

The radioactive residues in egg appeared to reach plateau concentrations by the end of the study, and ranged from 0.134 mg/kg (carbinol label) to 0.204 mg/kg (triazole label). The samples selected were taken from the evening collection on Day 6 (these samples had the largest residue in both groups) and the last collection (morning of Day 8). The concentration of ^{14}C-flutriafol derived radioactivity in the egg samples are summarized in Table 5.

Table 5 Concentration of the TRR in eggs

Time point	[^{14}C-triazol]flutriafol, mg/kg	[^{14}C-carbinol]flutriafol, mg/kg
Day 1 (pre dose)	0.0005	0.0000
Day 1 pm	0.001	NE
Day 2 am	0.041	0.032
Day 2 pm	0.089	0.016
Day 3 am	0.088	0.051
Day 3 pm	0.135	NE
Day 4 am	0.129	0.079
Day 4 pm	NE	0.116
Day 5 am	0.145	0.101
Day 5 pm	0.184	NE
Day 6 am	0.167	0.117
Day 6 pm	0.206 [a] (0.205[b])	0.160 [a] (0.159[b])
Day 7 am	0.190	0.126
Day 7 pm	0.204	0.121
Day 8 am (sacrifice)	0.184 [a] (0.204[b])	0.133 [a] (0.134[b])

NE: No eggs were produced.

[a] Values determined using tissue solubilizer

[b] Value determined by extraction + analysis of PES

Parent flutriafol was the most abundant component of the residue in all egg samples extracted (present at 0.088–0.119 mg/kg). Free 1,2,4-triazole was detected in the eggs (0.056–0.060 mg/kg). In addition, a mixture of two isomeric hydroxyflutriafol derivatives (referred to as M5) was present in all egg samples examined (0.009–0.010 mg/kg). The most abundant compound was 1,2,4-triazole (0.048 mg/kg) in muscle treated with [^{14}C-triazole] flutriafol. Other components in the muscle were present at < 0.01 mg/kg. The primary component of the fat residue was flutriafol (0.012–0.028 mg/kg, 75–80% of TRR). Free 1,2,4-triazole was detected in fat (0.004 mg/kg). The distribution of ^{14}C residue components in egg and tissue samples is presented in Table 6.

Table 6 Distribution of metabolites in egg and tissue sample

Component	Egg, Day 6 PM		Egg, Day 8		Muscle		Fat		Liver	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
[^{14}C-triazole]flutriafol										
Flutriafol[a]	0.099	48.3	0.103	50.5	0.000	0.0	0.028	80.0	0.013	3.2
M3[a]	0.018	8.8	0.023	11.3	0.006	9.4	0.001	2.9	0.027	6.6
M4[a]	0.003	1.5	0.006	2.9	0.000	0.0	0.000	0.0	0.024	5.8
M5[ab]	0.009	4.4	0.009	4.4	0.001	1.6	0.001	2.9	0.006	1.5
1,2,4-Triazole[a]	0.060	29.3	0.056	27.5	0.048	75.0	0.004	11.4	0.057	13.9
Others[a]	0.011	5.4	0.001	0.5	0.005	7.8	0.000	0.0	0.043	10.5
PES[c]	0.005	2.4	0.006	2.9	0.004	6.3	0.001	2.9	0.241	58.6
Total (TRR)	0.205	100.1	0.204	100.0	0.064	100.1	0.035	100.1	0.411	100.0
[^{14}C-carbinol]flutriafol										
Flutriafol[a]	0.119	74.8	0.088	65.7	0.000	0.0	0.012	75.0	0.007	1.9
M3[a]	0.021	13.2	0.017	12.7	0.005	45.5	0.001	6.3	0.025	7.0
M4[a]	0.005	3.1	0.005	3.7	0.000	0.0	0.000	0.0	0.025	7.0
M5[ab]	0.009	5.7	0.010	7.5	0.001	9.1	0.000	0.0	0.007	1.9
1,2,4-Triazole[a]	0.000	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000	0.0
Others[a]	0.001	0.6	0.006	4.5	0.001	9.1	0.002	12.5	0.055	15.3
PES[c]	0.004	2.5	0.008	6.0	0.004	36.4	0.001	6.3	0.240	66.9
Total (TRR)	0.159	99.9	0.134	100.1	0.011	100.1	0.134	100.1	0.359	100.0

[a] Components extracted with acetonitrile/water
[b] M5 is a mixture of two isomeric hydroxyflutriafol derivatives
[c] Post extracted solids

Extraction of the liver samples released 33.1 to 41.4% of the TRR. Flutriafol was detected in liver at low concentrations (0.007–0.013 mg/kg, 1.9–3.2% TRR). Free 1,2,4-triazole was present in liver at 0.057 mg/kg (13.9% TRR), and the mixture of hydroxyflutriafols (M5) was present at 0.006 to 0.007 mg/kg. A significant amount of the residue (58.6–66.9% TRR) was not extracted with acetonitrile/water. The residue remaining in the post-acetonitrile/water –extracted solids was subjected to a variety of enzymatic and chemical treatments, which resulted in the gradual release of most of the residue. Sequential treatments with two proteases (pepsin and pancreatin) released the largest portion of the residue (21.2–25.3% of TRR). However, the radioactivity released was complex in nature when examined by HPLC and TLC.

Table 7 Characterisation of radioactive residues in PES fraction of liver

Component	[^{14}C-triazole]flutriafol		[^{14}C-carbinol]flutriafol	
	mg/kg	%TRR	mg/kg	%TRR
Subtotal in primary extract[a]	0.170	41.4	0.119	33.1
Solubilised by enzyme treatment[b,c]	0.104	25.3	0.076	21.2
Solubilized by 1N HCl (ambient temp. x24h) [b]	0.017	4.1	0.021	5.8
Solubilized by 1N NH_4OH (ambient temp. x22h) [b]	0.036	8.8	0.029	8.1
Solubilized by 6N HCl (reflux. x19h) dichloromethane soluble [b]	0.003	0.7	0.019	5.3
Solubilized by 6N HCl (reflux. x19h) aqueous soluble [b]	0.046	11.2	0.090	25.1
Remaining (by difference) [b]	0.035	8.5	0.005	1.4
Sub total PES[cb]	0.241	58.6	0.240	66.9
Total (TRR)	0.411	100.0	0.359	100.0

[a] Components extracted with acetonitrile/water.
[b] Associated with solids remaining after extraction with acetonitrile/water
[c] Solubilised with successive treatments with pepsin and pancreatin + bile salts

The major metabolic processes were formation of two hydroxyl flutriafol derivatives by hydroxylation, and formation of free 1,2,4-triazole. Based on the findings, the proposed metabolic pathway of flutriafol in hens is shown in Figure 3.

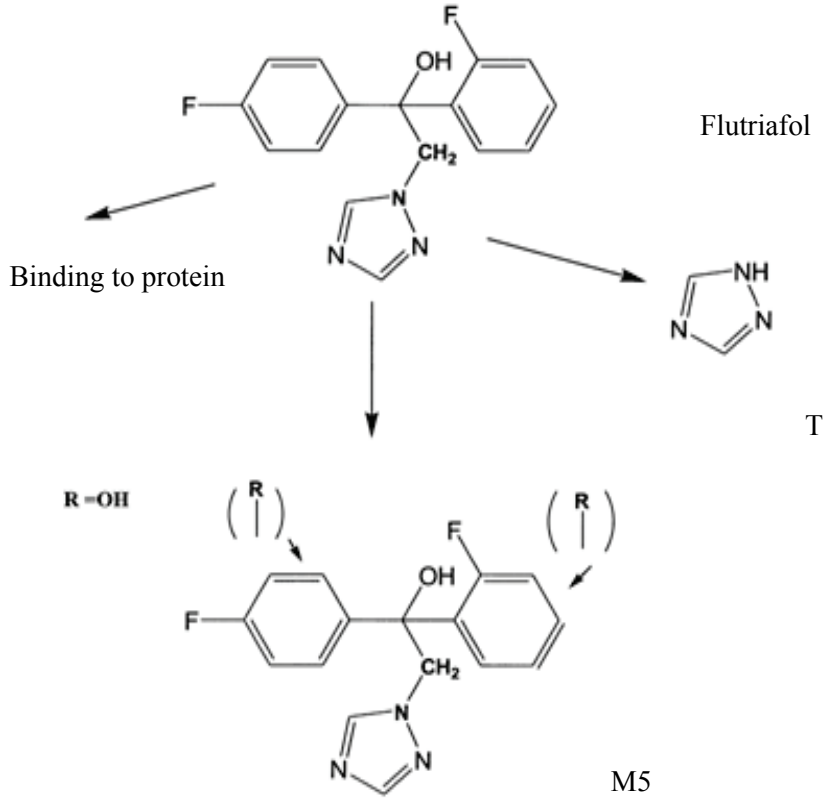


Figure 3 Proposed Metabolic Pathway of Flutriafol in Laying Hens

Summary of animal metabolism

Metabolism of ^{14}C labelled flutriafol has been studied in lactating cow and laying hens. In both studies, flutriafol was metabolized to several metabolites. All metabolites detected in the metabolism of lactating cow were also found in the metabolism of rats. The major metabolic processes in laying hens were the binding of flutriafol to liver proteins, the formation of hydroxyl flutriafol derivatives (fluorophenyl moiety), and the formation of free 1,2,4-triazole.

Plant metabolism

Plant metabolism studies were performed on apples, cereals (wheat and barley), oilseed rape and sugar beet using the triazole- and carbinol-^{14}C-labeled flutriafol.

Apples

The metabolism of flutriafol was investigated in apples (variety Gala) following a single topical application of two radiolabelled forms [^{14}C-triazole]-flutriafol and [^{14}C-carbinol]-flutriafol (Figure 1) at a rate of 0.118 kg/ha and volume of 200 L/ha (Brice, 2007: 1625 FLU). The application rate was equivalent to 1 × the single application rate for apples. The test substance, as a 12.5% active ingredient suspension concentrate (SC), was applied at early fruit development (BBCH growth stage 74). Fruit and foliage samples were collected at a typical harvest time for the variety (BBCH growth stage 87), 64 days after application. The apple fruits were analysed for flutriafol and metabolites. In view of the very low levels of unknowns in the apple extracts, it was subsequently decided to analyse the apple foliage from trees treated with [^{14}C-triazole]-flutriafol as an aid to identification of metabolites.

A representative portion of each fruit sample and foliage ([^{14}C-triazole]-flutriafol only) sample was homogenised to a powder in dry ice using a blender. Samples of the fruit and foliage homogenate were combusted in an oxidiser to ^{14}C in order to provide an initial TRR value for each sample. A representative sub-sample of each homogenate (200 g for fruit and 100 g for foliage) was then extracted sequentially by maceration with acetonitrile (3 × 100 mL) and acetonitrile/water (1:1, v/v, 2 × 100 mL). The fruit samples were further extracted with 0.1 M sodium hydroxide (100 mL) and 0.1 M hydrochloric acid (100 mL). Following acetonitrile and acetonitrile/water extraction, a portion of each fruit and foliage residue was air dried and aliquots were analysed by LSC, following combustion. Following extraction with basic and acid, the remaining unextracted fruit residue was determined by calculation. The acetonitrile and acetonitrile/water extracts were combined for chromatographic analysis. Work-up resulted in organic and aqueous fractions. Each fraction was analysed separately by HPLC and/or TLC.

The TRR in apple fruits and foliage at mature harvest following a single topical application of [^{14}C-triazole]-flutriafol or of [^{14}C-carbinol]-flutriafol and percent distribution of the radioactive residues in the various extracts are summarised in Table 8. TRR values from the two radiolabelled forms of the test substance were similar with most of the radioactivity (72–78% TRR, equivalent to 0.030–0.051 mg/kg) extracted using acetonitrile or acetonitrile/water (1:1, v/v). Basic and acidic extraction released < 3% TRR (≤ 0.002 mg/kg) and this low residue was not examined further. Unextracted radioactivity comprised 18 to 23% TRR (0.009–0.012 mg/kg).

Table 8 Recovery of Total Radioactive Residues in apple fruit and foliage

Solvent	[^{14}C-triazole]flutriafol		[^{14}C-carbinol]flutriafol	
	TRR (%)	mg/kg	TRR (%)	mg/kg
Fruit				
Acetonitrile	70.3	0.046	67.8	0.028
Acetonitrile: water (1:1 v/v)	7.4	0.005	4.1	0.002
0.1M NaOH	2.4	0.002	2.6	0.001
0.1M HCl	2.1	0.001	2.4	0.001
Unextracted residue	17.8	0.012	23.0	0.009
Total	100	0.065	100	0.041
Foliage				
Acetonitrile: water (1:1 v/v)	83.6	3.497	-	-
Unextracted residue	16.4	0.685	-	-
Total	100	4.182	-	-

TRR% value for each fruit sample derived by direct combustion of the homogenate was 105% and 93% of the values for the triazole and carbinol treated samples respectively

Components of the TRR from apple fruits were characterised or identified following work-up and chromatographic analysis of relevant extracts. Flutriafol comprised 50 to 56% TRR (0.023–0.032 mg/kg). The total unknowns comprised 22% TRR (0.013 mg/kg) in the triazole labelled sample and 9% TRR (0.003 mg/kg) in the carbinol labelled sample. The largest unknowns were comprised only 5% and 3% TRR (0.003 and 0.001 mg/kg) respectively. UV detection of the reference standards was poor and it was only possible to confirm the retention time of flutriafol. It was not possible to confirm the presence of Triazole alanine and Triazole acetic acid in extracts using co-chromatography. The response in the region for Triazole alanine suggested that Triazole alanine was formed as a metabolite in apple crops at trace levels (< 0.001 mg/kg). The TLC systems used for this study did not move Triazole acetic acid from the origin. It was not possible to confirm the presence of traces of Triazole acetic acid. If present, the level of Triazole acetic acid would be < 0.0002 mg/kg (the radioactive residue retained at the origin).

In attempt to confirm the presence of trace levels of Triazole alanine and Triazole acetic acid in apple fruit, the foliage from the [^{14}C-triazole]-flutriafol application was analysed since this was expected to contain higher radioactive residues. The results showed that flutriafol comprised 48% TRR (2.00 mg/kg) and the remaining radioactivity comprised 29% TRR (1.21 mg/kg). TLC analysis showed that traces of Triazole alanine were present at low levels in the foliage extract (0.13 mg/kg) and comparison of chromatograms from fruit and foliage confirmed that Triazole alanine was present

in the fruit extracts at trace levels. However, as with the fruit extracts, the presence of trace levels of Triazole acetic acid could not be confirmed. Table 9 summarizes distribution of radioactive components in fruits at harvest.

Table 9 Characterisation of total radioactive residues in apple fruits

Component	Organic fraction		Aqueous fraction		Total	
	TRR (%)	mg/kg	TRR (%)	mg/kg	TRR (%)	mg/kg
[^{14}C-triazole]-flutriafol						
Flutriafol	48.2	0.031	1.7	0.001	49.9	0.032
Largest unknown	0.4	< 0.001	4.2	0.003	4.6	0.003
Total unknown	6.7	0.004	14.9	0.009	21.6	0.013
[^{14}C-carbinol]-flutriafol						
Flutriafol	51.9	0.021	4.2	0.002	56.2	0.023
Largest unknown	2.2	0.001	0.5	< 0.001	2.8	0.001
Total unknown	5.9	0.003	2.9	< 0.001	8.8	0.003

The radioactive residue was predominantly flutriafol. TLC data suggested some conversion to Triazole alanine. The Triazole alanine metabolite is known to oxidise to Triazole acetic acid in plants, however it did not appear that Triazole acetic acid was present in apple extracts. The free 1,2,4-triazole metabolite was not detected in the fruit or foliage samples.

Sugar beet

The metabolism of flutriafol was studied in sugar beet using [^{14}C-triazole] and [^{14}C-carbinol] flutriafol (Figure 1) (Aikens, 2003: 1080 FLU). Flutriafol radiolabelled was prepared as 125 g/L SC formulations and sprayed at a nominal rate of 125 g ai/ha onto sugar beet grown in containers outdoors. Samples of foliage and beet (root) were taken and analysed at the following three times: within three hours of application (0 days after treatment, DAT), five days pre-harvest (16 DAT) and at harvest (21 DAT). Additionally samples of foliage and beet were taken 6 days after treatment (6 DAT sample) and 11 days after treatment (11 DAT sample) these samples were not analysed. The total radioactive residue (TRR) was measured in the separated beet root and remaining foliage in the plants samples immediately after application, at the intermediate sampling and harvest, and the nature of the radioactive residue in foliage samples was investigated.

The plants were removed from the soil and the soil crown and foliage removed, at the soil level, from the beet root. Adhered soil was washed from the beet root. Beet root and foliage samples were separately homogenised, with dry ice, using a commercial Waring blender. Portions (at least three) of homogenised samples were taken for measurement of the TRR by combustion/LSC. Foliage samples only were extracted. A subsample (10 g of the foliage sample) of the homogenised sample was extracted with acetonitrile (three times) and then with acetonitrile/water (1:1, v/v, two times). The extract solutions were analysed by HPLC and TLC. Final harvest foliage samples were subsequently extracted using mild acidic and basic solvent mixtures.

The TRRs in foliage samples taken just after application were 1.37 and 1.27 mg/kg for the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. No significant residue was observed in the root. In the 16 DAT sample, the TRRs were 0.342 and 0.381 mg/kg in the foliage for [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. The TRR of the root from the 16 DAT samples was $\underline{< 0.0}$05 mg/kg in both labelled forms. At harvest, the TRRs were 0.747 and 0.596 mg/kg in the foliage, and 0.009 and 0.005 mg/kg in the remaining root for [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. As the TRR in all root samples were < 0.01 mg/kg, these samples were not analysed further.

Virtually all (93.9–97.1%, 1.20–1.33 mg/kg) of the residues in the post application samples were extracted by neutral solvents. Similar amounts of the TRR were extracted by these solvents in the foliage taken at intermediate 3 (88.8–91.1%, 0.312–0.338 mg/kg) and at harvest (91.3–92.9%, 0.544–0.694 mg/kg). Further residues were extracted from the harvest samples with mild acid and basic solvent mixtures (2.9–3.8%, 0.022–0.023 mg/kg). In the final harvest samples with

unextractable radioactivity in the foliage represented < 5% TRR (≤ 0.03 mg/kg). The results are summarized in Table 10.

Table 10 Total radioactive residues and extractability (incl. partition of extracts) of sugar beet following application of ^{14}C-Flutriafol

Plant portion	Foliage				Root			
Radiolabel	[^{14}C-carbinol]		[^{14}C-triazole]		[^{14}C-carbinol]		[^{14}C-triazole]	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Sampling event	Application							
Days after treatment (DAT)	0							
Extract:								
Solvent : water	1.195	93.9	1.328	97.1	na	na	na	na
0.1 M NaOH/0.1 M HCl	na	na	na	na	na	na	na	na
Total extracts	1.195	93.9	1.328	97.1	na	na	na	na
Non-extracted residue	0.078	6.1	0.040	2.9	na	na	na	na
TRR	1.273	100	1.368	100	< 0.001	100	0.001	100
Sampling event	Intermediate 3							
Days after treatment (DAT)	16							
Extract:								
Solvent : water	0.338	88.8	0.312	91.1	na	na	na	na
0.1 M NaOH/0.1 M HCl	na	na	na	na	na	na	na	na
Total extracts	0.338	88.8	0.312	91.1	na	na	na	na
Non-extracted residue	0.043	11.2	0.030	8.9	na	na	na	na
TRR	0.381	100	0.342	100	0.005	100	0.003	100
Sampling event	Harvest							
Days after treatment (DAT)	21							
Extract:								
Solvent : water	0.544	91.3	0.694	92.9	na	na	na	na
0.1 M NaOH/0.1 M HCl	0.023	3.8	0.022	2.9	na	na	na	na
Total extracts	0.567	95.1	0.716	95.8	na	na	na	na
Non-extracted residue	0.029	4.8	0.031	4.2	na	na	na	na
TRR	0.596	99.9	0.747	100	0.005	100	0.009	100

mg/kg: mg flutriafol equivalents/kg

na: not analysed

Flutriafol was the major residue in foliage samples, accounting for 69.2 and 70.8% TRR (0.412 and 0.519 mg/kg) at harvest, no other radioactive metabolite at harvest represented more than 5.4 and 5.0% TRR (0.033 and 0.038 mg/kg). All of the chromatographic data indicated that there had been no cleavage of the flutriafol molecule that resulted in separation of the [^{14}C-carbinol] and [^{14}C-triazole] radiolabel positions. This conclusion was supported indirectly by the general agreement (in terms of %TRR), between [^{14}C-carbinol] and [^{14}C-triazole] samples, in the pattern of extractability of the radioactive residues and the proportions of the individual metabolites themselves. One metabolite of flutriafol in foliage extracts of sugar beet was identified as a hexose conjugate of flutriafol. This component represented a maximum of 0.026 mg/kg, 4.4% TRR at harvest (21 DAT). Table 11 summarises the proportion of radioactive components in .foliage of sugar beet.

Table 11 The proportions of radioactivity in sugar beet foliage extract samples following application of ^{14}C-Flutriafol

Component	Application day (0 DAT)				Intermediate 3 (16 DAT)				Final harvest (21 DAT)			
	[^{14}C-carbinol]		[^{14}C-triazole]		[^{14}C-carbinol]		[^{14}C-triazole]		[^{14}C-carbinol]		[^{14}C-triazole]	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Flutriafol	1.165	91.6	1.304	95.4	0.260	68.2	0.266	77.7	0.412	69.2	0.529	70.8
R1	0.002	0.2	0.003	0.2	0.020	5.3	0.028	8.1	0.016	2.6	0.023	3.1
R2	< 0.001	0.0	< 0.001	0.0	0.005	1.4	0.002	0.7	0.027	4.5	0.034	4.6
R3	< 0.001	0.0	< 0.001	0.0	0.005	1.4	0.003	0.9	0.014	2.5	0.016	2.1
R4	< 0.001	0.0	0.001	0.1	0.010	2.7	0.002	0.5	0.033	5.4	0.033	4.4

Component	Application day (0 DAT)				Intermediate 3 (16 DAT)				Final harvest (21 DAT)			
	[^{14}C-carbinol]		[^{14}C-triazole]		[^{14}C-carbinol]		[^{14}C-triazole]		[^{14}C-carbinol]		[^{14}C-triazole]	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
R5a	0.001	0.1	< 0.001	0.0	0.017	4.4	0.004	1.1	0.023	3.8	0.038	5.0
R5b	0.008	0.7	0.012	0.9	0.013	3.3	0.009	2.6	0.019	3.1	0.018	2.4
R6	0.008	0.7	0.003	0.2	0.003	0.7	< 0.001	0.0	0.005	0.8	0.008	1.0
Others	0.010	0.8	0.005	0.4	0.005	1.4	< 0.001	0.0	0.019	3.1	0.018	2.4

Cereals

The metabolism of flutriafol has been studied in wheat and barley using [^{14}C-triazole] and [^{14}C-carbinol] flutriafol (Figure 1) (Cavell, 1982: 80 FLU). Formulated ^{14}C-labelled flutriafol was applied, by foliar spray, to wheat and barley plants growing in the greenhouse and in the field. The application rate was approximately 90 g/ha. In the greenhouse, plants were treated with ^{14}C-flutriafol just before ear emergence. In the field, ^{14}C-triazole-labelled flutriafol was applied to wheat just before ear emergence and to barley just after ear emergence. ^{14}C-carbinol-labelled flutriafol was applied just before ear emergence to barley and just after ear emergence to wheat.

Plant matrices (grain and straw) were homogenised and extracted with acetonitrile. The solid residues were soaked with water for two hours and then the same volume of acetonitrile was added centrifuged and decanted. The extraction step with acetonitrile/water (1:1, v/v) was repeated. The remaining solids were combusted and analysed by liquid scintillation counting (LSC). The acetonitrile/water extract was rotary evaporated and the remaining water was partitioned into ether. The ether phase was analysed by thin layer chromatography (TLC). The water phase was reduced by rotary evaporation and the residue submitted to cation exchange column treatment. The analytes were eluted sequentially with water, 1M HCl and 1M NH_4OH, respectively. The acidic and ammonia fraction were analysed by TLC and the water fraction was discarded. The acetonitrile fraction from the first extraction step was reduced by rotary evaporation, the residue re-dissolved in water and partitioned into ether. The ether phase was analysed by TLC.

The total radioactive residues of the grain and straw were found by combustion analysis. Results for greenhouse and field grown plants are summarised in Table 12.

Table 12 Total radioactive residue (mg/kg flutriafol equivalent) in cereal grain and straw after application of flutriafol at rates of 81–105 g ai/ha

Treatment	Crop	Growth stage at application	Component analysed	Application rate, g ai/ha	TRR, mg/kg
^{14}C-carbinol-flutriafol	Indoor barley	26 days before ear emergence	Grain	81	0.02
	Indoor wheat	4 days before ear emergence	Grain	81	0.01
	Outdoor barley	13 days before ear emergence	Grain	90	0.007
			Straw	90	0.72
	Outdoor wheat	after ear emergence	Grain	88.6	0.006
			Straw	88.6	0.53
^{14}C-triazolyl-flutriafol	Indoor barley	26 days before ear emergence	Grain	90	0.41
			Straw	90	2.1
	Indoor wheat	4 days before ear emergence	Grain	90	0.18
	Outdoor barley	after ear emergence	Grain	84.2	0.10
			Straw	84.2	0.12*
	Outdoor wheat	20 days before ear emergence	Grain	105	0.05
			Straw	105	0.65

* low residue level due to heavy rainfall soon after spraying

TRR = Total radioactive residues calculated as flutriafol

At maturity in the greenhouse, no residues of flutriafol (radioactive limit of detection 0.005 mg/kg) were detected in the grain. Following application of ^{14}C-triazole-labelled flutriafol, Triazole alanine accounted for approximately half the radioactive residue in the grain (0.08 and 0.04 mg/kg in barley and wheat respectively): Triazole acetic acid was also characterised as a

significant metabolite (0.04 and < 0.01 mg/kg in barley and wheat respectively). Total radioactive residues in barley and wheat grain following ^{14}C-carbinol-labelled flutriafol applications were considerably lower (0.02 and 0.01 mg/kg respectively). In barley straw, flutriafol was the major radioactive component, and accounted for 63% of the TRR (1.3 mg/kg).

At maturity in the field, the TRR in the wheat grain following ^{14}C-triazole-labelled flutriafol applications was 0.05 mg/kg and no flutriafol was detected (limit of detection 0.0002 mg/kg). Triazole alanine and Triazole acetic acid accounted for 58 and 26% of this residue respectively. The TRR in barley grain was 0.10 mg/kg. Flutriafol, Triazole alanine and Triazole acetic acid accounted for 24, 8 and 5% of this residue respectively. The TRRs in the grain in both plants following ^{14}C-carbinol-labelled flutriafol applications were less than 0.01 mg/kg. Radioactive residues in the straw ranged from 0.12 to 0.72 mg/kg. Flutriafol was the major component (57% in wheat straw from ^{14}C-triazole-labelled flutriafol treatment). The results are summarized in Table 13.

Table 13 Characterisation and distribution of the total radioactive residue in cereal grain and straw

Treatment	Crop	Component analysed	Flutriafol		Triazole alanine		Triazole acetic acid		Bound	Othersa
			%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	%TRR
^{14}C-carbinol-flutriafol	Indoor barley	Grain	na	na	na	na	na	na	na	na
	Indoor wheat	Grain	na	na	na	na	na	na	na	na
	Outdoor barley	Grain	36	0.002	nd	nd	nd	nd	26	38
		Straw	38	0.27	nd	nd	nd	nd	40	22
	Outdoor wheat	Grain	na	na	na	na	na	na	na	na
		Straw	na	na	na	na	na	na	na	na
^{14}C-triazolyl-flutriafol	Indoor barley	Grain	~1	-	40	0.08	26	0.04	7	21
		Straw	63	1.32	nd	nd	nd	nd	16	5
	Indoor wheat	Grain	nd	nd	48	0.04	8	0.006	5	34
	Outdoor barley	Grain	24	0.02	8	0.004	5	0.002	35	28
		Straw	na	na	na	na	na	na	na	na
	Outdoor wheat	Grain	nd	nd	58	0.015	26	0.005	5	11
		Straw	57	0.37	nd	nd	nd	nd	23	20

na : not analysed

nd : not detected

[a] including small percentage of uncharacterised components and losses during analysis

Radioactive residues in the straw were of a similar level for greenhouse and field-grown plants, whether ^{14}C-triazole-labelled flutriafol or ^{14}C-carbinol-labelled flutriafol was applied. Flutriafol was the dominant component of the radioactive residue. Triazole alanine and Triazole acetic acid were not detected in the straw. Some (16–40%) of the radioactive residue was not extractable.

Oilseed rape

The metabolism of flutriafol was investigated in oilseed rape using [^{14}C-triazole] and [^{14}C-carbinol] flutriafol (Figure 1) (Shaw, 2003: 1079 FLU). Flutriafol radiolabelled with carbon-14 was prepared as 125 g/L SC formulations and sprayed at a nominal rate of 125 g ai/ha onto oilseed rape grown in containers outdoors. The two radiolabelled forms of ^{14}C-flutriafol were applied to separate plants. The plants were at the early pod set growth stage (BBCH 71; it was approximately 10% of the potential pods had formed) at the time of application. Plants were sampled just after treatment (0 DAT), at three intermediate stages (7, 14 and 21 DAT) and at harvest (42 DAT). The TRR was measured and the nature of the radioactive residue was investigated in the whole plant taken just after application, in the separated pods and remaining plant at the intermediate sampling (14 DAT) and in the separated seeds and remaining plant at harvest.

Plants were sampled at the 1st and 3rd intermediate times (7 and 21 DAT) but not analysed. Plants were cut of 1–2 cm above the soil surface. Roots were not sampled. Plants were taken from all appropriate containers at each sampling event.

The TRR was measured by combustion/LSC in the whole plant taken just after application, in the separated pods and remaining plant from the intermediate sampling (14 DAT) and in the separated seeds and remaining plant at harvest. All sampled plants and plant parts were subjected to a series of solvent/water extractions, with acetonitrile, acetonitrile/water (1:1, v/v) and in some cases with water. Seeds taken at harvest (42 DAT) were crushed using a pestle and mortar and then extracted twice with hexane. Plants from the intermediate sampling (14 DAT) and harvest (42 DAT) were further subjected to a sequential treatment/extraction involving incubation with hemicellulase and cellulase enzymes, extraction with 0.1 M HCl or 0.1 M NaOH at ambient temperature, extraction with 6 M HCl or 2 M NaOH under reflux. The pooled solvent/water extracts of the harvest samples (42 DAT) (seeds and remaining plants) were split into three portions. The pH of one portion was adjusted to pH 2 using concentrated HCl. The pH of another portion was adjusted to pH12 using 10 M NaOH. The pH of the third portion was not adjusted. Each portion was partitioned twice with ethyl acetate.

Combined acetonitrile, acetonitrile/water and water extracts were analysed by HPLC using radio- and UV detector. TLC was also carried with several sample extracts using either a normal phase system with different developing phases or a reversed phase system with acetonitrile/water as developing phase. The further extract solutions (after solvent partition in some cases) were analysed by TLC and in some cases by HPLC.

The TRR in the forage samples (whole plant) taken just after treatment (0 DAT) were 0.782 and 1.50 mg/kg for the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. At the pod development stage (BBCH 79, 14 DAT), TRRs were 0.751 and 0.779 mg/kg in the pods, and 1.17 and 1.60 mg/kg in the remaining plant ([^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively). At harvest (42 DAT), TRRs were 1.32 and 0.729 mg/kg in the seeds, and 0.246 and 0.355 mg/kg in the remaining plant ([^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively).

Virtually all (97.9–98.3%, 0.769–1.47 mg/kg) of the residues at 0 DAT were extracted by acetonitrile and acetonitrile/water. Proportionately less of the TRR was extracted by these solvents in the remaining plants taken at 14 DAT (85.6–91.6%, 0.997–1.47 mg/kg) and at 42 DAT (60.4–76.2%, 0.187–0.214 mg/kg). In the pods taken at 14 DAT, 40.1–41.2% TRR (0.309–0.312 mg/kg) was extracted by acetonitrile and acetonitrile/water. In the seeds taken at 42 DAT, 27.1–31.5% TRR (0.198–0.415 mg/kg) was extracted by hexane and 42.1–42.5% TRR (0.307–0.559 mg/kg) by acetonitrile and acetonitrile/water.

Varying proportions of the TRRs were released by further incubation with enzymes and extraction with acid and base. In the pods taken at 14 DAT, 16.3–17.1% TRR (0.127–0128 mg/kg) was released by enzymes, 0.3–0.5% TRR (0.002–0.004 mg/kg) by 0.1 M HCl, 2.8–4.1% TRR (0.021–0.032 mg/kg) by 0.1 M NaOH, 16.9–18.5% TRR (0.127–0.144 mg/kg) by 6 M HCl and 13.4–15.8% TRR (0.104–0.119 mg/kg) by 2 M NaOH. The remaining non-extracted residues accounted for 5.8–7.1% TRR (0.044–0.055 mg/kg). In the remaining plant at this time, 3.3–5.0% TRR (0.053–0.058 mg/kg) was released by enzymes. Each of the further extractions released 0.2–2.8% TRR (0.002–0.033 mg/kg). The remaining non-extracted residues accounted for 1.4–2.9% TRR (0.022–0.034 mg/kg). In seeds taken at 42 DAT, enzymes, 0.1 M HCl and 0.1 M NaOH each released 0.3–3.0% TRR (0.002–0.039 mg/kg). Between 7.9 and 8.8% TRR (0.064–0.104 mg/kg) was released by 6 M HCl and 2.5–9.1% TRR (0.018–0.120 mg/kg) by 2 M NaOH. The remaining non-extracted residues accounted for 4.3–16.8% TRR (0.057–0.122 mg/kg). In the remaining plant at 42 DAT, enzymes, 0.1 M HCl and 0.1 M NaOH each released 0.4–4.0% TRR (0.001–0.014 mg/kg). Between 5.5 and 11.4% TRR (0.014–0.040 mg/kg) was released by 6 M HCl and 2.4–3.0% TRR (0.007–0.009 mg/kg) by 2 M NaOH. The remaining non-extracted residues accounted for 11.1–20.1% TRR (0.027–0.071 mg/kg). The results are summarized in Table 14.

Table 14 Total radioactive residues and extractability (incl. partition of extracts) of oilseed rape following application of ^{14}C-Flutriafol

Sampling event	Application		2nd intermediate				Harvest			
DAT	0		14				42			
Plant portion	Whole plant (forage)		Pods		Remaining plant		Seeds		Remaining plant	
Radiolabel	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]
	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Extract/fraction										
Hexane	ns	ns	ns	ns	ns	ns	0.198 (27.1)	0.415 (31.5)	ns	ns
Solvent:water	1.466 (97.9)	0.767 (98.3)	0.312 (40.1)	0.309 (41.2)	1.467 (91.6)	0.997 (85.6)	0.307 (42.1)	0.559 (42.5)	0.214 (60.4)	0.187 (76.2)
Enzyme	ns	ns	0.127 (16.3)	0.128 (17.1)	0.053 (3.3)	0.058 (5.0)	0.009 (1.3)	0.018 (1.4)	0.014 (4.0)	0.006 (2.6)
0.1 M HCl	ns	ns	0.004 (0.5)	0.002 (0.3)	0.003 (0.2)	0.002 (0.2)	0.002 (0.3)	0.005 (0.4)	0.002 (0.6)	0.001 (0.4)
0.1 M NaOH	ns	ns	0.032 (4.1)	0.021 (2.8)	0.006 (0.4)	0.007 (0.6)	0.009 (1.2)	0.039 (3.0)	0.004 (1.1)	0.003 (1.2)
Organic			0.029 (3.7)	0.018 (2.4)	np	np	0.006 (0.8)	0.025 (1.9)	np	np
Aqueous			0.003 (0.4)	0.003 (0.4)	np	np	0.003 (0.4)	0.014 (1.1)	np	np
6 M HCl	ns	ns	0.144 (18.5)	0.127 (16.9)	0.024 (1.5)	0.033 (2.8)	0.064 (8.8)	0.104 (7.9)	0.040 (11.4)	0.014 (5.5)
Organic			0.096 (12.3)	0.086 (11.5)	0.015 (0.9)	0.019 (1.6)				
Aqueous			0.048 (6.2)	0.041 (5.5)	0.009 (0.6)	0.014 (1.2)				
2 M NaOH	ns	ns	0.104 (13.4)	0.119 (15.8)	0.026 (1.6)	0.033 (2.8)	0.018 (2.5)	0.120 (9.1)	0.009 (2.4)	0.007 (3.0)
Organic			0.091 (11.7)	0.088 (11.7)	0.023 (1.4)	0.026 (2.2)	0.017 (2.3)	0.067 (5.1)	np	np
Aqueous			0.013 (1.7)	0.031 (4.1)	0.003 (0.2)	0.007 (0.6)	0.001 (0.1)	0.053 (4.0)	np	np
Total extracts	1.466 (97.9)	0.769 (98.3)	0.723 (92.9)	0.706 (94.1)	1.579 (98.6)	1.130 (97.0)	0.607 (83.3)	1.260 (95.8)	0.283 (79.9)	0.218 (88.9)
Non-extracted residue	0.031 (2.1)	0.013 (1.7)	0.055 (7.1)	0.044 (5.8)	0.022 (1.4)	0.034 (2.9)	0.122 (16.8)	0.057 (4.3)	0.071 (20.1)	0.027 (11.1)
TRR	1.497	0.782	0.779	0.751	1.601	1.165	0.729	1.316	0.355	0.246

ns: No sample

np: Not partitioned

Flutriafol was the major radioactive component in forage samples taken just after treatment. The parent test substance accounted for 96.9 and 96.0% TRR (0.758 and 1.437 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. All other radioactive residues extracted by acetonitrile and acetonitrile/water accounted for 1.4 and 1.9% TRR (0.011 and 0.029 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively).

In the pods at 14 DAT, flutriafol was the major radioactive component in the solvent/water extracts, accounting for 32.4 and 31.5% TRR (0.243 and 0.245 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. Other radioactive components in these extracts did not individually represent more than 1.6 and 2.6% TRR (0.012 and 0.020 mg/kg) in total accounted for 8.8 and 8.6% TRR (0.064 and 0.067 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively).

At 14 DAT, flutriafol was also the major radioactive component in the solvent/water extracts of the remaining plant samples, accounting for 76.1 and 81.8% TRR (0.886 and 1.31 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. Other prominent components were R5a and R5b. In plants treated with [^{14}C-triazole] flutriafol, they accounted for 2.7 and 2.6% TRR (0.031 and 0.030 mg/kg). In plants treated with [^{14}C-carbinol] flutriafol, they accounted for 2.7 and 3.1% TRR (0.043 and 0.050 mg/kg). The remaining radioactive components in these extracts did not individually represent more than 0.9 and 1.2% TRR (0.015 and 0.014 mg/kg). Further treatment and extraction of the plant samples following solvent/water extraction released

additional quantities of radioactive residues. The weak acid extracts were not partitioned or analysed as the radioactivity concentrations were all less than 0.01 mg/kg. Depending on the radioactivity concentrations, weak base, strong acid and base extracts were partitioned with organic solvent (ethyl acetate) and each fraction analysed chromatographically by TLC. In all cases where an acid or base extract was partitioned, the greater proportion of the radioactivity partitioned into the organic fraction. Flutriafol and C6 were predominantly found and other components were C1, C2, C3 and C4.

At 42 DAT, flutriafol was the major radioactive component in the solvent/water extracts of the remaining plant samples, accounting for 52.4 and 47.6% TRR (0.129 and 0.169 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. None of the remaining components in these extracts individually represented more than 6.1 and 4.5% TRR (0.015 and 0.016 mg/kg). Flutriafol was present in the enzyme extracts and in the strong acid extracts. In total, flutriafol accounted for 3.2 and 11.0% TRR (0.008 and 0.039 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. All other components did not individually represent more than 0.8 and 1.7% TRR (0.002 and 0.006 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively).

The seeds at 42 DAT were initially extracted with hexane and these extracts analysed chromatographically. Flutriafol was the only radioactive component in these extracts, therefore accounting for 31.5 and 27.2% TRR (0.415 and 0.198 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. Flutriafol was the major radioactive component in the subsequent solvent/water extracts of the seeds, accounting for 29.8 and 27.4% TRR (0.392 and 0.200 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively). Components R5a and R5b each accounted for 3.8% TRR (0.028 mg/kg) in plants treated with [^{14}C-carbinol] flutriafol, and for 3.8 and 3.6% TRR (0.050 and 0.048 mg/kg) in plants treated with [^{14}C-triazole] flutriafol. Component R1 in plants treated with [^{14}C-triazole] flutriafol accounted for 3.5% TRR (0.046 mg/kg). Other radioactive components in these seed extracts did not individually represent more than 1.7 and 2.1% TRR (0.015 and 0.023 mg/kg). Flutriafol was present in the enzyme extracts, in the weak base extracts and in the strong acid and strong base extracts. In total, flutriafol accounted for 6.3 and 7.3% TRR (0.082 and 0.054 mg/kg) in samples treated with the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. Component C6, which was present in the strong acid and strong base extracts only, accounted for a total of 3.0 and 2.9% TRR (0.039 and 0.021 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively). In total, C1 accounted for 3.3 and 1.4% TRR (0.044 and 0.011 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively).

Table 15 Concentrations and proportions of radioactive components in the initial solvent/water extracts of oilseed rape samples taken following application of ^{14}C-flutriafol

Sampling event	Application		2nd intermediate				Harvest			
DAT	0		14				42			
Plant portion	Whole plant (forage)		Pods		Remaining plant		Seeds		Remaining plant	
Radiolabel	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]
	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Hexane extracts Flutriafol							0.198 (27.2)	0.415 (31.5)		
Solvent/ water extracts[a]										
Flutriafol	1.437 (96.0)	0.758 (96.9)	0.245 (31.5)	0.243 (32.49	1.309 (81.8)	0.886 (76.1)	0.200 (27.4)	0.392 (29.8)	0.169 (47.6)	0.129 (52.4)
R1	d	0.003 (0.4)	d	0.012 (1.6)	d	0.003 (0.3)	d	0.046 (3.5)	0.016 (4.5)	0.012 (4.9)
R2	d	d	0.007 (0.9)	0.008 (1.1)	0.006 (0.4)	0.007 (0.6)	0.013 (1.8)	0.023 (1.7)	d	0.004 (1.6)
R3	d	d	0.012 (1.5)	0.006 (0.8)	0.015 (0.9)	0.014 (1.2)	0.012 (1.6)	d	0.002 (0.6)	0.003 (1.2)

Sampling event	Application		2nd intermediate				Harvest			
DAT	0		14				42			
Plant portion	Whole plant (forage)		Pods		Remaining plant		Seeds		Remaining plant	
Radiolabel	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]
	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
R4	d	d	0.008 (1.0)	0.009 (1.2)	0.015 (0.9)	0.012 (1.0)	0.015 (2.1)	d	0.003 (0.8)	0.005 (2.0)
R5a	0.007 (0.5)	d	0.013 (1.7)	0.011 (1.5)	0.043 (2.7)	0.031 (2.7)	0.028 (3.8)	0.050 (3.8)	0.009 (2.5)	0.014 (5.7)
R5b		d	0.020 (2.6)	0.011 (1.5)	0.050 (3.1)	0.030 (2.6)	0.028 (3.8)	0.048 (3.6)	0.009 (2.5)	0.015 (6.1)
R6	0.013 (0.9)	0.002 (0.3)	d	d	0.010 (0.6)	0.003 (0.3)	d	d	0.002 (0.6)	d
Others[b]	0.009 (0.6)	0.006 (0.8)	0.007 (0.9)	0.007 (0.9)	0.021 (1.3)	0.011 (0.9)	0.011 (1.5)	d	0.005 (1.4)	0.004 (1.6)
Total[c]	1.466 (97.9)	0.769 (98.3)	0.312 (40.1)	0.309 (41.2)	1.467 (91.6)	0.997 (85.6)	0.505 (69.2)	0.974 (74.0)	0.214 (60.4)	0.187 (76.2)

[a] Includes acetonitrile, acetonitrile/water and water extracts

[b] Radioactivity distributed throughout regions of the chromatogram other than those specified and which did not contain any discrete radioactive components

[c] Totals may differ from the sums of the individual component values due to rounding

[d] Not apparent or below the limit of detection (LOD = 0.0003 mg/kg)

Table 16 Concentrations and proportions of radioactive components in the further extracts of oilseed rape samples taken following application of ^{14}C-flutriafol

Sampling event	2nd intermediate				Harvest			
DAT	14				42			
Plant portion	Pods		Remaining plant		Seeds		Remaining plant	
Radiolabel	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]
	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Enzyme extracts								
Flutriafol	0.119 (15.3)	0.119 (15.8)	0.045 (2.8)	0.052 (4.5)	0.003 (0.4)	0.006 (0.5)	0.010 (2.8)	a
C1	0.005 (0.6)	0.004 (0.5)	0.004 (0.2)	0.005 (0.4)	0.003 (0.4)	0.007 (0.5)	0.004 (1.1)	a
Others	0.003 (0.4)	0.005 (0.7)	0.004 (0.2)	0.002 (0.2)	0.003 (0.4)	0.005 (0.4)	0.001 (0.3)	a
0.1 M NaOH (organic)								
Flutriafol	0.026 (3.3)	0.014 (1.9)	np	np	0.004 (0.5)	0.010 (0.8)	np	np
C1	0.001 (0.1)	0.002 (0.3)	np	np	0.001 (0.1)	0.003 (0.2)	np	np
Others	0.001 (0.1)	0.002 (0.3)	np	np	0.001 (0.1)	0.012 (0.9)	np	np
0.1 M NaOH (aqueous)	a	a	np	np	a	a	np	np
6 M acid (organic)								
Flutriafol	0.009 (1.2)	0.010 (1.3)	0.003 (0.2)	0.005 (0.4)	0.016 (2.2)	0.018 (1.4)	0.018 (5.1)	0.005 (2.0)
C1	0.003 (0.4)	0.003 (0.4)	< 0.001 (< 0.1)	0.001 (0.1)	0.002 (0.3)	0.004 (0.3)	0.001 (0.3)	< 0.001 (<0.4)
C4	0.003 (0.4)	0.004 (0.5)	0.001 (0.1)	0.001 (0.1)	b	b	b	< 0.001 (<0.4)
C6	0.075 (9.6)	0.061 (8.1)	0.010 (0.6)	0.012 (1.0)	0.018 (2.5)	0.030 (2.3)	0.005 (1.4)	0.002 (0.8)
Others	0.006 (0.8)	0.008 (1.1)	0.001 (0.1)	0.001 (0.1)	0.001 (0.1)	0.008 (0.6)	0.001 (0.3)	0.001 (0.4)
6 M acid (aqueous)								

Sampling event	2nd intermediate				Harvest			
DAT	14				42			
Plant portion	Pods		Remaining plant		Seeds		Remaining plant	
Radiolabel	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]	[carbinol]	[triazole]
	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Flutriafol	0.014 (1.8)	0.014 (1.9)	0.004 (0.2)	0.004 (0.3)	0.020 (2.7)	0.008 (0.6)	0.011 (3.1)	0.003 (1.2)
C1	0.008 (1.0)	0.006 (0.8)	0.001 (0.1)	0.004 (0.3)	0.004 (0.5)	0.006 (0.5)	0.001 (0.3)	0.001 (0.4)
C6	0.014 (1.8)	0.009 (1.2)	0.001 (0.1)	[b]	[b]	[b]	[b]	[b]
Others	0.012 (1.5)	0.012 (1.6)	0.003 (0.2)	0.006 (0.5)	0.003 (0.4)	0.031 (2.4)	0.003 (0.8)	0.001 (0.4)
2 M NaOH (organic)								
Flutriafol	0.048 (6.2)	0.046 (6.1)	0.012 (0.7)	0.016 (1.4)	0.011 (1.5)	0.040 (3.0)	np	np
C1	0.008 (1.0)	0.012 (1.6)	0.003 (0.2)	0.002 (0.2)	0.001 (0.1)	0.004 (0.3)	np	np
C2	0.003 (0.4)	0.004 (0.5)	0.001 (0.1)	0.001 (0.1)	[b]	[b]	np	np
C3	0.001 (0.1)	0.002 (0.3)	[b]	[b]	[b]	[b]	np	np
C6	0.027 (3.5)	0.021 (2.8)	0.006 (0.4)	0.006 (0.5)	0.003 (0.4)	0.009 (0.7)	np	np
Others	0.003 (0.4)	0.003 (0.4)	0.001 (0.1)	0.001 (0.1)	0.002 (0.3)	0.013 (1.0)	np	np
2 M NaOH (aqueous)								
C1	0.010 (1.3)	0.020 (2.7)	0.003 (0.2)	0.006 (0.5)	< 0.001 (<0.1)	0.020 (1.5)	np	np
Others	0.003 (0.4)	0.011 (1.5)	< 0.001 (< 0.1)	0.001 (0.1)	0.001 (0.1)	0.033 (2.5)	np	np
Total Flutriafol	0.216 (27.8)	0.203 (27.0)	0.064 (3.9)	0.077 (6.6)	0.054 (7.3)	0.082 (6.3)	0.039 (11.0)	0.008 (3.2)
Total C1	0.035 (4.4)	0.047 (6.3)	0.011 (0.7)	0.018 (1.5)	0.011 (1.4)	0.044 (3.3)	0.006 (1.7)	0.001 (0.4)
Total C6	0.116 (14.9)	0.091 (12.1)	0.017 (1.1)	0.018 (1.5)	0.021 (2.9)	0.039 (3.0)	0.005 (1.4)	0.002 (0.8)

[a] Radioactivity too low for meaningful chromatography

[b] not apparent

np: not partitioned

C6: defluorinated flutriafol

In the forage samples taken at 0 DAT, and in the remaining plant samples at 14 DAT and 42 DAT, most of the residues were extractable by acetonitrile/water. In the pods and seeds, significant residues were released only by successive additional enzyme and acid/base treatments. Flutriafol was found both in the initial acetonitrile/water extracts and in the further enzyme/acid/base extracts, suggesting some binding to the plant matrix. A hexose conjugate of flutriafol was released by acetonitrile/water, while the other identified residue, a defluorinated flutriafol, was found only in the further extracts. All extractable residues contained both the carbinol carbon and triazole ring radiolabel centres indicating that no cleavage of the flutriafol molecule had occurred.

Summary of plant metabolism

Metabolism of ^{14}C-flutriafol labelled in the triazole ring and carbinol carbon has been studied in apples, sugar beet, barley, wheat and oilseed rape, which are suitable to cover the crop groups pome fruits, root/tuber vegetables, cereals and pulses/oilseeds. Flutriafol is the major component of the residues found in apples, sugar beet (forage), cereals (straw) and oilseed rape (seed and remaining plant). The following metabolic pathways were identified in the plant metabolism studies available.

cleavage of the flutriafol molecule between the methylene and the carbinol carbon and subsequent formation of triazole, triazole acetic acid and triazole alanine (this was only observed in the metabolism studies conducted in wheat and barley)

hexose conjugation of flutriafol

defluorination of flutriafol

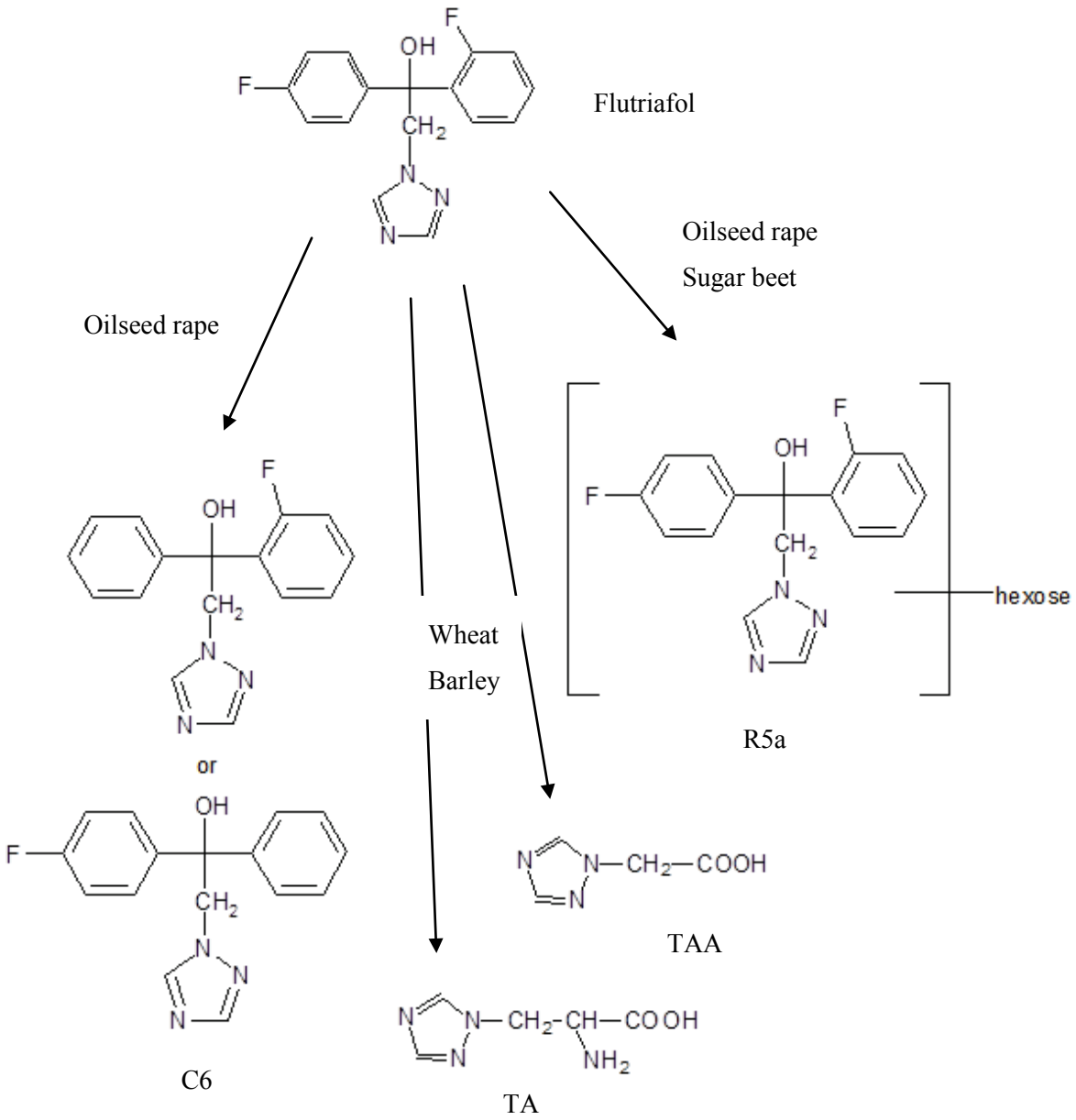


Figure 4 Proposed metabolic pathway of flutriafol in plants

Environmental fate in soil

The Meeting received information on aerobic degradation in soil, photolysis on soil surface, rotational crop, adsorption/desorption, column leaching and hydrolytic degradation study. Because flutriafol is intend for use as foliar and also on peanuts, aerobic degradation, soil photolysis, rotational crop and hydrolytic degradation study relevant to the current evaluations were reported below (FAO Manual 2009).

The fate and behaviour of flutriafol in soils were investigated using [^{14}C-triazole] and [^{14}C-carbinol] labelled compounds.

Aerobic degradation

The metabolism and degradation of flutriafol has been studied in 6 different soils under aerobic conditions at a nominal average temperature of 20 °C for 252 days (Arnold, 1982: 173 FLU). ^{14}C-triazole labelled flutriafol was applied to a range of six soils and ^{14}C-carbinol labelled flutriafol to two of these. The treated soil characteristics are summarized in Table 17. All the soils were treated with 100 g ai/ha and incubated at 40% moisture holding capacity (MHC) at 20 °C in a stream of moist CO_2-free air for periods of 126 or 252 days. In addition, one soil type sample (sandy clay loam) was treated with an exaggerated application rate (1,000 g ai/ha) and was incubated at 15% and 40% MHC and up to 30 °C to determine the effect of moisture content and of temperature on the rate of degradation of ^{14}C-flutriafol.

Table 17 Soils used to investigate the route and rate of aerobic degradation of ^{14}C-flutriafol

Soil designation	18 Acres	Frensham	18 Acres	Frensham	Flexford	Horsley	Boxford	Chalgrove
Radiolabel	^{14}C-carbinol		^{14}C-triazole					
Soil type	Sandy clay loam	Loamy sand	Sandy loam	Loamy sand	Sandy loam	Loamy sand	Clay loam	Sandy clay loam
% Sand	57	82	62	82	69	80	40	53
% Silt	13	6	16	10	11	2	20	17
% Clay	30	12	22	9	21	17	40	30
pH value (H_2O)	6.8	5.8	6.8	5.7	5.6	6.5	7.7	6.4
Organic matter (%)	4.7	1.8	4.9	2.3	4.4	2.7	4.3	3.7
Cation exchange capacity (meq/100 g)	38.0	9.8	38.6	14.7	13.1	14.4	40.6	10.7
MHC (Zero suction)	90.8	50.6	66.0	50.8	71.6	57.5	95.1	57.1

Total recovery of radioactivity was comprised of that in extracts of soil, evolved as $^{14}CO_2$ and radioactivity remaining unextracted from soil. Recovery values ranged from 89–111% of the applied radioactivity (AR) with a mean of 99.8%.

Table 18 Recovery and extraction of radioactivity from soils treated with ^{14}C-labelled flutriafol at a nominal level corresponding to 100 g ai/ha and incubated at 20 °C under aerobic conditions

Incubation time (days)	Flutriafol (% recovery)	Extraction (% recovery)	Unextracted (% recovery)	$^{14}CO_2$ (% recovery)	Recovery (% AR)
^{14}C-carbinol					
18 Acres					
0	99.1	100.0	0.0	0.0	105
21	97.8	99.0	0.6	0.4	111
63	95.5	98.1	1.0	0.8	102
126	94.9	96.8	2.0	1.2	103
Frensham					
0	98.5	100.0	0.0	0.0	103
21	96.0	98.4	0.8	0.7	106
63	93.3	96.6	1.7	1.7	100
126	91.7	94.6	2.8	2.6	103
^{14}C-triazole					
18 Acres					
0	97.8	99.9	0.1	0.0	105

Incubation time (days)	Flutriafol (% recovery)	Extraction (% recovery)	Unextracted (% recovery)	$^{14}CO_2$ (% recovery)	Recovery (% AR)
^{14}C-carbinol					
21	95.0	98.2	1.6	0.1	94
63	88.8	96.2	3.5	0.4	100
126	85.4	93.9	5.2	1.0	102
252	83.8	90.1	7.1	2.8	98
Frensham					
0	94.2	100.0	0.0	0.0	106
21	95.0	98.3	1.5	0.2	93
63	88.2	95.9	2.9	1.2	99
126	87.9	93.3	4.5	2.2	103
252	82.7	87.9	8.6	3.6	101
Boxford					
0	99.2	99.9	0.0	0.0	103
21	95.0	98.1	1.8	0.1	110
63	93.6	96.2	3.0	0.7	102
126	88.2	92.6	5.5	1.9	104
Chalgrove					
0	96.8	100.0	0.0	0.0	101
21	97.5	99.3	0.7	0.0	111
63	96.7	99.1	0.8	0.0	99
126	94.7	99.1	0.9	0.1	104
Horsley					
0	97.3	99.9	0.1	0.0	103
21	95.4	98.5	1.5	0.1	109
63	91.7	97.2	2.5	0.3	106
126	85.4	94.2	5.0	0.8	103
Flexford					
0	98.2	100.0	0.0	0.0	101
21	98.2	99.3	0.8	0.0	110
63	96.6	98.9	1.0	0.1	104
126	95.0	97.5	2.4	0.1	103

In six moist (40% MHC) soils treated at 100 g ai/ha rate and incubated at 20 °C, approximately 3–12% loss of extractable parent was observed over 126 days period. Treatment at 10 × rate (1,000 g ai/ha and at 40% MHC, 20 °C) and incubation of 100 g ai/ha treatments at 30 °C (at 40% MHC) had little if any effect on the rate of flutriafol degradation in the one soil type studied. Flutriafol at 100 g ai/ha incubated in a soil at 15% MHC (at 20 °C) showed negligible degradation over 126 days period. Little $^{14}CO_2$ was evolved from any of the treatments (less than 4% after 126 days incubation). There were no differences in rates of $^{14}CO_2$ evolution from the two radiolabels.

Two degradation products were observed on autoradiograms from TLC of some soil extracts (^{14}C-triazole labelled treatments). These products were quantified in ’18 Acres’ and ‘Frensham’ after 126 days and together represented approximately 4% or less of the total recovered radioactivity. Only small amounts of radioactivity remained unextracted from soil.

Table 18 Recovery and extraction of radioactivity from 18 Acres soil treated with ^{14}C-triazole labelled flutriafol at a nominal level corresponding to 100 g ai/ha and 1,000 g ai/ha and incubated under different conditions

Incubation Time (days)	Flutriafol (% recovery)	Extraction (% recovery)	Unextracted (% recovery)	$^{14}CO_2$ (% recovery)	Recovery (% AR)
100 g/ha, 15% MHC, 20°C					
0	97.7	99.7	0.3	0.0	107
21	95.9	98.9	1.1	0.0	91
63	96.6	98.9	1.0	0.0	100
126	96.8	98.4	1.5	0.1	101
100 g/ha, 40% MHC, 30°C					
0	98.4	99.9	0.1	0.0	106
21	92.6	97.5	2.5	0.1	93
63	89.1	95.0	4.8	0.2	99

Incubation Time (days)	Flutriafol (% recovery)	Extraction (% recovery)	Unextracted (% recovery)	$^{14}CO_2$ (% recovery)	Recovery (% AR)
126	89.8	93.6	6.1	0.3	99
1000 g/ha, 40% MHC, 20°C					
0	94.9	96.7	0.3	0.0	103
63	91.7	97.3	2.5	0.2	99
252	86.8	93.4	5.8	0.9	100

Flutriafol is slowly degraded in laboratory incubated soils. Approximately 85% remained after 252 days in a loamy sand and a sandy clay loam. Small amounts of $^{14}CO_2$ were released from both the triazole ring and carbinol carbon atom. Minor extractable ^{14}C-degradation products accounted for 4% or less of the applied compound. Approximately 90% or more of the radioactivity could be extracted from soil after 252 days.

Table 19 DT_{50} and DT_{90} values of flutriafol in soils under aerobic conditions

Soil	Texture	DT_{50} (days)	DT_{90} (days)	r^2
20 °C, 40% MWHC, 100 g ai/ha				
18 Acres (carbinol label)	Sandy clay loam	2017	6700	0.87
Frensham (carbinol label)	Loamy sand	1264	4200	0.91
18 Acres (triazole label)	Sandy clay loam	1125	3736	0.81
Frensham (triazole label)	Loamy sand	1290	4286	0.89
Boxford	Clay loam	811	2694	0.94
Chalgrove	Sandy clay loam	3492	11599	0.78
Horsley	Loamy sand	672	2231	1.00
Flexford	Sandy loam	2464	8185	0.97
Effect of incubation conditions at 18 Acres				
20 °C, 15% MHC, 100g/ha	Sandy clay loam	-	-	-
30 °C, 40% MHC, 100g/ha	Sandy clay loam	1058	3514	0.61
20 °C, 40% MHC, 1000g/ha	Sandy clay loam	2031	6748	0.98

-: no meaningful value could be calculated due to the limited overall degradation

The DT_{50} and DT_{90} values have been calculated from the existing data using non-linear regression analysis of the first order kinetic equation.

Soil photolysis

The photodegradation of flutriafol was investigated on a sandy loam soil (Cavell, 1982: 172 FLU). Glass plates were coated with slurry made from a sieved (0.5 mm) sandy loam soil and glass distilled water. The slurry was spread onto the glass plates with a TLC spreader at a thickness of 1 mm. ^{14}C-triazole and ^{14}C-carbinol labelled flutriafol at a rate of approximately 94 g ai/ha was applied evenly, by syringe, to soil plates. The plates were air-dried for 1 hour before exposure to light. Soil plates (1 × 10 cm) treated with either radiochemical were placed in stoppered borosilicate or quartz glass test-tubes and irradiated in a photochemical reactor fitted with a merry-go-round accessory for 7days. Borosilicate glass containers were used because the high energy wavelengths of UV light, produced by the apparatus but not found in sunlight, are not transmitted by this glass, thereby simulating "natural" sunlight. Quartz glass transmits all the UV light produced by the photochemical reactor and so gives rise to more stringent conditions for photolysis. In addition, duplicate soil plates treated with each radiochemical were each put into separate, quartz glass conical flasks which were stopped and then placed outside in an unshaded location for exposure to daylight. All the flasks were left outside for 30 days.

Soil samples were scraped from the plates, extracted with 30 mL acetonitrile and afterwards with acetonitrile/water (1:1, v/v). The extracts were concentrated and analysed by TLC. Extracted soil samples were combusted to determine levels of non-extractable residues.

After 7 days photochemical reactor irradiation, with the soil plates contained in quartz vessels, 60–67% of the total radioactivity applied to the soil plates was chracterised as flutriafol. 1,2,4-Triazole and 2,4'-difluorobenzophenone accounted for 2–3% of the radioactive residues. The

remainder of the radioactive residue (14–18%) consisted of a mixture of ^{14}C-polar extractable compounds and unextractable radioactive residues. The percentage of unextractable ^{14}C-residues was generally greater for the ^{14}C-triazole labeled materials.

For the outdoor experiment, 91.8% of the applied radioactivity was recovered from the 30 day dark control soil of which 87.7% was characterized as flutriafol (> 95% recovered radioactivity).

Table 20 Distribution and characterisation of radioactivity in irradiated and dark control samples treated with ^{14}C-triazole and ^{14}C-carbinol labelled flutriafol (results expressed in % of applied radioactivity)

Radiolabel position	Flutriafol	1,2,4-triazole	2,4'-difluoro-benzophenone	Polar baseline components	Unknown components	Unextracted residues	Recovery
Immediately after application							
Triazole	93.4	-	-	0.2	0.6	0.3	95.6
Carbinol	88.0	-	-	0.3	0.8	0.2	94.7
Artificial Light (borosilicate tubes, after 7 days irradiation)							
Triazole	70.4	4.1	-	5.5	5.4	7.2	94.3
Carbinol	76.5	-	3.6	3.0	3.4	3.4	93.3
Artificial Light (quarts tubes, after 7 days irradiation)							
Triazole	59.8	1.8	-	4.5	5.2	10.2	95.0
Carbinol	66.7	-	3.0	6.6	7.5	3.4	87.2
Natural light (after 30 days irradiation)							
Triazole	74.5	<1.0	-	2.7	*ca.* 5.0	6.2	94.6
Carbinol	82.4	-	1.8	1.4	7.0	4.0	101.3
Dark control sample							
Triazole	87.7	-	-	3.5		0.6	98.1

Despite generally good light conditions during the period in which the soil plate were irradiated, photodegradation of the compound was less extensive than when the treated soil was irradiated in a photochemical reactor. After 30 days irradiation, 75–82% of the applied radioactivity was characterized as flutriafol. The photodegradation products formed were similar to those seen after artificial irradiation and accounted for 7–10% of the applied radioactivity. Flutriafol appears to be relatively photochemically stable on a dry soil surface.

Residues in rotational crops

Confined rotational crop studies

Study 1

A study to investigate the degree and nature of the residue in rotational crops was conducted following an application of ^{14}C-flutriafol to the soil (Skidmore, 1988: 82 FLU). The soil was treated with ^{14}C-flutriafol, labelled in either triazole ring or carbinol carbon, at an application rate equivalent to approximately 250 g ai/ha. The pods filled with the treated soil were maintained under greenhouse conditions and watered as required. At intervals of 30, 120 and 365 days after treatment, wheat, peas, sugar beet and oilseed rape were sown into the soil. At harvest, the crops were separated into their component parts and the levels of radioactivity measured using combustion/LSC. The nature of these residues was investigated on representative crop components using a range of chromatographic techniques.

Table 21 Total radioactive residues (TRRs) found in crops (mg/kg flutriafol equivalent) at time points of harvesting

Crop		TRR (mg/kg)					
		30 days after treatment		120 days after treatment		365 days after treatment	
		^{14}C-triazole	^{14}C-carbinol	^{14}C-triazole	^{14}C-carbinol	^{14}C-triazole	^{14}C-carbinol
Wheat	Grain	1.04	0.04	1.22	0.02	0.30	< 0.01
	Chaff	1.82	2.90	1.58	0.88	0.20	0.10
	Straw	6.47	10.46	1.32	0.93	0.20	0.13

Crop		TRR (mg/kg)					
		30 days after treatment		120 days after treatment		365 days after treatment	
		^{14}C-triazole	^{14}C-carbinol	^{14}C-triazole	^{14}C-carbinol	^{14}C-triazole	^{14}C-carbinol
Pea	Peas	0.32	0.01	0.32	< 0.01	0.20	< 0.01
	Pods	0.14	0.05	0.10	0.03	0.10	< 0.01
	Foliage	1.08	1.25	0.63	0.33	0.10	< 0.01
Sugar beet	Roots	0.08	0.02	0.09	< 0.01	0.03	< 0.01
	Foliage	0.60	0.20	0.57	0.19	0.35	0.13
Rape	Seed	na	na	2.16	0.03	0.60	< 0.01
	Pods	na	na	2.13	0.97	0.30	0.13
	Foliage	na	na	0.67	0.28	0.10	0.04

na: not analysed

Table 22 Total radioactive residues (TRRs) and the nature of residues in wheat straw and grain and sugar beet foliage and root samples

Crop component	^{14}C-label	Planting intervals (day)	TRR (mg/kg)	Flutriafol (%)	4-Hydroxy-flutriafol (%)	Triazole (%)	Triazole alanine (%)	Triazole acetic acid (%)	Un-known (%)	Base-line (%)	Remained on chromato-plates (%)	Un extracted (%)
Wheat grain	Triazole	120	1.18	-	-	-	48.5	18.8	-	-	4.8	12.7
Wheat straw	Triazole	120	2.45	38.2	1.5	-	-	15.5	16.8[a]	-	5.5	17.1
Sugar beet foliage	Triazole	120	0.56	17.0	2.5	-	2.5	21.0	15.9[a]	1.4	0.5	3.6
Sugar beet root	Triazole	120	0.12	4.3	-	17.3	19.6	2.7	-	4.7	2.6	35.6
Wheat grain	Triazole	365	0.31	-	-	-	50.5	14.2	-	-	0.6	15.2
Wheat straw	Triazole	365	0.16	30.7	-	-	1.1	22.2	3.0	-	3.5	31.4
Wheat straw	Carbinol	120	1.07	43.3	-	-	-	-	25.5[b]	0.7	1.8	18.3
Sugar beet foliage	Carbinol	120	0.31	25.7	-	-	-	-	51.2[c]	-	0.5	7.0

[a] consists of at least two compounds

[b] consists of at least three compounds

[c] consists of at least five compounds

In general, radioactive residues from the 30 and 120 day planting intervals were similar except wheat straw. The results showed that the major components found were flutriafol, triazole alanine and triazole acetic acid. Small levels of 4-hydroxy-flutriafol were found in the wheat straw and sugar beet foliage.

Study 2

A confined rotational crop study was conducted using [^{14}C] triazole and [^{14}C] carbinol flutriafol with lettuce, radish, and wheat at 30 day and 120 day plant-back intervals (Dohn, 2009: 1624 FLU). Two radiolabeled forms of flutriafol were used, and each test chemical was applied to four 1.39 m^2 test plots. The test plots were located outdoors, and consisted of wooden boxes filled with sandy loam soil to a depth of 0.46 m. The applications for the 30 day plant back interval occurred 90 days after the applications for the 120 day plant back interval. This allowed all crops to be planted on the same day. Four test plots (one for each radiolabel and plant back interval) were used to grow wheat. The remaining four plots were used for lettuce and radish, with the area of each plot equally divided between the two crops. The test substance was dissolved in ethanol and applied to test plots with hand operated pump sprayers. The target application rate was 25 mg/m^2 (250 g/ha). The rate actually applied to all plots was 260 g/ha (104% of target). Crop samples were homogenized in the presence of dry ice using food processing equipment, and the total radioactive residues were measured by combustion analysis/LSC.

The TRRs in plant samples grown in [^{14}C] triazole flutriafol treated soil were consistently greater than the plant samples obtain from [^{14}C] carbinol flutriafol treated plots. The differences in TRR values observed in the wheat grain samples were particularly noteworthy. Residues in crops planted 120 days after flutriafol application to soil had similar TRR values to those found in crops planted 30 days after flutriafol application. Radish tops had higher residues than the corresponding radish roots samples, indicating that flutriafol and its metabolites were translocated within the xylem of the plant tissue. The TRR in the vegetative portions of wheat increased with maturity, in the order forage ←hay ← straw.

Table 23 Total radioactive residues (TRR) values in 30 and 120 day rotational crops by combustion analysis

Commodity	Plant-back interval (days)	[^{14}C]triazole[a] (mg/kg)	[^{14}C]carbinol[a] (mg/kg)
Lettuce	30	0.076 ± 0.007	0.048 ± 0.004
Lettuce	120	0.075 ± 0.012[b]	0.050 ± 0.003
Radish Tops	30	0.177 ± 0.013	0.060 ± 0.010
Radish Tops	120	0.084 ± 0.006	0.048 ± 0.007
Radish Roots	30	0.066 ± 0.007	0.023 ± 0.004
Radish Roots	120	0.051 ± 0.006	0.020 ± 0.003
Wheat Forage	30	0.230 ± 0.018	0.130 ± 0.009
Wheat Forage	120	0.242 ± 0.021	0.130 ± 0.010
Wheat Hay	30	0.668 ± 0.042	0.357 ± 0.017
Wheat Hay	120	0.497 ± 0.015	0.290 ± 0.013
Wheat Straw	30	1.749 ± 0.127	1.129 ± 0.083
Wheat Straw	120	1.395 ± 0.057	1.220 ± 0.037
Wheat Grain	30	0.648 ± 0.054	0.032 ± 0.005
Wheat Grain	120	0.528 ± 0.024	0.028 ± 0.002

[a] Values from combustion/LSC of five replicate sub samples.

[b] Values from combustion/LSC of ten replicate sub samples.

Flutriafol was a significant component of the residue in all treated crop samples, ranging from barely detectable in wheat grain (0.003 to 0.009 mg/kg) to a high of 0.416 mg/kg in one straw sample. The most significant residues in all crops that contained only the triazole label position were identified/characterized as Triazole alanine (present in all crops), Triazole acetic acid (found in wheat hay, straw, and grain), and Triazole lactic acid (found in all crop matrices except wheat grain). The free 1,2,4-triazole was not detected in any crop samples. Two metabolite groups (M28 and M30) that contained both radiolabeled positions were observed in wheat hay and straw. These residues were present at higher concentration in straw (0.148 to 0.165 mg/kg for M28, and 0.094 to 0.143 mg/kg for M30). M28 was characterized as a single component, which was a glucoside with an aglycone more polar than flutriafol. Characterization data on M30 indicated that this component consisted of two compounds, one a glucoside yielding an aglycone more polar than flutriafol (but different from the M28 aglycone). The second component of M30 was not a glucoside (or was a very poor substrate for ß-glucosidase), but was partially hydrolyzed by acid under moderate conditions. No other metabolites that contained both label positions were observed in any crop samples in significant (> 0.05 mg/kg) concentrations.

Table 24 Nature of the residue in lettuce grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol	
	30-Day Plant Back		30-Day Plant Back		120-Day Plant Back	
	Flutriafol equivalents (mg/kg)	% TRR	Flutriafol equivalents (mg/kg)	% TRR	Flutriafol equivalents (mg/kg)	% TRR
Flutriafol	0.035	49.3	0.030	61.2	0.039	50.6
M31	0.006	8.5	0.011	22.4	0.006	7.8
1,2,4-triazole	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.012	16.9	n.d.	-	0.012	15.6
Triazole acetic acid	n.d.	-	n.d.	-	n.d.	-
Triazole alanine	0.009	12.7	n.d.	-	0.008	10.4
Other extractable components	0.002	2.8	0.003	6.1	0.002	2.6

Component	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol	
	30-Day Plant Back		30-Day Plant Back		120-Day Plant Back	
	Flutriafol equivalents (mg/kg)	% TRR	Flutriafol equivalents (mg/kg)	% TRR	Flutriafol equivalents (mg/kg)	% TRR
Total extracted	0.064	90.1	0.044	89.8	0.067	87.0
PES	0.007	9.9	0.005	10.2	0.010	13.0
TRR (Extracted+PES)	0.071	100	0.049	100	0.077	100
Subtotal identified[a]	0.056	78.9	0.030	61.2	0.059	76.6
TRR by combustion (for comparison)	0.076 ± 0.007	-	0.048 ± 0.004	-	0.075 ± 0.012	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 25 Nature of the residue in radish roots grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]triazole flutriafol	
	30-Day Plant Back		120-Day Plant Back	
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.015	21.4	0.012	25.0
1,2,4-Triazole	n.d.	-	n.d.	-
Triazole lactic acid	0.003	4.3	0.001	2.1
Triazole acetic acid	n.d.	-	n.d.	-
Triazole alanine	0.033	47.1	0.020	41.7
Other Extractable Components	0.005	7.1	0.005	10.4
Total Extracted	0.056	80.0	0.038	79.2
PES	0.014	20.0	0.010	20.8
TRR (Extracted+PES)	0.070	100	0.048	100
Subtotal identified[a]	0.051	72.9	0.033	68.8
TRR by combustion (for comparison)	0.066 ± 0.007	-	0.051 ± 0.006	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 26 Nature of the residue in radish top grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol	
	30-Day Plant Back		30-Day Plant Back		120-Day Plant Back	
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.098	53.3	0.043	74.1	0.046	54.8
M30	0.001	0.5	0.001	1.7	0.001	1.2
M28	0.003	1.6	0.004	6.9	0.003	3.6
1,2,4-Triazole	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.004	2.2	n.d.	-	0.001	1.2
Triazole acetic acid	n.d.	-	n.d.	-	n.d.	-
Triazole alanine	0.051	27.7	n.d.	-	0.021	25.0
Other Extractable Components	0.010	5.4	0.002	3.4	0.004	4.8
Total Extracted	0.167	90.8	0.050	86.2	0.076	90.5
PES	0.017	9.2	0.008	13.8	0.008	9.5
TRR (Extracted+PES)	0.184	100	0.058	100	0.084	100
Subtotal identified[a]	0.153	83.2	0.043	74.1	0.068	81.0
TRR by combustion (for comparison)	0.177 ± 0.013	-	0.060 ± 0.010	-	0.084 ± 0.006	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 27 Nature of the residue in wheat forage grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]triazole flutriafol	
	30-Day Plant Back		120-Day Plant Back	
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.114	53.5	0.136	58.1
M30	0.003	1.4	0.006	2.6
M28	0.016	7.5	0.013	5.6
1,2,4-Triazole	n.d.	-	n.d.	-
Triazole lactic acid	0.013	6.1	0.009	3.8
Triazole acetic acid	n.d.	-	n.d.	-
Triazole alanine	0.026	12.2	0.024	10.3
Other Extractable Components	0.019	8.9	0.022	9.4
Total Extracted	0.191	89.7	0.210	89.7
PES	0.022	10.3	0.024	10.3
TRR (Extracted+PES)	0.213	100	0.234	100
Subtotal identified[a]	0.153	71.8	0.169	72.2
TRR by combustion (for comparison)	0.230 ± 0.018	-	0.242 ± 0.021	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 28 Nature of the residue in wheat hay grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol	
	30-Day Plant Back		30-Day Plant Back		120-Day Plant Back		120-Day Plant Back	
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.183	27.6	0.130	41.7	0.152	31.7	0.140	50.4
M30	0.048	7.3	0.043	13.8	0.031	6.5	0.037	13.3
M28	0.025	3.8	0.024	7.7	0.020	4.2	0.026	9.4
1,2,4-Triazole	n.d.	-	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.049	7.4	n.d.	-	0.036	7.5	n.d.	-
Triazole acetic acid	0.060	9.1	n.d.	-	0.036	7.5	n.d.	-
Triazole alanine	0.123	18.6	n.d.	-	0.067	14.0	n.d.	-
Other Extractable Components	0.038	5.7	0.026	8.3	0.041	8.5	0.023	8.3
Total Extracted (ACN:H_2O)	0.526	79.5	0.223	71.5	0.383	79.8	0.226	81.3
Flutriafol Extracted (1 N NaOH)[a]	0.036	5.4	1N NaOH extraction not performed on the PES of these samples.					
Others extracted (1 N NaOH)[a]	0.052	7.9						
PES[b]	0.048	7.3	0.089	28.5	0.097	20.2	0.052	18.7
TRR (Extracted+ PES)	0.662	100	0.312	100	0.480	100	0.278	100
Subtotal identified[c]	0.451	68.1	0.130	41.7	0.291	60.6	0.140	50.4
TRR by combustion (for comparison)	0.668 ± 0.042	-	0.357 ± 0.017		0.497 ± 0.015	-	0.290 ± 0.013	-

[a] Extracted from PES

[b] mg/kg after 1 N NaOH extraction

[c] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 29 Nature of the residue in wheat straw grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol	
	30-Day Plant Back		30-Day Plant Back		120-Day Plant Back		120-Day Plant Back	
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.298	17.0	0.416	34.0	0.263	18.1	0.413	31.9
M30	0.133	7.6	0.094	7.7	0.123	8.4	0.143	11.0
M28	0.160	9.1	0.148	12.1	0.165	11.3	0.159	12.3
1,2,4-Triazole	n.d.	-	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.191	10.9	n.d.	-	0.170	11.7	n.d.	-
Triazole acetic acid	0.104	5.9	n.d.	-	0.084	5.8	n.d.	-
Triazole alanine	0.130	7.4	n.d.	-	0.059	4.1	n.d.	-
Other extractable components	0.132	7.5	0.099	8.1	0.107	7.3	0.147	11.4
Total Extracted	1.148	65.3	0.757	61.9	0.971	66.7	0.862	66.6
Flutriafol Extracted (1 N NaOH)[a]	0.093	5.3	1 N NaOH extraction not performed on this sample.		0.097	6.7	1 N NaOH extraction not performed on this sample	
Others Extracted (1 N NaOH)[a]	0.289	16.4			0.233	16.0		
PES[b]	0.228	13.0	0.465	38.1	0.155	10.6	0.433	33.4
TRR (Extracted+ PES)	1.758	100	1.222	100	1.456	100	1.295	100
Subtotal identified[c]	0.816	46.4	0.416	34.0	0.673	46.2	0.413	31.9
TRR by combustion (for comparison)	1.749 ± 0.127	-	1.129 ± 0.083	-	1.395 ± 0.057	-	1.220 ± 0.037	-

[a] Extracted from PES

[b] mg/kg after 1 N NaOH extraction

[c] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 30 Nature of the residue in wheat grain grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]triazole flutriafol	
	30-Day Plant Back		120-Day Plant Back	
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.009	1.3	0.003	0.5
1,2,4-Triazole	n.d.	-	n.d.	-
Triazole lactic acid	n.d.	-	n.d.	-
Triazole acetic acid	0.195	28.8	0.155	27.9
Triazole alanine	0.388	57.2	0.325	58.6
Other extractable components	0.010	1.5	0.019	3.4
Total Extracted	0.602	88.8	0.502	90.5
PES	0.076	11.2	0.053	9.5
TRR (Extracted+PES)	0.678	100	0.555	100
Subtotal identified[a]	0.592	87.3	0.483	87.0
TRR by combustion (for comparison)	0.648 ± 0.054	-	0.528 ± 0.024	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Study 3

A confined rotational crop study was conducted using [^{14}C] triazole and [^{14}C] carbinol flutriafol with lettuce, radish, and wheat at a 365 day plant-back interval (Dohn, 2009: 1622 FLU). This study was conducted as a companion study to Study 2. Two radiolabeled forms of flutriafol were used (see table below), and each test chemical was applied to two 1.39 m^2 test plots. The test plots were located outdoors, and consisted of wooden boxes filled with sandy loam soil to a depth of 0.46 m. Two test plots (one for each radiolabel) were used to grow wheat. The remaining two plots were used for

lettuce and radish, with the area of each plot equally divided between the two crops. The test substance was dissolved in ethanol and applied to test plots with hand operated pump sprayers. The target application rate was 25 mg/m^2 (250 g/ha). The rate actually applied to all plots was 260 g/ha (104% of target). Lettuce, radish roots and tops (foliage), and wheat forage, hay, straw, and grain were harvested. Crop samples were homogenized in the presence of dry ice using food processing equipment, and the total radioactive residues were measured by combustion analysis/LSC.

The TRRs in plant samples grown in [^{14}C] triazole flutriafol treated soil were consistently greater than the plant samples obtain from [^{14}C] carbinol flutriafol treated plots. The difference in TRR values observed in the wheat grain samples was quite large (40-fold). Radish tops had higher residues than the corresponding radish roots samples, indicating that flutriafol and its metabolites were translocated within the xylem of the plant tissue.

Table 31 Total radioactive residues (TRR) in 365 day rotational crops measured combustion analysis

Commodity	[^{14}C]triazole[a] (mg/kg)	[^{14}C]carbinol[a] (mg/kg)
Lettuce	0.123 ± 0.005	0.019 ± 0.002
Radish Tops	0.107 ± 0.003	0.071 ± 0.003
Radish Roots	0.059 ± 0.003	0.008 ± 0.001
Wheat Forage	0.075 ± 0.008	0.061 ± 0.002
Wheat Hay	0.191 ± 0.016	0.083 ± 0.005
Wheat Straw	0.798 ± 0.038	0.480 ± 0.013
Wheat Grain	0.440 ± 0.025	0.011 ± 0.001

[a] Values from combustion/LSC of five replicate sub-samples

The nature of the radioactive residues in the crop samples were extracted with acetonitrile/water and characterized by HPLC and TLC analyses. All crop samples grown in treated soil were extracted and analysed, except for the radish roots from the [^{14}C] carbinol flutriafol treatment. Flutriafol was detected in all crop samples (but present at very small concentrations in wheat grain and radish root < 0.01 mg/kg). The most significant metabolites in all rotational crops contained only the triazole label. These were triazole alanine that was present in all matrices, triazole acetic acid that was found in lettuce and all wheat matrices, and triazole lactic acid that was found in all crop samples except wheat grain. No free 1,2,4-triazole was found in any crop at any sampling interval. There were no metabolites detected in any crops that contained only the carbinol carbon.

Several metabolites present at small concentrations that contained both radiolabeled positions were detected in lettuce and the vegetative parts of wheat. These metabolites were characterized in the companion study (Study 2). One metabolite (M31) was unique to lettuce, and was present at small concentrations (0.002 to 0.006 mg/kg in the two 365 Day lettuce samples). Several metabolites containing both radiolabeled positions were detected in wheat hay and straw. One such metabolite, M28, was characterized as a glucose conjugate, with a corresponding aglycone more polar than flutriafol (as judged by reverse phase HPLC behavior). M28 was present in the 365 Day hay at 0.003 to 0.006 mg/kg, and in straw at 0.019 to 0.024 mg/kg. Wheat hay and straw also contained a two-label metabolite, M30 (subsequently shown to consist of two components), which was present at concentrations of 0.002 to 0.006 mg/kg in wheat hay, and 0.025 to 0.035 mg/kg in wheat straw. One component of M30 was shown to be a glucose conjugate, and the corresponding aglycone was distinct from the aglycone derived from M28.

Table 32 Nature of the residue in lettuce grown in flutriafol treated soil

Component	[^{14}C]triazole flutriafol		[^{14}C]triazole flutriafol	
	365-Day Plant Back			
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.021	16.5	0.015	75.0
M31	0.006	4.7	0.002	10.0
1,2,4-Triazole	n.d.	-	n.d.	-
Triazole lactic acid	0.058	45.7	n.d.	-
Triazole acetic acid	0.004	3.1	n.d.	-
Triazole alanine	0.020	15.7	n.d.	-

Component	[^{14}C]triazole flutriafol		[^{14}C]triazole flutriafol	
	365-Day Plant Back			
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Other Extractable Components	0.010	7.9	0.001	5.0
Total Extracted	0.119	93.7	0.018	90.0
PES	0.008	6.3	0.002	10.0
TRR (Extracted+PES)	0.127	100	0.020	100
Subtotal identified[a]	0.103	81.1	0.015	75.0
TRR by combustion (for comparison)	0.123 ± 0.005	-	0.019 ± 0.002	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid

n.d.: not detected

Table 33 Nature of the residue in radish grown in flutriafol treated soil

Component	Root		Tops			
	[^{14}C]triazole flutriafol		[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol	
	365-Day Plant Back					
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.006	11.5	0.057	52.3	0.056	84.8
1,2,4-Triazole	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.003	5.8	0.007	6.4	n.d.	-
Triazole acetic acid	n.d.	-	n.d.	-	n.d.	-
Triazole alanine	0.038	73.1	0.029	26.6	n.d.	-
Other extractable components	0.002	3.8	0.004	3.7	0.003	4.5
Total Extracted	0.049	94.2	0.097	89.0	0.059	89.4
PES	0.003	5.8	0.012	11.0	0.007	10.6
TRR (=Extracted+PES)	0.052	100.0	0.109	100.0	0.066	100.0
Subtotal identified[a]	0.047	90.4	0.093	85.3	0.056	84.8
TRR by combustion (for comparison)	0.059 ± 0.003	-	0.107 ± 0.003	-	0.071 ± 0.003	-

[a] Flutriafol + Triazole alanine + Triazole lactic acid.

n.d. = not detected

Table 34 Nature of the residue in wheat forage and hay grown in flutriafol treated soil

Component	Wheat forage				Wheat hay			
	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol	
	365-Day Plant Back							
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.029	37.7	0.044	75.9	0.018	9.9	0.038	47.5
M30	n.d.	-	0.006	10.3	0.002	1.1	0.006	7.5
M28	n.d.	-	n.d.	-	0.003	1.6	0.006	7.5
1,2,4-Triazole	n.d.	-	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.010	13.0	n.d.	-	0.043	23.6	n.d.	-
Triazole acetic acid	0.001	1.3	n.d.	-	0.051	28.0	n.d.	-
Triazole alanine	0.024	31.2	n.d.	-	0.029	15.9	n.d.	-
Other extractable components	0.007	9.1	n.d.	-	0.011	6.0	0.007	8.8
Total Extracted	0.071	92.2	0.050	86.2	0.157	86.3	0.057	71.3
PES	0.006	7.8	0.008	13.8	0.025	13.7	0.023	28.8
TRR (=Extracted+PES)	0.077	100.0	0.058	100.0	0.182	100.0	0.080	100.1
Subtotal identified[a]	0.064	83.1	0.044	75.9	0.141	77.5	0.038	47.5
TRR by combustion (for comparison)	0.075 ± 0.008	-	0.061 ± 0.002	-	0.191 ± 0.016	-	0.083 ± 0.005	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid.

n.d. = not detected

Table 35 Nature of the residue in wheat straw and grain grown in flutriafol treated soil

Component	Wheat straw				Wheat grain			
	[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol		[^{14}C]triazole flutriafol		[^{14}C]carbinol flutriafol	
	365-Day Plant Back							
	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR	Flutriafol Equivalents (mg/kg)	% TRR
Flutriafol	0.233	31.9	0.243	49.7	0.005	1.1	0.004	40.0
M30	0.025	3.4	0.035	7.2	n.d.	-	n.d.	-
M28	0.024	3.3	0.019	3.9	n.d.	-	n.d.	-
1,2,4-Triazole	n.d.	-	n.d.	-	n.d.	-	n.d.	-
Triazole lactic acid	0.161	22.0	n.d.	-	n.d.	-	n.d.	-
Triazole acetic acid	0.053	7.3	n.d.	-	0.203	45.7	n.d.	-
Triazole alanine	0.008	1.1	n.d.	-	0.136	30.6	n.d.	-
Other extractable components	0.063	8.6	0.060	12.3	0.030	6.8	0.003	30.0
Total Extracted	0.567	77.6	0.357	73.0	0.374	84.2	0.007	70.0
PES	0.164	22.4	0.132	27.0	0.070	15.8	0.003	30.0
TRR (=Extracted+PES)	0.731	100.0	0.489	100.0	0.444	100.0	0.010	100.0
Subtotal identified[a]	0.455	62.2	0.243	49.7	0.344	77.5	0.004	40.0
TRR by combustion (for comparison)	0.798 ± 0.038	-	0.480 ± 0.013	-	0.440 ± 0.025	-	0.011 ± 0.001	-

[a] Flutriafol + Triazole alanine + Triazole acetic acid + Triazole lactic acid.

n.d. = not detected

In summary, the results of the confined crop rotation studies show that following application of ^{14}C flutriafol at 260 g ai/ha, detectable radioactivity was found in lettuce, radish roots and tops, wheat forage, hay, straw and grain sown at 30, 120 and 365 days after application to soil. The majority of the extracted radioactivity was composed of parent flutriafol in most of commodities except wheat grain. Triazole lactic acid and triazole alanine were detected in various commodities, present at more than 10% TRR for any individual commodity. Triazole alanine was detected as a major compound in wheat grains, and triazole acetic acid was also found at > 10% TRR. Parent flutriafol, triazole alanine, triazole lactic acid and triazole acetic acid are the residue components in follow or succeeding crops.

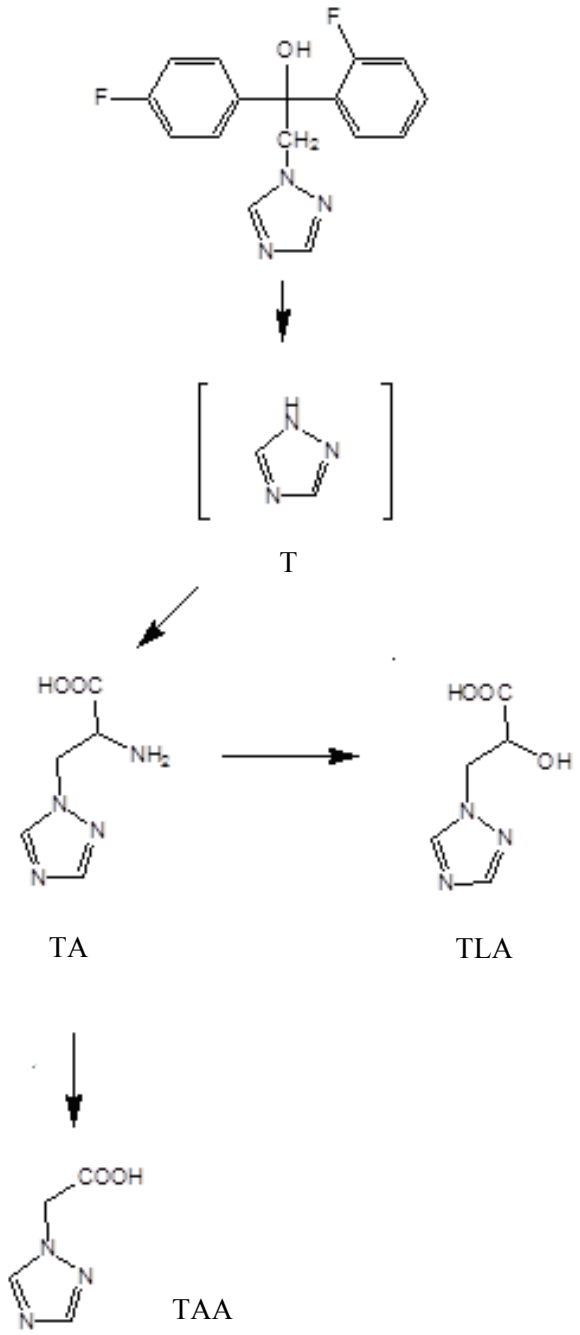


Figure 5 Proposed Metabolic Pathway of Flutriafol in Rotational Crops

Field rotational crop studies

Residue in the rotational crops was studied using the plots treated in the UK (Dick, 1988: 209 FLU). Flutriafol as a 125 g/L SC was initially applied to cereals, as a foliar spray at the four sites used for the study, in first year at 0.188 kg ai/ha and in each subsequent year at 0.25 kg ai/ha. After five consecutive years of application with a total rate applied of 1.188 kg/ha, rotational crops were sown/planted on the test sites in the sixth growth season and grown to maturity. Soil in which either sugar beet or fodder beet was grown was analysed for residues of flutriafol in two depth horizons 0–15 and 15–30 cm. Soil residues in the 0–15 cm horizon at each site ranged between 0.09–0.30 mg/kg and in the 15–30 cm horizon between < 0.01–0.13 mg/kg. These residues when expressed as kg ai/ha for the combined depths were equivalent to 0.41–0.55 kg ai/ha which represents 35–47% of the total flutriafol that had been applied over the previous years.

No residues (< 0.01 mg/kg) of flutriafol were found in the roots of sugar beet grown in plots at any site. Foliage samples of sugar beet contained low or non-detectable residues of flutriafol, < 0.01–0.07 mg/kg. Fodder beet roots from each of the sites contained low or non-detectable residues of flutriafol, < 0.01–0.02 mg/kg. Foliage samples of fodder beet also contained low residues, 0.01–0.08 mg/kg. Potatoes and carrots contained either non detectable or low residues of flutriafol, < 0.01 and 0.02 mg/kg respectively. Grain from the spring barley contained a low residue of flutriafol, 0.05 mg/kg. Residues in the spring barley straw samples were 0.38 mg/kg.

Table 36 Residues of flutriafol in rotational crops (Dick, 1988)

Site	Total of application rate, kg/ha	Soil concentration after 5 years, mg/kg	Crop	Crop part analysed	Residue of flutriafol, mg/kg
Benson West Horsley Boxford Stanton St John	1.188	0.35 0.22 0.31 0.30	Sugarbeet	Root	< 0.01 < 0.01 < 0.01 < 0.01
Benson West Horsley Boxford Stanton St John	1.188	0.35 0.22 0.31 0.30	Sugarbeet	Foliage	not analysed 0.07 0.04 < 0.01
Benson West Horsley Boxford Stanton St John	1.188	0.30 0.21 0.20 0.30	Fodderbeet	Root	< 0.01 0.02 < 0.01 0.01
Benson West Horsley Boxford Stanton St John	1.188	0.30 0.21 0.20 0.30	Fodderbeet	Foliage	0.02 0.08 0.02 0.01
Benson	1.188	0.35	Potatoes	Tuber	< 0.01
			Carrots	Root	0.01[C] 0.02
			Spring Barley	Grain	0.03[C] 0.05
			Spring Barley	Straw	0.02[C] 0.38

[C]: control sample

The field studies were carried out using the plots treated at the highest rate (4.0 kg ai/ha) in three trial site in the UK (Wheals, 1993: 83, 84, 453 FLU). The field studies were designed to provide samples of rotational crops grown in soil with artificially high levels of aged flutriafol residues, close to or in excess of the maximum predicted concentration in soil after 15 years continuous application of the maximum label rate (0.25 kg ai/ha). The soil at the trial sites used had been treated in 1988 with a single application of flutriafol at a nominal rate of 4.0 kg ai/ha, and when sampled in 1990 had been found to contain flutriafol residues of between 0.13 and 4.0 mg/kg equivalent to 0.42 kg ai/ha to 3.9 kg ai/ha. The rotational crops were sown in 1991 and grown to maturity. Representative samples of the crops were taken at harvest and analysed for residues of flutriafol and its major metabolites triazole alanine and triazole acetic acid. The results of the studies are summarized in Table 37.

Table 37 Residues of flutriafol in rotational crops (Wheals, 1993)

Site	Soil concentration of flutriafol after 2 years, mg/kg	Crop (Variety)	Crop part analysed	Residue, mg/kg		
				Flutriafol	Triazole alanine	Triazole acetic acid
Berkshire Berkshire Suffolk	0.76 0.67 0.23	Potato (Maris Piper)	Tuber	< 0.05 < 0.05 < 0.05	0.09 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05

Site	Soil concentration of flutriafol after 2 years, mg/kg	Crop (Variety)	Crop part analysed	Residue, mg/kg		
				Flutriafol	Triazole alanine	Triazole acetic acid
Berkshire Berkshire Suffolk	0.60 0.53 0.13	Sunflower (Naindor)	Seed	< 0.05 < 0.05 < 0.05	0.06 0.33 0.22	0.09 0.35 0.14
Berkshire Berkshire	0.82 0.66	Maize (Sonia)	Grain	< 0.05 < 0.05	0.14 0.18	< 0.05 < 0.05
Berkshire Berkshire	0.82 0.66		Straw	0.31 0.16	< 0.05 < 0.05	< 0.05 < 0.05
Lincolnshire Warwickshire	2.0 0.68	Spring barley (Digger)	Grain	< 0.03 0.07	0.08 0.22	< 0.05 < 0.05
Lincolnshire Warwickshire	2.0 0.68		Straw	0.24 1.5	< 0.05 < 0.05	<0.1 <0.1
Lincolnshire Warwickshire Suffolk	2.0 0.68 0.35	Spring wheat (Axona)	Grain	< 0.03 0.03 0.03	0.28 0.67 3.0	< 0.05 0.14 0.84
Lincolnshire Warwickshire Suffolk	2.0 0.68 0.35		Straw	0.29 2.5 2.1	< 0.05 < 0.05 < 0.05	<0.1 <0.1 0.25
Somerset Lincolnshire Suffolk	4.0 2.0 0.32	Oilseed rape (Puma)	Seed	< 0.05 < 0.05 < 0.05	6.6 0.59 17	< 0.05 < 0.05 0.11
Lincolnshire Cambridgeshire Suffolk	1.0 0.63 0.31	Pea (Countess)	Seed	< 0.05 < 0.05 < 0.05	0.15 0.47 7.7	< 0.05 < 0.05 < 0.05
Lincolnshire Cambridgeshire Suffolk	1.0 0.63 0.31		Haulm	0.28 3.8 3.8	< 0.05 < 0.05 0.22	< 0.05 < 0.05 0.13
Somerset Warwickshire Cambridgeshire	3.0 0.68 0.63	Cabbage (Stonehead)	Head	< 0.05 0.12 0.05	< 0.05 0.32 0.05	< 0.05 < 0.05 < 0.05
Somerset Lincolnshire Cambridgeshire	2.8 1.0 0.63	Carrot (Primo)	Root	< 0.05 < 0.05 0.13	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05
Stockwith Dorrington Balsham	2.0 0.92 0.56	Sugarbeet	Root	< 0.01 0.01 0.03	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05
Stockwith Dorrington Balsham	2.0 0.92 0.56		Foliage	0.03 0.04 0.41	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05

Environmental fate in water systems

Hydrolysis

The hydrolytic stability of ^{14}C-flutriafol was investigated in various buffer solutions at 25 °C in the dark for up to 30 days (Hawkins, 1987: 213 FLU). The buffer solutions were at pH 5.0, 7.0 and 9.0 and were sterilized prior to addition of the ^{14}C-flutriafol. ^{14}C-flutriafol (2.03 mg) was dissolved in acetonitrile (6.8 mL) and sterilised by passing through a 0.2 μm millipore filter into a sterile flask. A 94 μL aliquot of this solution was added to each flask to give a nominal ^{14}C-flutriafol concentration in the test solution of 1.0 μg/mL. Duplicate samples were taken immediately after application and at 2, 8, 16 and 30 days. The radioactivity present was quantified by LSC. Aliquots of the test solutions were directly analysed by TLC.

Table 38 Hydrolytic degradation of ^{14}C-Flutriafol in sterile solutions buffered at pH 5, 7 and 9 at 25 °C

pH	Incubation time (days)	Flutriafol	
		µg/mL	% of applied radioactivity
5	0	0.91, 0.90	97.2, 96.2
	30	0.89, 0.93	93.4, 97.4
7	0	0.92, 0.92	96.8, 96.4
	30	0.92, 0.92	95.9, 97.1
9	0	0.90, 0.91	96.1, 96.0
	30	0.93, 0.93	96.9, 98.0

Recoveries of radioactivity in the test solutions at the three different pH values were 98.7–99.4% of the applied radioactivity at zero-time. At the later analysis times, 95.4–101.4% of the applied radioactivity was recovered from the respective buffer solutions. Concentrations of ^{14}C-flutriafol were 0.89–0.93 µg equivalents/mL buffer for all samples. TLC, followed by radioscanning of the zero time and Day 30 samples showed that 93.4–98.0% of the radioactivity in the buffer solutions co-chromatographed with flutriafol.

Since no discrete radioactive components were found in the Day 30 samples, other than those found in zero time samples, no further analysis was carried out on intermediate test solutions. ^{14}C-flutriafol is hydrolytically stable in buffered solutions at pH values of 5, 7 and 9 at 25 □C in the dark for periods up to 30 days.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Descriptions of analytical methods together with validation data for residues of flutriafol in plant, animal and soil matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetonitrile/water. After solvent partition cleanup, the flutriafol residues are prepared for gas chromatography or undergo further cleanup prior to GC or LC analysis. Flutriafol residues can be measured either by nitrogen-phosphorous (NPD) or mass selective detectors (MSD), typically to an LOQ of 0.01 mg/kg. Since the methods use standard extraction solvents and standard detection techniques, they have the potential to be incorporated into existing multi-residue methods.

Analytical methods together with validation data for residues of triazole metabolites in plant, animal matrices were also submitted to the Meeting. Samples were usually extracted with methanol/water. The aliquots of extract were processed through SPE cleanup and/or derivation steps. Determination for derivatives of triazole metabolites (1,2,4-triazole, triazole alanine and triazole acetic acid) were conducted using HPLC with mass spectrometric detector (MS/MS). The LOQ for the metabolites were 0.01 mg/kg.

Detailed descriptions of all these analytical methods are presented below.

Plant matrices

Apple (1329 FLU)

Analyte:	Flutriafol	LC-MS-MS
LOQ:	0.01 mg/kg	
Description	Apple samples are extracted with acetonitrile/water (1:1). An aliquot of the homogenate is filtered over single-use filter (0.45 µm). Residues of flutriafol in apple are determined by HPLC equipped with a. mass spectrometry/mass spectrometry (MS/MS) detector.	

Apple (fruit, juice and wet pomace) (1471 FLU)

Analyte:	Flutriafol	GC-MSD
LOQ:	0.01 mg/kg	

Description Samples are extracted with acetonitrile/water (70:30 v/v). An aliquot of the extract is diluted with 0.5 M sodium sulphate solution and portioned with toluene. The residue is cleaned-up with a silica SPE and BE-NH_2 SPE column. Residues of flutriafol in apple fruit, juice and wet pomace are determined and quantified using gas chromatography employing mass selective (MSD) detection.

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ: 0.01 mg/kg

Description Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 1.0 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot is purified through a Bond Elut Certify II SPE cartridge, then undergo two derivatizations; the first an esterification using HCl/butanol and the second an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot was directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Grapes (1606 FLU)

Analyte: Flutriafol LC-MS-MS

LOQ: 0.01 mg/kg

Description Samples are extracted with acetonitrile/water (70:30 v/v). A portion of the extract is diluted with 0.5 M sodium sulphate solution and portioned with toluene. The residue is cleaned-up with a silica SPE column. Residues of flutriafol in grape fruit, juice and raisins are determined and quantified using gas chromatography employing mass selective (MSD) detection.

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ: 0.01 mg/kg

Description Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 1.0 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot undergo two derivatizations directly; the first an esterification using HCl/butanol and the second an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot was directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Banana (1626 FLU)

Analyte: Flutriafol GC-MSD

LOQ: 0.01 mg/kg

Description: Residues of flutriafol are extracted from the sample matrix by maceration with acetonitrile/water (70:30, v/v). An aliquot of the extract is diluted with 0.5 M sodium sulphate solution and portioned with toluene. The residues undergo a silica SPE cleanup, followed by stacked ENVI-Carb/BE-NH_2 SPE cleanup. Determination and quantitation of flutriafol are conducted using gas chromatography employing mass selective (MSD) detection.

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ: 0.01 mg/kg

Description Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 1.0 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot is purified through a Bond Elut Certify II SPE cartridge, then undergo two derivatizations; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Sweet pepper (1264 FLU)

Analyte: Flutriafol LC-MS-MS

LOQ: 0.01 mg/kg

Description: Samples are extracted with acetonitrile and after filtering, the sample is diluted with acetonitrile/water (50:50, v/v). The diluted extract is analysed directly by HPLC with mass spectrometric (MS/MS) detection.

Soya bean (1468 FLU)

Analyte: Flutriafol GC-NPD

LOQ: 0.01 mg/kg for soya bean seed hulls and refined oil, 0.05 mg/kg for soya bean meal, 0.5 mg/kg for AGF

Description: Soya beans are extracted with a mixture of acetonitrile/water (70:30, v/v). A portion of the extract is diluted with 0.5 M sodium sulphate solution and portioned with toluene. The residues undergo further clean-up through a silica SPE chromatography. Determination and quantitation of flutriafol are conducted using gas chromatography equipped with nitrogen/phosphorus detector (NPD).

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ: 0.01 mg/kg

Description: Soya beans are extracted with methanol/water (80:20 v/v). For soya bean refined oil, the matrix is diluted with hexane. The aqueous methanolic extracts are brought to a final volume with methanol/water (80:20 v/v). Individual aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot undergoes two derivatizations directly; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Wheat (1811 FLU)

Analyte: Flutriafol GC-NPD or MSD

LOQ: 0.01 mg/kg

Description: Residues of flutriafol are extracted by maceration with acetonitrile/water (70:30). An aliquot of the extract is diluted with 0.5 M sodium sulphate solution and flutriafol residues are extracted with toluene. The toluene extract is evaporated and the extract is then subjected to a silica solid phase clean up prior to analysis by gas chromatography using a nitrogen-phosphorous detector (NPD) or mass selective detector (MSD).

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ:

Description: Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 1.0 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot is purified through a Bond Elut Certify II SPE cartridge, then undergo two derivatizations; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-triazole. The derivative is partitioned into ethyl acetate which is evaporated to dryness, then dissolved in acetonitrile/water (30:70, v/v). Determination and quantitation for derivatives of 1,2,4-triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Wheat (1047 FLU)

Analyte: Flutriafol LC-MS-MS

LOQ: 0.01 mg/kg

Description: Samples are extracted with acetonitrile and after evaporating off the solvent, the sample is cleaned up with the Envi-Carb and primary amino phase columns, using ethyl acetate as eluents. The eluate is evaporated to dryness and the residue taken up in HPLC mobile phase (acetonitrile/water/formic acid (50:50:0.2, v/v)) for analysis using HPLC with mass spectrometric (MS/MS) detection.

Peanut (1605 FLU)

Analyte:	Flutriafol	GC-MSD	
LOQ:	0.01 mg/kg for peanut nutmeat, hay, meal and refined oil		
Description:	Homogenized peanut nutmeat, hay and meal samples are extracted with acetonitrile/water (70:30, v/v), using a single extraction. Peanut oil are extracted with acetonitrile/water (70:30, v/v), using multiple extraction. In both cases, an aliquot of the extract is diluted with 0.5 M sodium sulphate solution and partitioned with toluene. The residue is cleaned-up with a silica SPE column. Residues of flutriafol in samples are determined and quantified using gas chromatography employing mass selective (MSD) detection.		
Analyte:	Triazole metabolites	LC-MS-MS	Method: Meth-160
LOQ:	0.01 mg/kg for peanut nutmeat, hay, meal and refined oil		
Description:	Samples are extracted with methanol/water (80:20 v/v). The aqueous methanolic extracts are brought to a final volume with methanol/water (80:20 v/v). Individual aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot undergoes two derivatizations directly; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.		

Coffee beans (975 FLU, Analytical Report)

Analyte:	Flutriafol	GC-MSD
LOQ:	0.05 mg/kg	
Description:	Samples are extracted with acetonitrile/water (70:30), and then partitioned with toluene. After concentration by evaporation, the purification proceeds by a silica solid phase extraction, where the elution is done with acetone/hexane (40:60). The extracts is concentrated by evaporation and suspended by acetone. The quantitative determination is done by gas chromatography using a mass selective detector.	

Coffee (green coffee beans, cleaned green beans, roasted beans, spent grounds, liquor extract and instant coffee)

Analyte:	Flutriafol	GC-MSD, LC-MS-MS
LOQ:	0.01 mg/kg for green coffee beans, cleaned green beans, roasted beans, spent grounds, liquor extract and instant coffee	
Description:	Homogenized samples are extracted with acetonitrile/water (70:30, v/v), and an aliquot of the extract is diluted with 0.5 M sodium sulphate solution and then partitioned with toluene. After concentration by evaporation, the purification proceeds by a silica solid phase extraction, where the elution is done with acetone/hexane (15:85). The extracts is concentrated by evaporation and suspended by acetone or acetonitrile/water (50:50). The quantitative determination is done by gas chromatography using a mass selective detector or HPLC with mass spectrometric (MS/MS) detection..	
Analyte:	Triazole metabolites	LC-MS-MS
LOQ:	0.01 mg/kg	
Description:	Samples are extracted with methanol/water (80:20 v/v). The aqueous methanolic extracts are brought to a final volume with methanol/water (80:20 v/v). Individual aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot undergoes two derivatizations directly; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.	

Method DFG S 19

Apple, grapes (1294 FLU, 1299 FLU)

Analyte:	Flutriafol	LC-MS-MS	Agrisearch Method Flutriafol/Crops/DB/04/1
LOQ:	0.01 mg/kg		

Description Apple is extracted with acetone/water followed by partition with ethyl acetate/cyclohexane. The extracts were further purified by gel permeation chromatography. The eluate is concentrated, and residues of flutriafol are determined by liquid chromatography equipped with a mass spectrometry/mass spectrometry (MS/MS) detector.

This same method was used for determination of flutriafol in grapes (Flutriafol/Crops/DMK/03/1)

Soya bean (978 FLU, 979 FLU, 980 FLU/ Analytical report)

Analyte: Flutriafol GC-NPD Method: DFG S19

LOQ: 0.01 mg/kg

Description: 50 g sample is weighed and acetone/dichloromethane (1:1) is added prior to homogenization of samples. The extract is partitioned with dichloromethane and anhydrous sodium sulphate is then added for the complete removal of water. An aliquot of the organic phase is cleaned up by gel permeation chromatography using a mixture of ethyl acetate/cyclohexane as eluents. The residue containing fraction is concentrated and added isooctane. The aliquot is purified through a silica gel column. The eluate is analysed by gas chromatography with nitrogen/phosphorous detector (NPD).

Coffee beans (989 FLU)

Analyte: Flutriafol GC-TSD Method: DFG S19

LOQ: 0.05 mg/kg

Description 50 g sample is weighed and acetone/dichloromethane (1:1) is added prior to homogenization of samples. The extract is partitioned with dichloromethane and anhydrous sodium sulphate is then added for the complete removal of water. An aliquot of the organic phase is cleaned up by gel permeation chromatography using a mixture of ethyl acetate/cyclohexane as eluents. The residue containing fraction is concentrated and added isooctane. The aliquot is purified through a silica gel column. The eluate is analysed by gas chromatography equipped with a specific thermoionic detector (TSD).

Validation data for methods on plant matrices are summarized in Table 39 and 40.

Table 39 Summary of Method Validation Data for flutriafol fortified into plant matrices

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Plant commodities							
Apple	0.01 1.0	5	102–106 98–103	104 100	1 2		1329 FLU
Apple	0.01 0.5 10	7 3 3	103–114 89–91 75–81	97	14		1471 FLU
Apple, juice	0.01 0.5 10	7 3 3	79–89 70–83 90–93	85	6.5		
Grapes	0.01 0.10	2 2	92, 97 93, 95	94	2.2		1606 FLU
Grape, juice	0.01 0.10	2 2	84, 92 99, 100	94	7.4		
Grape, raisins	0.01 0.10	2 2	104, 111 98, 107	105	5.5		
Banana	0.01 0.5 10.0	7 3 3	89–106 91, 93,94 90,95, 97	97 93 94	5.3 1.5 3.6		1626 FLU
Soya bean, seed	0.01 0.10	2 2	115, 117 101, 101	109	8.0		1468 FLU
Soya bean, meal	0.05 0.10	2 2	114, 115 93, 101	106	10.4		
Soya bean, hulls	0.01 0.10	2 2	76, 119 88, 89	93	19		
Soya bean, refined oil	0.01 0.05 0.10	2 2 2	70, 105 108, 138 113, 116	108	20		

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Soya bean, AGF	0.10 0.50 5.0	2 2 2	78, 101 114, 116 106, 117	105	14		
Sweet peppers	0.01 0.1	5 5	83–90 86–99	86 92	3 6		1264 FLU
Wheat, forage	0.01 0.5 1.0 5.0 15 25	10 5 1 2 1 1	77–117 89–104 99 113, 116 108 102	102	11.0		1811 FLU
Wheat, hay	0.01 0.5 1.0 5.0 15	9 6 1 2 1	75–107 97–113 88 89–95 83	96	9.9		
Wheat, straw	0.01 0.5 1.0 5.0 10	8 4 1 3 1	83–106 75–93 88 83–94 86	90	8.1		
Wheat, grain	0.01 0.5 5.0	10 8 2	77–119 78–100 108, 110	96	11		
Wheat, AGF	0.01 5.0	1 1	93 106	100			
Wheat, bran	0.01 5.0	1 1	88 88	88			
Wheat, flour	0.01 5.0	1 1	86 93	90			
Wheat, middlings	0.01 5.0	1 1	88 88	88			
Wheat, shorts	0.01 5.0	1 1	88 93	90			
Wheat, germ	0.01 5.0	1 1	103 106	104			
Wheat, whole plant	0.01 0.10 1.0 5.0	6 7 2 2	75–90 76–98 89, 95 85, 95	86	8		1047 FLU
Wheat, grain	0.01 0.10	6 5	80–87 80–90	84	4		
Wheat, straw	0.01 0.10 0.50 2.0	5 5 2 2	85–97 80–91 81, 100 81, 95	88	7		
Wheat, residual plant	0.01 0.10	1 1	91 79	85			
Wheat, ears	0.01 0.10	1 1	91 89	90			
Peanut, nutmeat	0.01 0.10	2 2	105, 105 105, 108	106	1.5		1605 FLU
Peanut, hay	0.01 0.10	2 2	99, 108 100, 98	101	4.6		
Peanut, meal	0.01 0.10	2 2	112, 108 102, 103	106	4.6		
Peanut, refined oil	0.01 0.10	2 2	88, 83 88, 90	87	3.0		
Coffee beans	0.05 0.50	4 3	81–95 83, 86, 96	87 88	6 6		975 FLU
Coffee beans	0.01 1.0	5 5	109–120 94–108	115 99	3.9 5.8		

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Coffee beans, roasted	0.01 1.0	5 5	71–120 78–95	84 88	24 7.6		
Coffee liquor extract	0.01 1.0	5 5	96–111 93–100	104 96	6.2 3.2		
Instant coffee	0.01 1.0	5 5	97–114 95–112	109 105	6.2 6.6		
Multi-residue method							
Apple	0.01 1.0	9	61–101 74–97	76 85	17.6 9.1	DB/04/1	1294 FLU
Grapes	0.01 1.0	8 9	61–103 65–107	81 89	19.0 17.2	DB/04/1	1299 FLU
Soya bean	0.05 1.00	3 3	91, 101, 109 103, 109, 112	101 108	8.9 4.4	DFG S19	978 FLU
Coffee beans	0.05 0.50	3 3	87, 87, 90 80, 97, 114	92	13.0	DFG S19	989 FLU

Table 40 Summary of Method Validation Data for triazole metabolites fortified into plant matrices

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Apple 1,2,4-triazole	0.01 0.5	3 3	91–93 86–92	90	3.1	1471 FLU
Apple Triazole alanine	0.01 0.5	3 3	89–93 98–103	96	5.4	1471 FLU
Apple Triazole acetic acid	0.01 0.5	3 3	98–102 102–105	101	2.8	1471 FLU
Apple, juice 1,2,4-triazole	0.01 0.5	3 3	87–90 92	90	2.2	1471 FLU
Apple, juice Triazole alanine	0.01 0.5	3 3	86–91 93–98	92	4.6	1471 FLU
Apple, juice Triazole acetic acid	0.01 0.5	3 3	106–110 111–114	110	2.9	1471 FLU
Grapes 1,2,4-triazole	0.01 0.10	2 2	81, 89 88, 89	87	3.9	1606 FLU
Grapes Triazole alanine	0.01 0.10	2 2	81, 92 90, 96	90	6.3	1606 FLU
Grapes Triazole acetic acid	0.01 0.10	2 2	85, 86 78, 79	82	4.1	1606 FLU
Grape, juice 1,2,4-triazole	0.01 0.10	2 2	76, 87 84, 94	85	7.5	1606 FLU
Grape, juice Triazole alanine	0.01 0.10	2 2	85, 89 90, 95	90	4.1	1606 FLU
Grape, juice Triazole acetic acid	0.01 0.10	2 2	70, 80 78, 87	79	7.0	1606 FLU
Raisins 1,2,4-triazole	0.01 0.10	2 2	88, 90 84, 84	86	3.0	1606 FLU
Raisins Triazole alanine	0.01 0.10	2 2	88, 98 90, 90	92	4.4	1606 FLU
Raisins Triazole acetic acid	0.01 0.10	2 2	110, 115 79, 85	97	18	1606 FLU
Banana 1,2,4-triazole	0.01 0.5	3 3	75, 76, 92 92, 92, 97	81 94	9.5 2.9	1626 FLU
Banana Triazole alanine	0.02 0.5	3 3	75, 78, 79 92, 92, 93	77 92	2.1 0.58	1626 FLU
Banana Triazole acetic acid	0.02 0.5	3 3	80, 84, 87 90, 92, 93	84 92	3.5 1.5	1626 FLU
Soya bean, seed 1,2,4-triazole	0.01 0.1 0.5	10 9 1	81–100 76–94 95	90	6.1	1468 FLU

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Soya bean, seed Triazole alanine	0.1 0.3 0.5 1.0 1.8	4 7 3 6 1	91–99 89–101 92, 92, 94 87–98 95	94	3.6	1468 FLU
Soya bean, seed Triazole acetic acid	0.01 0.05 0.1 0.5	7 3 9 1	90–113 109, 109, 112 102–109 111	105	5.7	1468 FLU
Soya bean, meal 1,2,4-triazole	0.01 0.1	1 1	80 88	84		1468 FLU
Soya bean, meal Triazole alanine	0.5 1.0	1 1	88 83	86		1468 FLU
Soya bean, meal Triazole acetic acid	0.05 0.5	1 1	99 95	97		1468 FLU
Soya bean, hulls 1,2,4-triazole	0.01 0.1	1 1	79 93	86		1468 FLU
Soya bean, hulls Triazole alanine	0.1 1.0	1 1	73 89	81		1468 FLU
Soya bean, hulls Triazole acetic acid	0.02 0.2	1 1	100 101	100		1468 FLU
Soya bean, refined oil 1,2,4-triazole	0.01 0.1	1 1	87 90	88		1468 FLU
Soya bean, refined oil Triazole alanine	0.01 0.1	1 1	106 102	104		1468 FLU
Soya bean, refined oil Triazole acetic acid	0.01 0.1	1 1	103 107	105		1468 FLU
Soya bean, AGF 1,2,4-triazole	0.01 0.1	1 1	89 90	90		1468 FLU
Soya bean, AGF Triazole alanine	0.2 1.0	1 1	94 97	96		1468 FLU
Soya bean, AGF Triazole acetic acid	0.1 1.0	1 1	107 105	106		1468 FLU
Peanut, nutmeat 1,2,4-triazole	0.01 0.1	2 2	88,86 84,85	86	1.7	1605 FLU
Peanut, nutmeat Triazole alanine	0.04 0.4	2 2	84, 80 84, 81	82	2.1	1605 FLU
Peanut, nutmeat Triazole acetic acid	0.01 0.1	2 2	92, 87 88, 86	88	2.6	1605 FLU
Peanut, hay 1,2,4-triazole	0.01 0.1	2 2	95, 71 84, 85	84	9.8	1605 FLU
Peanut, hay Triazole alanine	0.02 0.2	2 2	81, 75 74, 73	76	3.6	1605 FLU
Peanut, hay Triazole acetic acid	0.01 0.1	2 2	72, 78 81, 87	80	6.2	1605 FLU
Peanut, meal 1,2,4-triazole	0.01 0.1	2 2	94, 80 84, 87	86	5.9	1605 FLU
Peanut, meal Triazole alanine	0.2 2.0	2 2	85, 96 88, 92	90	4.8	1605 FLU
Peanut, meal Triazole acetic acid	0.01 0.1	2 2	82, 73 83, 83	80	4.9	1605 FLU
Peanut, refined oil 1,2,4-triazole	0.01 0.1	2 2	81, 82 95, 86	86	6.4	1605 FLU
Peanut, refined oil Triazole alanine	0.01 0.1	2 2	78, 95 108, 108	97	14	1605 FLU
Peanut, refined oil Triazole acetic acid	0.01 0.1	2 2	94, 100 94, 95	96	2.9	1605 FLU
Wheat, forage 1,2,4-triazole	0.01 0.5	10 10	71–92 79–92	84	5.9	1811 FLU
Wheat, forage Triazole alanine	0.01 0.5	10 11	75–99 84–109	92	7.7	1811 FLU
Wheat, forage Triazole acetic acid	0.01 0.5	10 10	76–108 74–97	87	8.6	1811 FLU

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Wheat, hay 1,2,4-triazole	0.01 0.5	9 9	72–95 79–100	83	7.9	1811 FLU
Wheat, hay Triazole alanine	0.01 0.5	9 9	77–106 77–97	90	9.1	
Wheat, hay Triazole acetic acid	0.01 0.5	9 9	75–119 78–100	95	11	1811 FLU
Wheat, straw 1,2,4-triazole	0.01 0.5	9 9	71–94 76–90	82	6.5	1811 FLU
Wheat, straw Triazole alanine	0.01 0.5	9 9	74–102 70–95	86	8.4	1811 FLU
Wheat, straw Triazole acetic acid	0.01 0.5	9 9	75–114 82–98	89	8.6	1811 FLU
Wheat, grain 1,2,4-triazole	0.01 0.5	10 10	77–100 79–107	89	8.2	1811 FLU
Wheat, grain Triazole alanine	0.01 0.5 0.8 1.2	10 6 5 1	74–117 71–95 84–103 70	90	14	1811 FLU
Wheat, grain Triazole acetic acid	0.01 0.5	10 10	73–116 70 - 95	88	12	1811 FLU
Wheat, AGF 1,2,4-triazole	0.01 0.5	1 1	103 86	94		1811 FLU
Wheat, AGF Triazole alanine	0.06 0.5	1 1	70 76	73		1811 FLU
Wheat, AGF Triazole acetic acid	0.06 0.5	1 1	89 92	90		1811 FLU
Wheat, bran 1,2,4-triazole	0.01 0.5	1 1	85 78	82		1811 FLU
Wheat, bran Triazole alanine	0.2 0.8 3.4	1 1 1	70 77 71	73	3.8	1811 FLU
Wheat, bran Triazole acetic acid	0.04 0.5	1 1	89 87	88		1811 FLU
Wheat, flour 1,2,4-triazole	0.01 0.5	1 1	72 85	78		1811 FLU
Wheat, flour Triazole alanine	0.03 0.5	1 1	85 81	83		1811 FLU
Wheat, flour Triazole acetic acid	0.03 0.5	1 1	75 79	77		1811 FLU
Wheat, middlings 1,2,4-triazole	0.01 0.5	1 1	91 88	90		1811 FLU
Wheat, middlings Triazole alanine	0.08 0.5 1.0	1 1 1	86 78 74	79	6.1	1811 FLU
Wheat, middlings Triazole acetic acid	0.04 0.5	1 1	88 88	88		1811 FLU
Wheat, shorts 1,2,4-triazole	0.01 0.5	1 1	84 86	85		1811 FLU
Wheat, shorts Triazole alanine	0.2 0.8 1.8	1 1 1	70 73 78	74	4.0	1811 FLU
Wheat, shorts Triazole acetic acid	0.04 0.5	1 1	79 77	78		1811 FLU
Wheat, germ 1,2,4-triazole	0.01 0.5	1 1	92 89	90		1811 FLU
Wheat, germ Triazole alanine	0.2 0.8 3.4	1 1 1	79 83 73	78	5.0	1811 FLU
Wheat, germ Triazole acetic acid	0.04 0.5	1 1	84 91	88		1811 FLU
Coffee beans 1,2,4-triazole	0.01 1.0	5 5	100–115 102–104	109 103	5.6 0.97	

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Coffee beans	0.03	5	79–95	87	6.9	
Triazole alanine	1.0	5	87–93	90	2.7	
Coffee beans	0.01	5	91–100	95	3.5	
Triazole acetic acid	1.0	5	102–106	104	1.7	
Coffee beans, roasted	0.025	5	72–93	83	12	
1,2,4-triazole	2.5	5	80–91	86	4.8	
Coffee beans, roasted	0.01	5	98–114	106	6.2	
Triazole alanine	1.0	5	91–101	97	3.9	
Coffee beans, roasted	0.01	5	99–110	105	4.4	
Triazole acetic acid	1.0	5	99–110	107	4.3	
Coffee liquor extract	0.035	5	86–98	91	5.1	
1,2,4-triazole	3.5	5	95–110	104	5.4	
Coffee liquor extract	0.01	5	90–116	106	10	
Triazole alanine	1.0	5	85–96	89	4.5	
Coffee liquor extract	0.01	5	75–116	98	15	
Triazole acetic acid	1.0	5	105–114	110	3.7	
Instant coffee	0.05	5	71–87	81	7.9	
1,2,4-triazole	5.0	5	90–97	94	2.8	
Instant coffee	0.01	5	75–108	98	14	
Triazole alanine	1.0	5	76–80	79	1.9	
Instant coffee	0.01	5	99–108	104	3.9	
Triazole acetic acid	1.0	5	105–115	110	3.3	

Animal matrices

Bovine liver, kidney and fat (1566 FLU)

Analyte:	Flutriafol	GC-MSD	Method: ICIA AM00306
LOQ:	0.01 mg/kg		
Description	Residues of flutriafol are extracted by homogenization with acetonitrile. Fat samples are first heated in a water bath, dissolved in hexane (acetonitrile-saturated), and then extracted with acetonitrile. Aliquots of the acetonitrile extracts are evaporated to dryness, reconstituted in dichloromethane, and subjected to gel permeation chromatographic (GPC) cleanup. The GPC eluates are evaporated to dryness and reconstituted in a suitable volume of acetone for gas chromatographic analysis. Determination and quantification of flutriafol are conducted using gas chromatography employing mass selective (MSD) detection.		
Analyte:	Triazole metabolites	LC-MS-MS	Method: Meth-160
LOQ:	0.01 mg/kg		
Description	Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 0.5 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot is purified through a Bond Elut Certify II SPE cartridge, then undergo two derivatizations; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, Triazole alanine and Triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.		

Milk and muscle (1566 FLU)

Analyte:	Flutriafol	GC-MSD	Method: ICIA AM00306
LOQ:	0.01 mg/kg		

Description Residues of flutriafol are extracted from milk by homogenization with acetonitrile/dichloromethane and from muscle by homogenization with dichloromethane. A water partition of the dichloromethane/acetonitrile extracts is conducted to first remove unwanted water-soluble co-extractives. The water is adjusted to pH 9 prior to liquid/liquid partition. The resulting dichloromethane phase then underwent further cleanup using the GPC. The GPC eluates are evaporated to dryness and reconstituted in a suitable volume of acetone for gas chromatographic analysis. Determination and quantification of flutriafol are conducted using gas chromatography employing mass selective (MSD) detection.

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ: 0.01 mg/kg

Description Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 0.5 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot is purified through a Bond Elut Certify II SPE cartridge, then undergo two derivatizations; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Triazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, Triazole alanine and Triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Chicken eggs, muscle and fat (1567 FLU)

Analyte: Flutriafol GC-MSD Method: ICIA AM00306

LOQ: 0.01 mg/kg

Description Residues of flutriafol are extracted from chicken eggs and muscle by homogenization with acetonitrile. Fat samples are first heated in a water bath, dissolved in hexane (acetonitrile-saturated), and then extracted with acetonitrile. A water partition of the dichloromethane/acetonitrile extracts is conducted to first remove unwanted water-soluble co-extractives. For egg and muscle samples, the water is adjusted to pH 9 prior to liquid/liquid partition. The resulting dichloromethane phase then undergoes further cleanup using the GPC. The GPC eluates are evaporated to dryness and reconstituted in a suitable volume of acetone for gas chromatographic analysis. Determination and quantification of flutriafol are conducted using gas chromatography employing mass selective (MSD) detection.

Analyte: Triazole metabolites LC-MS-MS Method: Meth-160

LOQ: 0.01 mg/kg

Description Samples are extracted with methanol/water (80:20 v/v). The extract is brought to a final volume of 100 mL with methanol/water (80:20 v/v). Individual 0.5 mL aliquots of each extract are processed separately through SPE cleanup and/or derivatization steps, which are specific for each metabolite. The Triazole acetic acid aliquot is purified through a C-18 SPE cartridge, then derivatized using HCl/butanol esterification. The Triazole alanine aliquot is purified through a Bond Elut Certify II SPE cartridge, then undergoes two derivatizations; the first is an esterification using HCl/butanol and the second is an acylation using heptafluorobutyric anhydride. The 1,2,4-Ttiazole aliquot is directly derivatized with dansylchloride to produce the dansyl derivative of 1,2,4-Triazole. Determination and quantitation for derivatives of 1,2,4-Triazole, triazole alanine and triazole acetic acid are conducted using HPLC employing mass spectrometric (MS/MS) detection.

Chicken liver (1567 FLU)

Analyte: Flutriafol GC-MSD Method: ICIA AM00306

LOQ: 0.01 mg/kg

Description: Residues of flutriafol are extracted by homogenization with acetonitrile. An aliquot of the acetonitrile extracts is evaporated to dryness, reconstituted in dichloromethane, and subjected to gel permeation chromatographic (GPC) cleanup. A silica SPE cleanup is employed following GPC cleanup. The eluate is evaporated to dryness and reconstituted in a suitable volume of acetone for gas chromatographic analysis. Determination and quantification of flutriafol are conducted using gas chromatography employing mass selective (MSD) detection.

Validation data for methods on animal matrices are summarized in Table 41 and 42.

Table 41 Summary of Method Validation Data for flutriafol fortified into animal matrices

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine fat	0.01 0.1 1.0	7 3 3	71–101 79–88 79–91	88	9.3	1566 FLU
Bovine muscle	0.01 0.1 1.0	7 3 3	81–109 90–104 61–94	90	12	1566 FLU
Bovine milk	0.01 0.1 1.0	7 3 3	96–110 102–110 110–114	106	5.4	1566 FLU
Chicken fat	0.01 0.1 1.0	7 3 3	64–84 72–81 75–85	76	6.0	1567 FLU
Chicken muscle	0.01 0.1 1.0	7 3 3	78–91 89–100 104–111	92	10	1567 FLU
Chicken eggs	0.01 0.1 1.0	7 3 3	85–97 88–101 94–100	93	4.9	1567 FLU

Table 42 Summary of Method Validation Data for triazole metabolites fortified into animal matrices

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine fat 1,2,4-triazole	0.01 0.1 1.0	3 3 3	73–83 92–99 91–102	90	9.6	1566 FLU
Bovine fat Triazole alanine	0.01 0.1 1.0	3 3 3	95–116 86–88 89–98	95	9.7	1566 FLU
Bovine fat Triazole acetic acid	0.01 0.1 1.0	3 3 3	74–76 74–79 77–82	77	2.6	1566 FLU
Bovine milk 1,2,4-triazole	0.01 0.1 1.0	3 3 3	78–93 91–100 95–99	92	7.4	1566 FLU
Bovine milk Triazole alanine	0.01 0.1 1.0	3 3 3	90–93 94–99 101–106	97	5.2	1566 FLU
Bovine milk Triazole acetic acid	0.01 0.1 1.0	3 3 3	78–95 79–83 83–87	84	5.1	1566 FLU
Bovine muscle 1,2,4-triazole	0.01 0.1 1.0	3 3 3	86–95 90–96 89–97	92	3.9	1566 FLU
Bovine muscle Triazole alanine	0.01 0.1 1.0	3 3 3	85–102 85–92 82–91	90	7.3	1566 FLU
Bovine muscle Triazole acetic acid	0.01 0.1 1.0	3 3 3	82–88 76–80 79–83	81	3.6	1566 FLU
Chicken fat 1,2,4-triazole	0.01 0.1 1.0	3 3 3	104–118 100–102 98–106	105	6.8	1566 FLU
Chicken fat Triazole alanine	0.01 0.1 1.0	3 3 3	84–92 92–96 93 - 95	92	4.9	1566 FLU
Chicken fat Triazole acetic acid	0.01 0.1 1.0	3 3 3	86–91 84–86 83–84	86	2.7	1566 FLU

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Chicken muscle 1,2,4-triazole	0.01 0.1 1.0	3 3 3	72–90 86–93 94–96	89	7.5	1566 FLU
Chicken muscle Triazole alanine	0.01 0.1 1.0	3 3 3	92–96 93–95 99–100	96	2.9	1566 FLU
Chicken muscle Triazole acetic acid	0.01 0.1 1.0	3 3 3	79–90 81–83 79–85	83	3.5	1566 FLU
Chicken eggs 1,2,4-triazole	0.01 0.1 1.0	3 3 3	75–89 77–95 77–86	83	6.8	1566 FLU
Chicken eggs Triazole alanine	0.01 0.1 1.0	3 3 3	76–89 84–92 87–96	88	5.5	1566 FLU
Chicken eggs Triazole acetic acid	0.01 0.1 1.0	3 3 3	106–119 106–116 114 - 119	113	5.1	1566 FLU

Analytical methods for determination of flutriafol in soil

Soil (453 FLU)

Analyte: Flutriafol GC-NPD

LOQ: 0.01 mg/kg

Description: Residues of flutriafol are extracted from the soil by heating under reflux in the presence of acetonitrile/pH 9 water (70:30, v/v). The extract is partitioned into dichloromethane. All the samples are subjected to adsorption chromatography to remove interfering endogenous materials. Determination was by gas-liquid chromatography using a nitrogen specific thermionic detector.

Table 43 Summary of Method Validation Data for soil

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Soil	0.1 0.5	10 8	81–110 68–82	98 77	8.1 6.8	453 FLU

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of flutriafol residues in apple, grape, soya bean, oilseed rape and wheat samples for plant and animal commodities stored frozen.

The stability study of flutriafol was conducted on apple stored frozen at approximately -18 °C (Watson, 2006: 1377 FLU). Samples of untreated homogenized apple were fortified with flutriafol at 0.1 mg/kg then placed immediately into froze storage (nominally -18 °C), except 0 day specimens which were fortified and extracted immediately. Additional samples of untreated apple were stored under identical conditions for procedural recovery determination and for concurrent analysis with stored fortified samples. At set time periods of 0, 3, 6 and 12 months, a sub-set of fortified samples were removed from freezer storage and analysed using Agrisearch Method Flutriafol/Crops/DB/04/1. The residues of flutriafol were determined using LC-MS-MS. The LOQ was 0.01 mg/kg.

The results demonstrate that flutriafol is stable when stored at -18 °C in apple samples for 12 months.

Table 44 Recovery of flutriafol from stored fortified samples of apples

Time stored	Procedural recovery (%)	Flutriafol after storage (% remaining)
0 day	81, 82	83, 74, 83, 83, 77
3 months (95 days)	89, 83	94, 90, 90, 85, 89
6 months (186 days)	94, 90	89, 88, 83, 92, 92
12 months (354 days)	88, 88	86, 85, 90, 124, 90

The storage stability of flutriafol and triazole metabolite residues in frozen samples was determined as part of crop residue study (Willard, 2007: 1471 FLU). Apple fruit and apple juice were the matrices. Storage stability was conducted by fortifying separate duplicate samples of control apples and apple juice with flutriafol, 1,2,4-Triazole, Triazole alanine and Triazole acetic acid at a level of 0.5 mg/kg and then storing these samples alongside the study generated samples under the same frozen conditions (<-10 °C , nominally -20 °C).

A sub-sample of the homogenised matrix is extracted with acetonitrile/water (70:30 v/v). Extracts are then diluted with 0.5M sodium sulphate and partitioned with toluene to remove water soluble co-extractives prior to clean-up using solid phase extraction cartridges including Silica followed by Envi-Carb/MEGA BE-NH_2. The residues of flutriafol were determined using GC-MS. The LOQ for flutriafol was 0.01 mg/kg for each matrix.

A sub-sample of the homogenised matrix is extracted with methanol/water (80:20, v/v) and celite. Internal standards are added to aliquots of the filtered extract before clean-up using appropriate SPE cartridge and/or derivatisation. The derivatised extract is partitioned into ethyl acetate before being taken to dryness and re-constituted in acetonitrile/water (30:70 v/v). The residues of triazole metabolites were determined using LC-MS-MS. The LOQ of the method is 0.01 mg/kg for each metabolite/matrix combination.

Apple fruit and juice samples were stored for up to 62 and 49 days prior to extraction for analysis of flutriafol residues. Apple fruit and juice samples were stored for up to 72 and 56 days prior to extraction for analysis of triazole metabolite residues. The results indicate that flutriafol and triazole metabolites are stable in/on apples under frozen conditions for periods exceeding those experienced by the study samples.

Table 45 Recovery of flutriafol, 1,2,4-Triazole, Triazole alanine and Triazole acetic acid from stored fortified samples of apples and apple juice

Time stored (days)		% Recovery (0.50 mg/kg fortification)		
		Procedural recovery	Stored Sample	% remaining
Apple fruit				
Flutriafol	0	89, 90, 91		
	63	91, 84	91, 98, 93	94
1,2,4-Triazole	0	92, 86, 86		
	62	89, 86	77, 82, 75	78
	131	89, 91	80, 74, 81	78
Triazole alanine	0	101, 98, 103		
	62	98, 98	94, 97, 94	95
	131	96, 98	98, 97, 97	97
Triazole acetic acid	0	103, 105, 102		
	62	101, 103	104, 102, 100	102
	131	104, 103	106, 102, 103	104
Apple juice				
Flutriafol	0	76, 70, 83		
	62	73, 77	77, 73, 84	78
1,2,4-Triazole	0	92, 89, 88		
	63	85, 92	80, 86, 84	83
	129	92, 88	89, 84, 87	87
Triazole alanine	0	104, 101, 100		
	63	96, 95	94, 89, 92	92
	129	101, 99	94, 97, 96	96

Time stored (days)		% Recovery (0.50 mg/kg fortification)		
		Procedural recovery	Stored Sample	% remaining
Triazole acetic acid	0	101, 102, 95		
	63	105, 100	100, 97, 103	100
	129	111, 110	108, 108, 108	108

As part of a crop residue study on grapes, two residue samples shown to contain flutriafol residues of approximately 0.2 mg/kg were re-analysed after being stored for a period of 12 months under frozen conditions using the analytical method (Greig, 2005: 935 FLU). Treated samples were analysed in duplicate along with a control sample and freshly fortified procedural recovery sample at 0.2 mg/kg. The samples were analysed for flutriafol using LC-MS-MS (Flutriafol/Crops/ DMK/03/1). The LOQ was 0.01 m/kg.

The results of analysis of the stored treated samples show no indication of decline compared to the result of the sample analysed 687 and 702 days earlier. Residues of flutriafol in grapes remain stable for at least 702 days (approx. 2 years) when samples are stored under deep frozen conditions.

Table 46 Freezer storage stability of flutriafol in grapes

Time stored (days)	Procedural recovery (%)	Residues, mg/kg	% remaining
-	86		
0		0.18	
687		0.20, 0.19	108
0		0.19	
702		0.18, 0.18	95

The freeze storage stability of flutriafol in soya bean and processed fractions of meal, hulls and refined oil was conducted by fortifying separate control samples of homogeneous matrix with flutriafol at a level of 0.1 mg/kg (Rice, 2007: 1468 FLU). These samples were placed in freezer storage alongside treated study samples and analysed after the treated study sample analysis had been completed to ensure the period of storage was sufficient to cover the period of storage of the treated study samples. The samples were stored for 5 months for soya bean grain and 50–51 days for processed fractions. Unfortified control samples were analysed at the same time alongside duplicate freshly fortified samples of control matrix at 0.1 mg/kg. The samples were analysed by GC-NPD, using the analytical method described above. The LOQ was 0.01 mg/kg.

The results of analysis of the stored samples show no indication of decline compared to freshly fortified samples analysed on the same day. Results show flutriafol to be stable when soya bean seed was stored frozen for at least 5 months and processed fractions were stored for 1–2 months (> 50 days).

Table 47 Recovery of flutriafol from stored fortified samples of soya bean and processed fractions

Time stored	Fortified level, mg/kg	Procedural recovery (%)	Residue, mg/kg	% remaining
Soya bean seed				
0 days	0.10	101, 101		
5 months	0.10	84, 98	0.108, 0.098, 0.099	101
Meal				
0 days	0.10	93, 101		
51 days	0.50	82, 117	0.456, 0.415, 0.445	88
Hulls				
0 days	0.10	88, 89		
51 days	0.10	120, 102	0.105, 0.085, 0.081	90
Refined oil				
0 days	0.10	116, 113		
50 days	0.10	120, 116	0.097, 0.103, 0.124	98

The storage stability of flutriafol residues in barley grain was investigated by fortifying at a rate equivalent to a residue of 1.0 mg/kg (Swaine, 1982: 73 FLU). The fortified samples were stored

deep frozen (-20 °C) for up to 12 months. Triplicate samples were taken for analysis at 0, 3, 6, 9 and 12 month intervals. The samples were analysed using a gas-liquid chromatographic method. Generally flutriafol residues were stable over the entire storage period.

Table 48 Recovery of flutriafol from stored fortified samples of barley grains

Storage period (months)	Residue, mg/kg	% remaining
0	1.01, 1.05, 1.06	100
3	0.96, 1.02, 1.12	100
6	0.84, 0.90, 0.91	89
9	0.94, 1.00,1 1.04	99
12	0.90, 0.93, 0.94	92

The storage stability of flutriafol was tested by fortifying wheat plant, straw and grain with the test substance and analysing the samples after different periods of deep-freeze storage (mean temperature: -23 ºC) (Mende, 2004: 1126 FLU). Samples of plat and straw were fortified at 0.5 mg/kg, grain samples at 0.1 mg/kg and analysed at 0, 3, 6 and 12 months of storage. All samples were analysed in duplicate. At each sampling date, three procedural recovery samples per matrix (one at LOQ, two at the nominal concentration of storage stability samples) and one blank sample per matrix were analysed. The samples were analysed according to the validated analytical method which includes extraction of the samples with acetonitrile, followed by solid phase extraction on activated carbon, elution with ethyl acetate and final determination by HPLC-MS-MS. The LOQ was 0.01 mg/kg.

Results from this study indicate that these conditions are fulfilled for flutriafol in all three matrices for at least 12 months of storage under deep-freeze conditions.

Table 49 Freezer storage stability of flutriafol in wheat matrices

Matrix/ Storage period	Fortification level, mg/kg	Procedural recovery (%)	Residue, mg/kg	% remaining
Wheat plant				
0 days	0.50	88, 105	0.464, 0.487	95
3 months			0.509, 0.493	100
6 months			0.437, 0.477	91
12 months		96, 102	0.472, 0.451	92
Wheat straw				
0 days	0.50	95, 101	0.462, 0.443	91
3 months			0.508, 0.486	99
6 months			0.507, 0.516	102
12 months		88, 94	0.466, 0.455	92
Wheat grain				
0 days	0.10	91, 111	0.095, 0.094	95
3 months			0.098, 0.098	98
6 months			0.105, 0.095	100
12 months		94, 100	0.096, 0.096	96

The storage stability study of flutriafol on wheat straw was conducted for a period of up to 12 months (Bennet, 1993: 75 FLU). The samples were fortified with 0.5 mg/kg of flutriafol and immediately placed in the deep freeze at < -18 ºC until required for analysis. Samples were extracted by maceration in the presence of 70/30 acetonitrile/pH 9 water. The extracts were partitioned into dichloromethane. All samples were subjected to adsorption chromatography to remove interfering endogenous materials. Final quantitative determination was conducted by gas-liquid chromatography using nitrogen selective detection and external standardisation.

Table 50 Recovery of flutriafol from stored fortified samples of wheat straw

Stored period (months)	Procedural recovery (%)	Residues, mg/kg	% remaining
0	72, 85	0.40, 0.40, 0.40	80
3	95, 103	0.47, 0.48, 0.50	97
6	99, 104	0.48, 0.49, 0.51	99

Stored period (months)	Procedural recovery (%)	Residues, mg/kg	% remaining
9	105, 110	0.49, 0.51, 0.52	101
12	88, 89	0.45, 0.46, 0.50	94

No residues (< 0.05 mg/kg) were found in untreated control samples.

The storage stability of flutriafol on oilseed rape has been conducted over a period of 12 months (Bennet, 1993: 79 FLU). The samples were fortified with 0.5 mg/kg of flutriafol and immediately placed in the deep freeze at <-18 ºC until required for analysis. Flutriafol residues were extracted by maceration with acetonitrile/water (70:30). After filtration, an aliquot was diluted with Na_2SO_4 solution and partitioned into toluene. The extracts after evaporation were cleaned up on silica column to remove interfering coextractives. Finally flutriafol was determined by gas-liquid chromatography using a thermionic detector.

Table 51 Recovery of flutriafol from stored fortified samples of oilseed rape

Stored period (months)	Procedural recovery (%)	Residues, mg/kg	% remaining
0	86, 91	0.48, 0.49, 0.50	98
3	94, 96	0.45, 0.48, 0.50	95
6	94, 95	0.40, 0.42, 0.42	83
9	81, 101	0.51, 0.51	102
12	107, 114	0.53, 0.56, 0.58	111

No residues (< 0.05 mg/kg) were found in untreated control samples.

Freezer storage stability of flutriafol has been demonstrated on green coffee beans, roasted coffee, liquor extract and instant coffee. The samples were fortified with 0.1 mg/kg of flutriafol and immediately placed in the deep freeze at <-20 °C until required for analysis. Flutriafol residues were extracted by maceration with acetonitrile/water (70:30). After filtration, an aliquot was diluted with 0.5 M sodium sulfate solution and partitioned into toluene. The extracts after evaporation were cleaned up on silica column to remove interfering coextractives. Finally flutriafol was determined by gas-liquid chromatography employing mass selective (MSD) detection or HPLC employing tandem mass spectrometric (MS/MS) detection.

Table 52 Recovery of flutriafol from stored fortified samples of coffee beans, roasted coffee beans, liquor extract and instant coffee

Commodity	Storage interval (days)	Fortified level (mg/kg)	Procedural recovery (%)	Residues, mg/kg	% remaining
Coffee green beans	0	0.01	109, 116, 113, 119, 120		
	371	0.1	92, 102	0.089, 0.086	88
Roasted coffee beans	0	0.01	76, 71, 81, 120, 73		
	275	0.1	80, 77	0.079, 0.083	81
Liquor extract	0	0.01	99, 109, 111, 103,96		
	187	0.1	81, 74	0.077, 0.077	77
Instant coffee	0	0.01	114, 97, 111, 110, 112		
	186	0.1	73, 83	0.079, 0.093	86

Ambient storage stability study was also performed on coffee green beans since the Vietnam samples could not be shipped frozen with dry ice from that country and were for that reason stored at ambient temperatures for a maximum of 7 days (excluding backup samples not analysed) during the periode from RAC sampling to freezer storage at the laboratory. The samples were fortified with 0.1 mg/kg of flutriafol.

Table 53 Recovery of flutriafol from stored fortified samples of coffee green beans

Stored period (months)	Procedural recovery (%)	Residues, mg/kg	% remaining
0	115, 108		
7	105, 105	0.081, 0.090	86

Stored period (months)	Procedural recovery (%)	Residues, mg/kg	% remaining
14	107, 116	0.107, 0.094	100
42	110, 115	0.098, 0.109	104

The study was designed to determine the storage stability of residues of triazole acetic acid in crops stored deep frozen for up to two years (Kwiatkowski and Robinson, 1995: 169 FLU). Samples of wheat grain and straw, oilseed rape, cabbage, pea seed and sugar beet were fortified with triazole acetic acid at 0.5 mg/kg and stored deep frozen at <-12 °C. The samples were analysed using the analytical method which involved extraction with acetonitrile/water (1:2, v/v), subsampling of an aliquot and purification on a cation exchange resin. The aliquot was esterified by heating under reflux with isobutanol/3 M HCl. The resulting ester was separated by partitioned into dichloromethane, cleaned up by adsorption chromatography on Florisil and then analysed by gas-liquid chromatography using nitrogen/phosphorus specific detector.

No decrease in triazole acetic acid residue level was found in wheat grain and straw, oilseed rape, cabbage, pea seed or sugar beet root after storage periods of 24 to 25 months at <-12 ºC.

Table 54 Storage stability of triazole acetic acid residues in plant commodities

Commodity	Fortification level (mg/kg)	Storage interval	Procedural mean recovery (%)	Residue, mg/kg	% remaining
Wheat grain	0.5		106		
	0.5	24 hours		0.52, 0.55	108
		5 months		0.47, 0.48	96
		10 months		0.53, 0.71	124
		16 months		0.54, 0.61	116
		25 months		0.50, 0.51	102
Wheat straw	0.5		106		
	0.5	24 hours		0.53, 0.58	112
		5 months		0.47, 0.52	100
		10 months		0.58, 0.58	116
		17 months		0.47, 0.49	96
		25 months		0.49, 0.55	104
Pea seed	0.5		102		
	0.5	0 time		0.42, 0.54	96
		5 months		0.37, 0.45	82
		10 months		0.58, 0.61	120
		25 months		0.51, 0.59	110
Cabbage	0.5		95		
	0.5	24 hours		0.48, 0.53	102
		5 months		0.48, 0.51	100
		9 months		0.51, 0.53	104
		17 months		0.63, 0.66	130
		24 months		0.56, 0.64	120
Sugar beet root	0.5		89		
	0.5	0 time		0.56	112
		5 months		0.50, 0.51	102
		10 months		0.53, 0.60	114
		17 months		0.57, 0.59	116
		25months		0.53, 0.61	114
Oilseed rape	0.5		103		
	0.5	24 hours		0.48, 0.51	100
		5 months		0.48, 0.53	102
		9 months		0.51, 0.52	104
		17 months		0.46, 0.50	96
		24 months		0.47, 0.53	100

Frozen storage stability for flutriafol and triazole metabolites residues in whole milk, cow muscle and cow fat was determined as part of the cattle feeding study (Willard, 2008: 1566 FLU). Sets were analysed at 0-day and after the longest storage period for each matrix (from sample collection until extraction for flutriafol and triazole metabolites analysis). Storage intervals, interval

set size and fortification levels were the same for all matrices. Method validation and freezer storage stability sample preparation were conducted concurrently, therefore, method validation analyses served to provide the 0-day stability data.

Storage stability results indicate that both flutriafol residues and triazole metabolite residues were stable in all three matrices during frozen storage. The results reported support frozen storage stability of flutriafol residues in whole milk for at least 146 days, in cow muscle for at least 138 days, and in cow fat for at least 126 days. Triazole metabolite residues were shown to be stable during frozen storage for at least 173 days in whole milk, 167 days in cow muscle, and 169 days in cow fat. The LOQ for both flutriafol and its triazole metabolites was 0.01 mg/kg for all matrices.

Table 55: Storage stability of flutriafol and triazole metabolites in milk, cow muscle and fat

Analyte in Sample	Fortified level (mg/kg)	Storage period (days)	Procedural recovery[1] (%)	% remaining	Mean % remaining
Flutriafol in whole milk	0.1 0.1	0 146	82, 88 100, 98	 82, 79	 80
Flutriafol in cow muscle	0.1 0.1	0 138	102, 110 111, 105	 87, 91	 89
Flutriafol in cow fat	0.1 0.1	0 126	95, 104 89, 90	 72, 95	 84
1,2,4-triazole in whole milk	0.1 0.1	0 173	96, 100, 91 98, 97	 94, 102	 98
Triazole alanine in whole milk	0.1 0.1	0 173	99, 94, 98 107, 101	 94, 93	 94
Triazole acetic acid in whole milk	0.1 0.1	0 173	81, 83, 79 90, 89	 88, 87	 88
1,2,4-triazole in cow muscle	0.1 0.1	0 167	92, 96, 90 102, 97	 90, 91	 90
Triazole alanine in cow muscle	0.1 0.1	0 167	85, 92, 87 101, 102	 102, 96	 99
Triazole acetic acid in cow muscle	0.1 0.1	0 167	76, 80, 78 76, 75	 72, 70	 71
1,2,4-triazole in cow fat	0.1 0.1	0 169	99, 92, 97 91, 88	 86, 94	 90
Triazole alanine in cow fat	0.1 0.1	0 169	86, 88, 88 98, 100	 96, 98	 97
Triazole acetic acid in cow fat	0.1 0.1	0 169	79, 74, 76 74, 76	 62, 70	 66

[a] Sample fortified and extracted on same day

Frozen storage stability for flutriafol and triazole metabolites residues in chicken eggs, muscle and fat was determined as part of the poultry feeding study (Willard, 2008: 1567 FLU). Sets were analysed at 0-day and after the longest storage period for each matrix (from sample collection until extraction for flutriafol and triazole metabolites analysis). Storage intervals, interval set size and fortification levels were the same for all matrices. Method validation and freezer storage stability sample preparation were conducted concurrently, therefore, method validation analyses served to provide the 0-day stability data.

Storage stability results indicate that both flutriafol residues and triazole metabolite residues were stable in all three matrices during frozen storage. The results reported support frozen storage stability of flutriafol residues in eggs for at least 117 days, in muscle for at least 120 days, and in fat for at least 116 days. Triazole metabolite residues were shown to be stable during frozen storage for at least 176 days in eggs, 166 days in muscle, and 171 days in fat. The LOQ for both flutriafol and its triazole metabolites was 0.01 mg/kg for all matrices.

Table 56 Storage stability of flutriafol and triazole metabolites in eggs, chicken muscle and fat

Analyte in Sample	Fortified level (mg/kg)	Storage period (days)	Procedural recovery[a] (%)	% remaining	Mean % remaining
Flutriafol in eggs	0.1 0.1	0 117	88, 93 84, 89	 70, 82	 76

Analyte in Sample	Fortified level (mg/kg)	Storage period (days)	Procedural recovery[a] (%)	% remaining	Mean % remaining
Flutriafol	0.1	0	96, 100		
in chicken muscle	0.1	120	99, 105	76, 85	80
Flutriafol	0.1	0	72, 80		
in chicken fat	0.1	116	75, 74	63, 67	65
1,2,4-triazole	0.1	0	77, 95, 86		
in eggs	0.1	176	105, 92	105, 81	93
Triazole alanine	0.1	0	84, 92, 89		
in eggs	0.1	176	104, 104	87, 89	88
Triazole acetic acid	0.1	0	108, 106, 116		
in eggs	0.1	176	78, 77	78, 75	76
1,2,4-triazole	0.1	0	89, 93, 86		
in chicken muscle	0.1	166	84, 86	94, 89	92
Triazole alanine	0.1	0	93, 98, 95		
in chicken muscle	0.1	166	102, 99	99, 103	101
Triazole acetic acid	0.1	0	81, 82, 83		
in chicken muscle	0.1	166	72, 73	74, 70	72
1,2,4-triazole	0.1	0	102, 102, 100		
in chicken fat	0.1	171	94, 92	81, 79	80
Triazole alanine	0.1	0	98, 96, 92		
in chicken fat	0.1	171	94, 103	119, 107	113
Triazole acetic acid	0.1	0	85, 84, 86		
in chicken fat	0.1	171	80, 82	78, 83	80

[a] Sample fortified and extracted on same day

USE PATTERN

Flutriafol is registered around in many countries for control of certain fungal diseases on fruits, vegetables, pulses, cereals and oilseed etc. It is applied as foliar and soil treatment. The Meeting received labels in many countries in Europe, North America, Latin America, Asia and Australia. The information available to Meeting on registered uses of flutriafol is summarized in Table 54.

Table 57 Registered uses of flutriafol relevant to the review

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of flutriafol	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
Pome fruits									
Apple	Belarus	SC	250 g/L	Foliar	0.025-0.0375			4	40
Apple	Italy	SC	250 g/L	Foliar	0.020-0.030		0.0020-0.0030	2	21
Apple	Kazakhstan	SC	250 g/L	Foliar	0.025-0.0375			4	30
Apple	Lithuania	SC	250 g/L	Tractor fine-medium spray	0.050	200-300		2	20
Apple	Ukraine	SC	250 g/L	Foliar	0.025-0.0375			2	30
Pome fruit	USA	SC	125 g/L	Apply a uniform spray to the entire tree canopy	0.119			4	14
Berries and other small fruits									
Grapes	Moldova	SC	250 g/L	Foliar	0.025-0.0313			6	20
Grapes	Romania	SC	250 g/L	Foliar	0.0375	1000		6	30
Grapes	Taiwan	SC	125 g/L	Foliar	0.0625			4	14
Grapes	Ukraine	SC	250 g/L	Foliar	0.025			2	45
Grapes	USA	SC	125 g/L	Foliar	0.073-0.091			6	14
Assorted tropical and sub-tropical fruits–inedible peel									
Banana	Brazil	SC	125 g/L	Foliar	0.125-0.188	15-20			3
Banana	Colombia	SC	250 g/L	Foliar	0.1				0
Banana	Malaysia	SC	125 g/L	Foliar	0.156	500		5	14
Fruiting vegetables, other than Cucurbits									
Peppers	Spain	SC	125 g/L	Foliar		300-600	0.0094-0.019	3	1

Crop	Country	Formulation		Application					PHI days
		Type	Conc. of flutriafol	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hl	Number max	
Pulses									
Soya bean	Argentina	SC	125 g/L	Foliar	0.050-0.075	200			28
Soya bean	Argentina	SC	125 g/L	Aerial application	0.050-0.075	20			28
Soya bean	Brazil	SC	125 g/L	Foliar	0.050-0.125	200		2	28
Soya bean	South Africa	SC	125 g/L	Foliar	0.125-0.156	300		2	49
Soya bean	USA	SC	125 g/kg	Foliar	0.064-0.128	> 93.5		3	21
Soya bean	USA	SC	125 g/kg	Aerial application	0.064-0.128	> 46.8		3	21
Cereal grains									
Wheat	Argentina	SC	125 g/L	Foliar	0.125	200			35
Wheat	Argentina	SC	125 g/L	Aerial application	0.125	20			35
Wheat	Australia	SC	250 g/L	Foliar	0.0625-0.125	100-150		2	49
Wheat	Australia	SC	250 g/L	Aerial application	0.0625-0.125	15		2	49
Wheat	Belarus	SC	250 g/L	Foliar	0.125			2	30
Wheat	Chile	SC	125 g/L	Foliar	0.125	200			15
Wheat	Chile	SC	125 g/L	Aerial application	0.125	30-50			15
Wheat	Croatia	SC	250 g/L	Foliar	0.125	200-300		2	35
Wheat	Estonia	SC	250 g/L	Foliar	0.125	200-300			
Wheat	Kazakhstan	SC	250 g/L	Foliar	0.125			2	40
Wheat	Lithuania	SC	250 g/L	Foliar	0.125	200-300		2	30
Wheat	Mexico	SC	125 g/L	Foliar	0.094-0.125			2	35
Wheat	Moldova	SC	250 g/L	Foliar	0.125			3	
Wheat	Romania	SC	250 g/L	Foliar	0.125	300		2	30
Wheat	Russia	SC	250 g/L	Foliar	0.125			2	40
Wheat	South Africa	SC	125 g/L	Foliar	0.125	300		2	42
Wheat	South Africa	SC	125 g/L	Aerial application	0.156	30		2	42
Wheat	Spain	SC	125 g/L	Foliar	0.125	300-600		a	b
Wheat	Spain	SC	125 g/L	Aerial application	0.125	40-80		a	b
Wheat	Ukraine	SC	250 g/L	Foliar	0.125			2	30
Wheat	USA	SC	250 g/L	Foliar	0.064-0.128	> 93.5		2	30
Wheat	USA	SC	250 g/L	Aerial application	0.064-0.128	> 46.8		2	30
Oilseed									
Peanut	Argentina	SC	125 g/kg	Foliar	0.0625	200			35
Peanut	USA	SC	125 g/kg	Foliar	0.064-0.128	> 93.5		4	7
Seed for beverages and sweets									
Coffee	Brazil	SC	125 g/L	Foliar	0.188-0.25	500		2	30
Coffee	Brazil	SC	125 g/L	Soil application	0.438-0.688			1	120
Coffee	Colombia	SC	125 g/L	Foliar	0.1-0.125			3	30
Coffee	Malaysia	SC	125 g/L	Foliar	0.25	500		4	30

[a] not necessary

[b] Between the end of stem elongation and flowering

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on flutriafol supervised field trials for the following crops.

Group	Commodity	Table
Pome fruits	Apples	Table 56, 57
Berries and other small fruits	Grapes	Table 58, 59

Group	Commodity	Table
Assorted tropical and sub-tropical fruits - inedible peel	Banana	Table 60
Fruiting vegetables, other than Cucurbits	Sweet peppers	Table 61
Pulses	Soya bean	Table 62, 63
Cereal grains	Wheat	Table 64, 65
Oilseed	Peanut	Table 66
Seed for beverages and sweets	Coffee	Table 67

Flutriafol SC formulation was applied for foliar spray and soil treatment. Each of the field trial sites generally consisted of untreated control plot and treated plot. Application rates and spray concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Apples

Flutriafol was applied to apples at 9 trials in North France (Northern Europe) and at 11 trials in South France, Greece, Italy and Spain (Southern Europe). Nine trials were conducted for decline curve studies and the other eleven for harvest studies. In all trials, 3 foliar applications of flutriafol (250 g/L SC formulation) were made at a rate of nominally 0.030 kg ai/ha with a 14 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.0 mg/kg. The recoveries of flutriafol from apple fruits fortified were 73–109% (0.01 mg/kg), 74–105% (0.1 mg/kg) and 98–103% (1.0 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg.

Table 58 Flutriafol residues on apples from supervised trials in Europe

Apples country, year (variety)	Application						PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
GAP, Lithuania	*SC*	*0.050*		*300*		*2*	*20*		
GAP, Italy	*SC*	*0.030*	*0.0030*			*2*	*21*		
Northern Europe									
North France, 2003 (Pink Lady)	SC	0.031 0.035 0.031	0.0040 0.0040 0.0040	771 886 776	75 - 77	3	0 21 28 35 42	0.05 0.01 0.02 0.02 0.01	Jones, 2005, 1294 FLU Sampling to extraction interval: 135–203 days
North France, 2003 (Jonagored)	SC	0.030 0.031 0.029	0.0039 0.0037 0.0036	788 856 804	77 - 81	3	0 21 28 35 42	0.08 0.02 < 0.01 < 0.01 < 0.01	 Extraction to quantitation interval: 6–13 days
North France, 2003	SC	0.031 0.029	0.0048 0.0046	650 633	78 - 81	3	0 21	0.05 0.02	

Apples country, year (variety)	Application						PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
(Golden Smother)		0.029	0.0046	640			28 35 42	< 0.01[a] < 0.01 0.02	
North France, 2003 (Golden)	SC	0.030 0.030 0.031	0.0026 0.0045 0.0039	1173 670 800	77 - 79	3	0 21 28 35 42	0.03 0.02 0.03 0.02 0.03	
North France, 2004 (Granny Smith)	SC	0.032 0.032 0.031	0.0032 0.0032 0.0030	1020 1027 1007	78 - 81	3	21	0.01	Jones, 2005, 1295 FLU Sampling to extraction interval: 113–162 days Extraction to quantitation interval: 13 days
North France, 2004 (Golden Delicious)	SC	0.031 0.032 0.031	0.0021 0.0021 0.0021	1526 1551 1513	78 - 85	3	21	0.01	
North France, 2004 (Golden)	SC	0.031 0.031 0.029	0.0028 0.0027 0.0026	1125 1173 1107	75 - 78	3	21	0.01	
North France, 2004 (Elstar)	SC	0.030 0.031 0.031	?	1258 1194 1183	76 - 78	3	21	< 0.01	
North France, 2004 (Golden)	SC	0.030 0.031 0.030	0.0030 0.0030 0.0029	993 1021 1064	81 - 85	3	21	0.02	
Southern Europe									
Greece, 2005 (Granny Smith)	SC	0.031 0.030 0.031		1520 1506 1509	77 - 86	3	0 7 14 21 28	0.03 0.01 < 0.01 0.01 0.01	Jones, 2006, 1329 FLU Storage interval: 84 days
Greece, 2006 (Granny Smith)	SC	0.029 0.029 0.030		771 780 796	85 - 87	3	21	0.02	Jones, 2007, 1478 FLU Storage interval: 83 days
South France, 2003 (Pink Lady)	SC	0.033 0.031 0.032	0.0032 0.0032 0.0032	1038 965 994	78 - 79	3	0 21 28 35 42	0.02 0.01 < 0.01 < 0.01 < 0.01	Jones, 2005, 1294 FLU Sampling to extraction interval: 129–213 days Extraction to quantitation interval: 6–12 days
South France, 2003 (Granny)	SC	0.031 0.031 0.031	0.0040 0.0040 0.0040	769 761 764	75 - 77	3	0 21 28 35 42	0.02 < 0.01 < 0.01 < 0.01 < 0.01	
Italy, 2003 (Nero Red)	SC	0.029 0.028 0.031	0.0017 0.0019 0.0018	1739 1475 1742	75 - 77	3	0 21 28 35 42	0.03 < 0.01 < 0.01 < 0.01 < 0.01	
Spain, 2003 (Golden)	SC	0.030 0.030 0.030	0.0024 0.0023 0.0024	1256 1326 1276	73 - 76	3	0 21 28 35 42	0.09 0.05 0.02 0.01 0.03	
South France, 2004 (Golden)	SC	0.030 0.030 0.029	?	1216 1210 1192	75 - 81	3	21	0.02	Jones, 2005, 1295 FLU Sampling to extraction interval: 126–156 days Extraction to quantitation interval: 13 days
South France, 2004 (Starkinson)	SC	0.029 0.031 0.032	0.0034 0.0034 0.0033	842 908 967	75 - 81	3	21	0.01	
Italy, 2004 (Imperatore Dallago)	SC	0.030 0.027 0.032	0.0027 0.0026 0.0025	1108 1040 1308	77 - 81	3	21	< 0.01	

Apples country, year (variety)	Application						PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
Italy, 2004 (Fuji)	SC	0.033 0.031 0.035	0.0022 0.0022 0.0022	1451 1380 1587	77 - 85	3	21	< 0.01	
Spain, 2004 (Golden)	SC	0.029 0.030 0.030	0.0039 0.0038 0.0035	759 804 847	74 - 79	3	21	0.01	

[a] Samples potentially mixed up

The Meeting received 16 trials on apple which were conducted in USA, in California, Idaho, Illinois, Michigan, New York, Pennsylvania, Utah, Virginia and Washington. All trials received 6 applications of flutriafol 125g/L SC at the application rate of 0.12 kg ai/ha with a 14 days interval. At the three residue decline trial sites (MI, PA and WA), additional plot received five applications of flutriafol 125g/L SC at the application rate of 0.12 kg ai/ha. At the two processing trial sites (NY and WA), additional plot was treated with the first three applications at the nominal application rate of 0.12 kg ai/ha and the last three applications at the 2× rate of 0.24 kg ai/ha.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.0 mg/kg and with triazole metabolite at levels ranging from 0.01 to 0.10 mg/kg. The concurrent recoveries of analytes from apple fruits were 70–105% for flutriafol, 87–112% for 1,2,4-Triazole, 85–128% for Triazole alanine, and 83–125% for Triazole acetic acid. The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in any of the whole fruit samples analysed. With the exception of one decline trial, no residues of Triazole acetic acid were found in any of the whole fruit samples analysed.

Table 59 Flutriafol and triazole metabolites residues on apples from supervised trials in USA

Apples country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
GAP, USA	*SC*	*0.119*		*4*	*14*					
USA/CA, 2006 (Granny smith)	SC	0.12	798 - 936	6	14	0.07, 0.05 mean 0.06	< 0.01	0.02, 0.02	< 0.01, < 0.01	Willard, 2007 1471 FLU
USA/ ID, 2006 (Macintosh)	SC	0.12	759 - 931	6	15	0.07, 0.09 mean 0.08	< 0.01	< 0.01, < 0.01	< 0.01,< 0.01	Storage interval:
USA/IL, 2006 (Golden Supreme)	SC	0.12	795 - 840	6	14	0.06, 0.06 mean 0.06	< 0.01	0.02, 0.02	< 0.01, < 0.01	Flutriafol 7 - 62 days
USA/MI, 2006 (Golden Delicious)	SC	0.12	801 - 843	6	14	0.09, 0.09 mean 0.09	0.06	0.04, 0.04	< 0.01, < 0.01	Triazole metabolites 12–72 days
USA/MI, 2006 (Ida Red)	SC	0.12	807 - 827	6	0	0.07, 0.07 mean 0.07		0.06, 0.06	< 0.01, < 0.01	
					7	0.05, 0.04 mean 0.05		0.07, 0.06	< 0.01, < 0.01	
					13	0.05, 0.05 mean 0.05	0.03	0.05, 0.05	< 0.01, < 0.01	
					21	0.07, 0.07 mean 0.07		0.07, 0.07	< 0.01, < 0.01	
					27	0.04, 0.04 mean 0.04		0.06, 0.05	< 0.01, < 0.01	

Apples country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
						0.05, 0.04 mean 0.05				
		0.12	804 - 838	5	0	0.06, 0.06 mean 0.06		0.08, 0.05	< 0.01, < 0.01	
					7	0.04, 0.04 mean 0.04		0.07, 0.08	< 0.01, < 0.01	
					13	0.04, 0.04 mean 0.04		0.07, 0.07	< 0.01, < 0.01	
					21	0.04, 0.04 mean 0.04		0.08, 0.09	< 0.01, < 0.01	
					27	0.03, 0.03 mean 0.03		0.07, 0.07	< 0.01, < 0.01	
USA/NY, 2006 (Cortland)	SC	0.12	924 - 981	6	15	0.05, 0.03 mean 0.04	0.03	0.02, 0.01	< 0.01, < 0.01	
USA/NY, 2006 (Ida Red)	SC	0.12	939 - 953	6	14	0.05, 0.07 mean 0.06	0.01	0.03, 0.02	< 0.01, < 0.01	
		0.12 and 0.24	933 - 942	6	14	0.10, 0.12 mean 0.11		0.03, 0.03	< 0.01, < 0.01	
USA/OR, 2006 (Pacific Gala)	SC	0.12	830 - 849	6	14	0.09, 0.12 mean 0.10	0.03	0.03, 0.02	< 0.01, < 0.01	
USA/OR, 2006 (Jonagold)	SC	0.12	815 - 840	6	14	0.05, 0.05 mean 0.05	0.02	0.03, 0.03	< 0.01, < 0.01	
USA/PA, 2006 (Royal Gala)	SC	0.12	895 - 903	6	14	0.11, 0.14 mean 0.12	0.03	0.02, 0.02	< 0.01, < 0.01	
USA/PA, 2006 (Loe Rome)	SC	0.12	789 - 808	6	0	0.14, 0.19 mean 0.17		0.05, 0.05	0.01, 0.02	
					7	0.09, 0.08 mean 0.09		0.05, 0.05	0.01, 0.01	
					14	0.05, 0.06 mean 0.05	0.05	0.05, 0.05	0.01, 0.01	
					21	0.07, 0.09 mean 0.08		0.06, 0.06	0.01, 0.01	
					28	0.06, 0.05 mean 0.06		0.05, 0.05	0.01, 0.01	
		0.12	800 - 815	5	0	0.14, 0.17 mean 0.16		0.03, 0.04	0.01, 0.01	

Apples country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
					7	0.05, 0.05 mean 0.05		0.04, 0.04	< 0.01, < 0.01	
					14	0.05, 0.06 mean 0.06		0.04, 0.04	< 0.01, < 0.01	
					21	0.07, 0.07 mean 0.07		0.04, 0.04	< 0.01, < 0.01	
					28	0.08, 0.05 mean 0.07		0.03, 0.03	< 0.01, < 0.01	
USA/UT, 2006 (Empire)	SC	0.12	748 - 804	6	14	0.03, 0.03 mean 0.03	< 0.01	< 0.01, < 0.01	< 0.01, < 0.01	
USA/VA, 2006 (Rome)	SC	0.12	706 - 748	6	13	0.06, 0.04 mean 0.05	0.06	0.03, 0.02	< 0.01, < 0.01	
USA/VA, 2006 (York)	SC	0.12	805 - 817	6	13	0.12, 0.09 mean 0.10	0.03	0.03, 0.02	< 0.01, < 0.01	
USA/WA, 2006 (Braeburn)	SC	0.12	861 - 879	6	0	0.09, 0.10 mean 0.10		< 0.01, < 0.01	< 0.01, < 0.01	
					7	0.10, 0.12 mean 0.11		< 0.01, < 0.01	< 0.01, < 0.01	
					14	0.09, 0.12 mean 0.11	< 0.01	0.01, 0.01	< 0.01, < 0.01	
					21	0.13, 0.13 mean 0.13		< 0.01, 0.01	< 0.01, < 0.01	
					27	0.07, 0.11 mean 0.09		0.01, < 0.01	< 0.01, < 0.01	
		0.12	864 - 871	5	0	0.16, 0.13 mean 0.14		0.02, 0.02	< 0.01, < 0.01	
					7	0.15, 0.13 mean 0.14		0.02, 0.02	< 0.01, < 0.01	
					14	0.14, 0.11 mean 0.13		0.02, 0.02	< 0.01, < 0.01	
					21	0.15, 0.16 mean 0.16		0.02, 0.02	< 0.01, < 0.01	
					27	0.09, 0.16 mean 0.13		0.02, 0.02	< 0.01, < 0.01	

Apples country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
USA/WA, 2006 (Red Delicious)	SC	0.12	861 - 872	6	14	0.13, 0.11 mean 0.12	0.02	0.04 0.03	< 0.01 < 0.01	
		0.12 and 0.24	859 - 877	6	14	0.17, 0.21 mean 0.19		0.04 0.04	< 0.01 < 0.01	

UTC: untreated control samples, TRT: treated samples

Grapes

Flutriafol was applied to grapes at 9 trials in North France (Northern Europe) and at 11 trials in South France (Southern Europe). Nine trials were conducted for decline curve studies and the other eleven for harvest studies. Grapes were sprayed twice at 0.075 kg ai/ha (250 g/L SC formulation) with a 14 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.0 mg/kg. The recoveries of flutriafol from grape fruits fortified were 61–104% (0.01 mg/kg), 87–95% (0.1 mg/kg) and 65–103% (1.0 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg.

Table 60 Flutriafol residues on grapes from supervised trials in Europe

Grapes country, year (variety)	Application						PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
GAP, Ukraine	*SC*	*0.025*				*2*	*45*		
GAP, Romania	*SC*	*0.038*		*1000*		*6*	*30*		
Northern Europe									
North France, 2003 (Chenin)	SC	0.077 0.080	0.057 0.062	1352 1289	81	2	0 10 21 28 35	0.04 < 0.01 0.01 < 0.01 < 0.01	Jones, 2005, 1299 FLU Sampling to extraction interval: 154–203 days Extraction to quantitation interval: 2 - 5 days
North France, 2003 (Cabernet Franc)	SC	0.075 0.075	0.011 0.011	669 687	83 85	2	0 10 21 28 35	0.07 0.03 0.02 0.01 0.01	
North France, 2003 (Cabernet Franc)	SC	0.073 0.076	0.013 0.013	558 578	83 - 85	2	0 10 21 28 35	0.18 0.11 0.08 0.05 0.05	
North France, 2003 (Grolleau)	SC	0.075 0.072	0.013 0.012	562 618	81 85	2	0 10 21 28 35	0.08 0.09 0.05 0.04 0.05	
North France, 2004 (Grolleau)	SC	0.080 0.076	0.069 0.069	1169 1110	83 - 85	2	21	0.03	Jones, 2005, 1296 FLU Sampling to extraction interval: 130–161 days Extraction to quantitation interval: 11 days
North France, 2004 (Cabernet Franc)	SC	0.083 0.079	0.012 0.012	685 671	83 85	2	21	0.07	
North France, 2004 (Pinot)	SC	0.077 0.075	0.012 0.011	667 667	79 81	2	21	< 0.01	
North France, 2004 (Chardonnay)	SC	0.077 0.080	0.011 0.010	705 769	79 81	2	21	0.02	
North France, 2004 (Gamay)	SC	0.074 0.075	0.0090 0.0091	815 823	81 - 85	2	21	0.02	
Southern Europe									

Grapes country, year (variety)	Application						PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
Greece, 2005 (Robits)	SC	0.074 0.077		973 971	78 82	2	0 7 10 21 28	0.10 0.11 0.07 0.05 0.04	Jones, 2006, 1330 FLU Storage interval: 125 days
Greece, 2006 (Muscat Humburg)	SC	0.075 0.071		802 762	81 - 85	2	21	0.03	Jones, 2007, 1396 FLU Storage interval: 145 days
South France, 2003 (Cabernet)	SC	0.077 0.074	0.011 0.0086	705 863	81 85	2	0 10 21 28 35	0.08 0.05 0.04 0.02 0.02	Jones, 2005, 1299 FLU Sampling to extraction interval: 171 - 227 days Extraction to quantitation interval: 3 - 6 days
South France, 2003 (Cabernet Sauvignon)	SC	0.076 0.073	0.023 0.023	324 312	83 85	2	0 10 21 28 35	0.19 0.13 0.09 0.06 0.05	
Italy, 2003 (Trebianno)	SC	0.082 0.076	0.011 0.010	764 732	81 - 85	2	0 10 21 28 35	0.13 0.03 0.03 0.02 0.01	
Spain, 2003 (Zelema)	SC	0.072 0.075	0.0072 0.0070	1001 1076	77 - 79	2	0 10 21 28 35	0.16 0.02 0.02 < 0.01 < 0.01	
South France, 2004 (Abouriou)	SC	0.077 0.080	0.014 0.013	570 604	81 - 85	2	21	0.04	Jones, 2005, 1296 FLU Sampling to extraction interval: 112–161 days Extraction to quantitation interval: 10 days
South France, 2004 (Tannat)	SC	0.072 0.075	0.016 0.016	460 480	83 85	2	21	0.02	
Italy, 2004 (Trebbiano)	SC	0.076 0.072	0.0094 0.0089	814 809	83 - 85	2	21	0.04	
Italy, 2004 (Sangiovese)	SC	0.073 0.074	0.0077 0.0065	939 1150	83 - 85	2	21	< 0.01	
Spain, 2004 (Grenache)	SC	0.079 0.075	0.0083 0.0083	948 900	83 85	2	21	< 0.01	

Thirteen residue trials were established in typical grape growing areas in USA, in New York, Pennsylvania, California, Washington and Idaho, using commercially viable varieties of grapes. All trials received 7 applications of flutriafol 125 g/L SC at rate of 0.128 kg ai/ha with a 10 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.0 mg/kg and with triazole metabolite at levels ranging from 0.01 to 0.10 mg/kg. The concurrent recoveries of analytes from grape fruits were 85–120% for flutriafol, 79–104% for 1,2,4-Triazole, 75–100% for Triazole alanine, and 72–97% for Triazole acetic acid. The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in any of the whole fruit samples analysed.

Table 61 Flutriafol and triazole metabolites residues on grapes from supervised trials in USA

Grapes country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
GAP, USA	*SC*	*0.073-0.091*		*6*	*14*					
USA/NY, 2007 (Concord)	SC	0.128	1403	7	1	0.58, 0.58 mean 0.58	0.03	0.03, 0.03	< 0.01, < 0.01	Rice, 2008 1606 FLU
					7	0.61, 0.54 mean 0.58	0.03	0.03, 0.04	< 0.01, < 0.01	Storage interval: 161–202 days
					14	0.39, 0.40 mean 0.40	0.02	0.04, 0.03	0.01, 0.01	
					21	0.45, 0.41 mean 0.43	0.03	0.05, 0.06	0.01, 0.01	
					28	0.38, 0.27 mean 0.32	0.06	0.05, 0.07	0.01, 0.01	
USA/ PA, 2007 (Niagara)	SC	0.128	561	7	14	0.39, 0.22 mean 0.30	0.06	0.07, 0.06	< 0.01, 0.01	
USA/CA, 2007 (Thompson Seedless)	SC	0.128	364 - 413	7	14	0.34, 0.28 mean 0.31	0.02	0.02, 0.02	< 0.01, < 0.01	
USA/CA, 2007 (Chenin Blanc)	SC	0.128	363 - 410	7	14	0.21, 0.21 mean 0.21	< 0.01	0.03, 0.03	< 0.01, < 0.01	
USA/CA, 2007 (Thompson Seedless)	SC	0.128	1546 - 1584	7	14	0.20, 0.21 mean 0.21	< 0.01	0.05, 0.04	< 0.01, < 0.01	
USA/CA, 2007 (Grenache)	SC	0.128	1500 - 1575	7	14	0.26, 0.44 mean 0.35	< 0.01	0.02, 0.02	< 0.01, < 0.01	
USA/CA, 2007 (Thompson Seedless)	SC	0.128	1178 - 1211	7	14	0.08, 0.15 mean 0.12	0.01	0.03, 0.04	< 0.01, < 0.01	
		0.256	1164 - 1186	7	14	0.34, 0.45 mean 0.40	< 0.01	0.04, 0.04	< 0.01, < 0.01	
USA/CA, 2007 (Thompson Seedless)	SC	0.128	325 - 339	7	14	0.27, 0.22 mean 0.25	0.01	0.02, 0.02	< 0.01, < 0.01	
		0.256	327 - 332	7	14	Not analyse				
USA/CA, 2007 (Ruby Red)	SC	0.128	1550 - 2335	7	14	0.27, 0.33 mean 0.30	< 0.01	< 0.01, 0.01	< 0.01, < 0.01	
USA/CA, 2007 (Crimson)	SC	0.128	675 - 725	7	1	0.40, 0.42 mean 0.41	0.01	0.02, 0.02	0.01, 0.01	
					7	0.42, 0.35 mean 0.39	< 0.01	0.02, 0.02	< 0.01, < 0.01	
					14	0.41, 0.33 mean 0.37	0.01	0.02, 0.02	< 0.01, < 0.01	
					21	0.34, 0.31 mean 0.33	0.01	0.02, 0.02	< 0.01, < 0.01	
					28	0.32,0.36 Mean 0.34	0.02	0.02, 0.02	< 0.01, 0.01	
USA/ WA, 2007 (White Reisling)	SC	0.128	1397 - 1408	7	14	0.84, 0.89 mean 0.86	< 0.01	0.02, 0.02	< 0.01, < 0.01	
USA/ ID, 2007 (Concord)	SC	0.128	468	7	14	0.60, 0.61 mean 0.61	< 0.01	< 0.01, < 0.01	< 0.01, < 0.01	
USA/CA, 2007 (Globes)	SC	0.128	664 - 699	7	14	0.27, 0.30 mean 0.28	0.03	0.05, 0.06	< 0.01, < 0.01	

UTC: untreated control samples, TRT, treated samples

Banana

Twelve residue trials were carried out in the major banana growing countries in Latin America, in Colombia, Costa Rica, Ecuador, Guatemala and Honduras. All trials received 8 applications of flutriafol 125g/L SC at rate of 0.121–0.130 kg ai/ha with an 8–11 days interval. The untreated control plot (UTC) contained banana plants with the raceme covered with the commercially normal protective bag. The treated plot (TRT) at each trial location had both banana plants with raceme covered with the protective bag (bagged) and plants with the raceme not covered with the protective bag (unbagged).

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 10 mg/kg and with triazole metabolite at levels ranging from 0.01 to 0.50 mg/kg. The concurrent recoveries of analytes from whole banana fruits were 86–112% for flutriafol (0.01 and 0.5 mg/kg), 71–97% for 1,2,4-Triazole (0.01 and 0.1 mg/kg), 80–107% for Triazole alanine (0.02 and 0.2 mg/kg), and 80–92% for Triazole acetic acid (0.02 and 0.2 mg/kg). The concurrent recoveries of analytes from pulp were 85–101% for flutriafol (0.01, 0.5 and 10 mg/kg), 71–102% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 85–99% for TA (0.025 and 0.50 mg/kg), and 79–99% for Triazole acetic acid (0.02 and 0.50 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg for all analytes. No storage stability study was conducted on banana samples.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine, and Triazole acetic acid. No residues of 1,2,4-Triazole and Triazole acetic acid were found above the LOQ in any of the whole fruit samples and the pulp samples analysed.

Table 62 Flutriafol and triazole metabolites residues on banana from supervised trials in Latin America

Banana country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg			Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		
								UTC	TRT	
GAP, Brazil *GAP, Colombia*	*SC* *SC*	*0.188* *0.1*	*25*			*3* *0*				
Colombia, 2009 (Valery)	SC	0.122 - 0.127	29.3 - 30.5	8	Bagged Whole fruit	0	0.03, 0.04 mean 0.04	< 0.01	0.01, 0.01	Willard, 2009 1626 FLU
					Pulp		0.09, 0.04 mean 0.07	< 0.01	0.02, 0.02	Storage interval:
					Unbagged Whole fruit	0	0.11, 0.09 mean <u>0.10</u>		< 0.01, < 0.01	Flutriafol 5–186 days
					Pulp		0.04, 0.05 mean <u>0.05</u>		0.01, 0.01	Triazole metabolites 24 - 205 days
Colombia, 2009 (Valery)	SC	0.123- 0.128	29.3 - 30.7	8	Bagged Whole fruit	0	0.06, 0.04 mean 0.05	0.01	0.02, 0.02	
					Pulp		0.03, 0.03 mean 0.03	0.01	0.03, 0.02	
					Unbagged Whole fruit	0	0.09, 0.10 mean <u>0.09</u>		0.01, 0.01	
					Pulp		0.06, 0.04 mean <u>0.05</u>		0.02, 0.02	

Banana country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg			Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		
								UTC	TRT	
Costa Rica, 2009 (Gran Enano)	SC	0.126-0.127	30.2 - 30.6	8	Bagged Whole fruit	0	0.06, 0.03 mean 0.05	< 0.01	0.01, 0.01	
						3	0.01, 0.05 mean 0.03		< 0.01, 0.01	
						5	0.03, 0.02 mean 0.02		0.01, 0.01	
						7	0.03, 0.02 mean 0.02		0.01, < 0.01	
						10	0.04, 0.01 mean 0.03		0.01, < 0.01	
					Pulp	0	0.01, 0.05 mean 0.03	< 0.01	0.01, 0.01	
						3	0.04, 0.02 mean 0.03		0.02, 0.01	
						5	0.03, 0.05 mean 0.04		0.01, 0.01	
						7	0.02, 0.05 mean 0.04		0.01, 0.02	
						10	0.02, 0.07 mean 0.05		0.01, 0.01	
					Unbagged Whole fruit	0	0.19, 0.16 mean 0.17		< 0.01, < 0.01	
						3	0.11, 0.06 mean 0.08		< 0.01, 0.01	
						5	0.07, 0.08 mean 0.08		< 0.01, 0.01	
						7	0.04, 0.06 mean 0.05		0.01, 0.01	
						10	0.04, 0.05 mean 0.05		0.01, < 0.01	
					Pulp	0	0.05, 0.08 mean 0.07		0.01, 0.01	
						3	0.12, 0.05 mean 0.08		0.01, 0.01	
						5	0.04, 0.04 mean 0.04		0.01, 0.01	
						7	0.05, 0.06 mean 0.05		0.01, 0.01	
						10	0.07, 0.05 mean 0.06		0.01, 0.01	
Costa Rica, 2009 (Valery)	SC	0.126-0.127	29.3 - 30.4	8	Bagged Whole fruit	0	0.02, 0.02 mean 0.02	< 0.01	< 0.01, 0.01	
					Pulp		0.01, 0.02 mean 0.01	< 0.01	0.01, < 0.01	
					Unbagged Whole fruit	0	0.15, 0.20 mean 0.17		< 0.01, < 0.01	
					Pulp		0.06, 0.04 mean 0.05		0.01, 0.01	

Banana country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg			Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		
								UTC	TRT	
Costa Rica, 2009 (Williams)	SC	0.126 - 0.127	30.3 - 30.4	8	Bagged Whole fruit	0	0.01, 0.02 mean 0.02	0.01	0.01, 0.01	
					Pulp		< 0.01, < 0.01 mean < 0.01	< 0.01	0.01, 0.01	
					Unbagged Whole fruit	0	0.05, 0.05 mean 0.05		< 0.01, < 0.01	
					Pulp		0.04, 0.10 mean 0.07		0.01, 0.01	
Ecuador, 2009 (Giant Cavendish)	SC	0.121-0.122	29.1 - 29.3	8	Bagged Whole fruit	0	< 0.01, < 0.01 mean < 0.01		0.04, 0.04	
						3	< 0.01, 0.01 mean 0.01		0.04, 0.04	
						5	0.02, 0.01 mean 0.02		0.05, 0.05	
						7	0.01, < 0.01 mean 0.01		0.05, 0.06	
						10	0.01, < 0.01 mean 0.01		0.01, 0.04	
					Pulp	0	< 0.01, < 0.01 mean < 0.01		0.05, 0.06	
						3	< 0.01, < 0.01 mean < 0.01		0.05, 0.04	
						5	< 0.01, < 0.01 mean < 0.01		0.05, 0.05	
						7	< 0.01, < 0.01 mean < 0.01		0.06, 0.04	
						10	< 0.01, < 0.01 mean < 0.01		0.06, 0.06	

Banana country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg			Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		
								UTC	TRT	
					Unbagged Whole fruit	0	0.15, 0.13 mean 0.14	0.02	0.03, 0.03	
						3	0.07, 0.09 mean 0.08		0.03, 0.03	
						5	0.07, 0.05 mean 0.06		0.03, 0.03	
						7	0.06, 0.09 mean 0.07		0.04, 0.03	
						10	0.05, 0.04 mean 0.05		0.03, 0.03	
					Pulp	0	0.03, 0.03 mean 0.03	0.02	0.04, 0.05	
						3	0.04, 0.02 mean 0.03		0.04, 0.04	
						5	0.05, 0.03 mean 0.04		0.05, 0.04	
						7	0.04, 0.03 mean 0.03		0.04, 0.04	
						10	< 0.01, < 0.01 mean < 0.01		0.04, 0.05	
Ecuador, 2009 (Cavendish Gran Enano)	SC	0.122	29.3 - 29.4	8	Bagged Whole fruit	0	< 0.01, < 0.01 mean < 0.01	0.01	0.02, 0.02	
					Pulp		< 0.01, < 0.01 mean < 0.01	0.02	0.03, 0.03	
					Unbagged Whole fruit	0	0.05, 0.08 mean 0.07		0.02, 0.02	
					Pulp		0.07, 0.04 mean 0.05		0.03, 0.04	
Ecuador, 2009 (Williams)	SC	0.121 - 0.122	29.0 - 29.2	8	Bagged Whole fruit	0	0.01, 0.01 mean 0.01	< 0.01	0.03, 0.03	
					Pulp		< 0.01, < 0.01 mean < 0.01	0.02	0.04, 0.03	
					Unbagged Whole fruit	0	0.05, 0.09 mean 0.07		0.02, 0.03	
					Pulp		0.10, 0.09 mean 0.09		0.05, 0.05	
Guatemala, 2009 (Seda)	SC	0.123-0.130	29.6 - 31.1	8	Bagged Whole fruit	0	0.03, 0.05 mean 0.04	< 0.01	< 0.01,< 0.01	
					Pulp		0.03, 0.04 mean 0.04	< 0.01	< 0.01,< 0.01	
					Unbagged Whole fruit	0	0.10, 0.10 mean 0.10		0.01, 0.01	
					Pulp		0.09, 0.07 mean 0.08		< 0.01, < 0.01	

Banana country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg			Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		
								UTC	TRT	
Honduras, 2009 (Grand Naine)	SC	0.124 - 0.126	29.8 - 30.3	8	Bagged Whole fruit Pulp	0	< 0.01, < 0.01 mean < 0.01 < 0.01, < 0.01 mean < 0.01	< 0.01 < 0.01	0.01, 0.01 0.02, 0.02	
					Unbagged Whole fruit Pulp		0.02, < 0.01 mean 0.01 0.01, < 0.01 mean 0.01		0.01, < 0.01 0.01, 0.02	
Honduras, 2009 (Williams)	SC	0.124 - 0.126	29.8 - 30.3	8	Bagged Whole fruit Pulp	0	< 0.01, < 0.01 mean < 0.01 < 0.01, < 0.01 mean < 0.01	< 0.01 < 0.01	0.01, 0.01 0.02, 0.02	
					Unbagged Whole fruit Pulp		0.02, 0.02 mean 0.02 0.03, < 0.01 mean 0.02		< 0.01, < 0.01 0.02, 0.01	
Honduras, 2009 (Grand Naine)	SC	0.124 - 0.126	29.8 - 30.1	8	Bagged Whole fruit Pulp	0	< 0.01, < 0.01 mean < 0.01 < 0.01, < 0.01 mean < 0.01	< 0.01 < 0.01	0.01, < 0.01 0.01, 0.01	
					Unbagged Whole fruit Pulp		0.03, 0.02 mean 0.02 0.04, 0.04 mean 0.04		0.01, < 0.01 0.02, 0.02	

Sweet peppers

A total of eight trials were residue decline studies were conducted on sweet pepper from 2003 to 2004 in Spain in Valencia and Almeria. At each trial, three foliar spray applications using the SC formulation containing 250 g/L flutriafol, at a rate of approximately 18.75 g ai/hL under greenhouse conditions. The volume required for applying ranged between 700 and 1025 L/ha (equivalent to 0.133–0.189 kg ai/ha). Three spray applications have been performed at 10 days interval at BBCH 72–89. The applications were performed according to the Good Agricultural Practices.

At four trials in 2003 were including five sampling events day after the third application. The storage period was maximally 76–82 days. At four trials in 2004 were including three sampling events and two preserved samples, day after the third application. The storage period was maximally 10–35 days. No storage stability studies have been carried out on sweet peppers.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 0.1 mg/kg. The recoveries of flutriafol from sweet peppers fortified at 0.01 mg/kg were 83–90% and at 0.1 mg/kg were 86–99%. The limit of quantification (LOQ) was 0.01 mg/kg.

Table 63 Flutriafol residues on sweet peppers from supervised trials in Spain

Sweet peppers country, year (variety)	Application						PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
GAP, Spain	*SC*		0.019	*1000*		3	*1*		
Spain, 2003 (La Muyo)	SC	0.14 0.14 0.13	0.019	715 710 701	85 87 89	3	0 3 7 14 21	0.19 0.11 0.11 0.07 0.05	Valencia and Almeria, 2004 1264 FLU, 1265 FLU Storage interval: 76-82 days
Spain, 2003 (Hermimo)	SC	0.15 0.16 0.15	0.019	814 833 814	85 87 89	3	0 3 7 14 21	0.13 0.15 0.13 0.10 0.12	
Spain, 2003 (California Vardenas)	SC	0.15 0.15 0.15	0.019	803 803 771	85 87 89	3	0 3 7 14 21	0.23 0.26 0.16 0.14 0.09	
Spain, 2003 (Sympati)	SC	0.19 0.19 0.19	0.019	1000 1025 1000	85 87 89	3	0 3 7 14 21	0.41 0.32 0.31 0.19 0.09	
Spain, 2004 (Retama)	SC	0.19 0.19 0.19	0.019	1000 1000 985	73 73 74	3	0 3 7	0.24 0.19 0.09	Almeria, 2005 1268 FLU 1269 FLU Storage interval 10-39 days
Spain, 2004 (California Vardenas)	SC	0.19 0.19 0.19	0.019	1010 985 990	72 74 75	3	0 3 7	0.29 0.21 0.19	
Spain, 2004 (California Vardenas)	SC	0.19 0.19 0.19	0.019	1015 1000 995	72 73 75	3	0 3 7	0.32 0.27 0.19	
Spain, 2004 (Aifos)	SC	0.19 0.19 0.18	0.019	1010 1010 970	73 75 76	3	0 3 7	0.41 0.36 0.28	

Soya bean

Three residue trials were carried out in Brazil. All trials received 2 applications of flutriafol 125g/L SC at a rate of 0.125 kg ai/ha or 0.25 kg ai/ha with a 14 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.05 to 1.0 mg/kg. The recoveries of flutriafol from soya bean seeds fortified at 0.05 mg/kg were 91–109% and at 1.0 mg/kg were 103–112%. The limit of quantification (LOQ) was 0.05 mg/kg.

Table 64 Flutriafol residues on soya bean seeds from supervised trials in Brazil

Soya bean country, year (variety)	Application				PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	no.			
GAP, Brazil	*SC*	*0.125*	*200*	*2*	*28*		
Brazil, 2002 (Suprema)	SC	0.125	200	2	0 21 28	0.12 0.81 < 0.05	Ciscato & Gebara, 2002, 978 FLU Storage interval: 6-7 months
		0.25	200	2	28	0.16	
Brazil, 2002 (Stwart)	SC	0.125	200	2	28	< 0.05	Ciscato & Gebara, 2002, 979 FLU Storage interval: 7-8 months
		0.25	200	2	28	0.16	

Soya bean country, year (variety)	Application				PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	no.			
Brazil, 2002 (BR154)	SC	0.125	200	2	28	< 0.05	Ciscato & Gebara, 2002, 980 FLU Storage interval: 8-9 months
		0.25	200	2	28	0.13	

Twenty one residue trials were carried out in USA, in Arkansas, Georgia, Illinois, Iowa, Kansas, Louisiana, Minnesota, Missouri, North Dakota, Ohio, South Dakota, Virginia and Wisconsin. All trials received 3 applications of flutriafol 125g/L SC. Two applications were at a rate of 0.059–0.065 kg ai/ha and a third application were at 0.117–0.125 kg ai/ha. Applications were made with a 14 days interval. Some trials included supplementary plots for assessment of different application regimes. At a trial site in LA, a second treated plot was applied at 5× rate of 0.308, 0.314 and 0.621 mg ai/kg for generating processing samples.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.5 mg/kg and with triazole metabolite at levels ranging from 0.01 to 2.0 mg/kg. The concurrent recoveries of analytes from soya bean seeds were 61–138% for flutriafol, 81–100% for 1,2,4-Triazole (0.01 to 0.5 mg/kg), 87–101% for Triazole alanine (0.1 to 2.0 mg/kg), and 93–113% for Triazole acetic acid (0.01 to 0.5 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in any of the unprocessed and processed samples analysed.

Table 65 Flutriafol and triazole metabolites residues on soya bean seeds from supervised trials in USA

Soya bean country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	Kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
GAP, USA	*SC*	*0.064-0.128*	*> 94*	*3*	*21*	*Do not apply more than 0.256 kg ai/ha per season.*				
USA/ VA, 2005 (DK-B46-51RR)	SC	0.064 0.065 0.122	181 175 174	3	27	0.05, 0.04 mean 0.04	0.11	0.35, 0.19	< 0.01, 0.01	Rice, 2007 1468 FLU Storage interval: 19–163 days
		0.062 0.062 0.060	176 175 172	3	27	0.05, 0.05 mean 0.05	NA	NA-	NA-	
		0.061 0.062	174 175	2	27	0.02, 0.02 mean 0.02	NA	NA	NA	
USA/GA, 2005 (DK-H7242RR)	SC	0.062 0.061 0.123	234 228 230	3	22	0.04, 0.04 mean 0.04	0.07	0.23, 0.17	< 0.01, < 0.01	
USA/AR, 2005 (A495042)	SC	0.061 0.061 0.125	140 140 140	3	21	0.06, 0.05 mean 0.06	0.42	0.35, 0.34	0.03, 0.03	
USA/OH, 2005 (SC9373RR)	SC	0.063 0.063 0.125	140 143 147	3	23	0.19, 0.14 mean 0.17	0.07	0.20, 0.17	0.01, 0.01	
USA/OH, 2005 (SC9374RR)	SC	0.062 0.063 0.125	140 143 147	3	23	0.20, 0.19 mean 0.19	0.05	0.25, 0.26	< 0.01, < 0.01	
USA/IL, 2005 (Asgrow3202)	SC	0.061 0.061 0.124	172 163 155	3	22	0.01, 0.01 mean 0.01	0.13	0.18, 0.18	< 0.01, < 0.01	
		0.062 0.061 0.062	174 162 155	3	22	0.02, 0.02 mean 0.02	NA	NA	NA	
		0.061 0.061	163 153	2	22	< 0.01, < 0.01	NA	NA	NA	

Soya bean country, year (variety)	Application				PHI Days	Residues, mg/kg				Ref
	Form	Kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
						mean < 0.01				
USA/IL, 2005 (NK43-B1)	SC	0.061 0.062 0.123	172 173 184	3	21	0.13, 0.09 mean 0.11	0.13	0.24, 0.33	< 0.01, < 0.01	
USA/MN, 2005 (Pioneer3906)	SC	0.061 0.061 0.123	148 148 148	3	21	< 0.01, < 0.01 mean < 0.01	0.13	0.13, 0.13	< 0.01, < 0.01	
USA/WI, 2005 (Pioneer91M50)	SC	0.062 0.062 0.124	145 159 160	3	22	0.02, 0.03 mean 0.02	0.05	0.10, 0.10	< 0.01, < 0.01	
USA/WI, 2005 (Asgrow AG2403RR)	SC	0.061 0.061 0.123	144 143 151	3	21	< 0.01, 0.02 mean 0.02	0.08	0.10, 0.11	< 0.01, < 0.01	
USA/ND, 2005 (Mycogen)	SC	0.061 0.063 0.125	144 140 140	3	21	0.04, 0.05 mean 0.04	0.10	0.16, 0.15	< 0.01, < 0.01	
USA/ND, 2005 (Croplan RT0073)	SC	0.061 0.062 0.123	142 154 136	3	20	0.06, < 0.01 mean 0.04	0.09	0.04, 0.05	< 0.01, < 0.01	
USA/ND, 2005 (S00J4)	SC	0.063 0.062 0.123	142 142 141	3	21	0.02, 0.02 mean 0.02	1.3	0.67, 0.67	0.02, 0.02	
USA/SD, 2005 (Dekalb DKB06-51)	SC	0.061 0.061 0.123	140 140 140	3	23	0.03, 0.02 mean 0.03	0.03	0.16, 0.13	< 0.01, < 0.01	
USA/IA, 2005 (Pioneer93M93)	SC	0.061 0.060 0.123	149 137 137	3	22	0.07, 0.07 mean 0.07	0.09	0.16, 0.13	< 0.01, 0.01	
		0.062 0.063 0.059	149 137 133	3	22	0.06, 0.08 mean 0.07	NA	NA	NA	
		0.060 0.060	137 133	2	22	0.02, 0.06 mean 0.04	NA	NA	NA	
USA/KS, 2005 (Pioneer93B85)	SC	0.062 0.060 0.117	154 150 146	3	23	0.08, 0.05 mean 0.06	0.18	0.37, 0.37	0.02, 0.02	
USA/MO, 2005 (Pioneer93M50)	SC	0.063 0.059 0.117	145 135 136	3	22	0.08, 0.09 mean 0.08	0.03	0.21, 0.12	< 0.01, 0.01	
USA/MO, 2005 (AgrowAS3802)	SC	0.061 0.061 0.125	141 142 144	3	20	0.30, 0.31 mean 0.30	0.07	0.18, 0.16	0.02, 0.01	
USA/LA, 2005 (DPL5806RR)	SC	0.062 0.061 0.123	141 144 142	3	1	0.06, 0.06 mean 0.06	NA	NA	NA	
					7	0.04, 0.04 mean 0.04	NA	NA	NA	
					14	0.04, 0.05 mean 0.04	NA	NA	NA	
					21	0.07, 0.07 mean 0.07	0.04	0.28, 0.26	0.01, 0.01	
					28	0.08, 0.08 mean 0.08	0.05	0.29, 0.27	0.01, 0.01	
USA/IA, 2005	SC	0.062 0.062	124 135	3	1	0.26, 0.16 mean 0.21	NA	NA	NA	

Soya bean country, year (variety)	Application				PHI Days	Residues, mg/kg			Ref
	Form	Kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid
							UTC	TRT	
(Pioneer93B87)		0.123	129		7	0.10, 0.10 mean 0.10	NA	NA	NA
					14	0.11, 0.09 mean 0.10	NA	NA	NA
					21	0.06, 0.08 Mean 0.07	0.03	0.20, 0.20	< 0.01, < 0.01
					28	0.09, 0.06 mean 0.08	0.05	0.20, 0.19	< 0.01, < 0.01
USA/LA, 2005 (DP5634)	SC	0.061 0.061 0.123	143 153 148	3	21	0.05, 0.05 mean 0.05	0.08	0.10, 0.09	< 0.01, < 0.01
	SC	0.308 0.314 0.621	143 153 148	3	21	0.26, 0.27, 0.31 mean 0.28	0.08	0.22, 0.18, 0.20	< 0.01, < 0.01, < 0.01

UTC: untreated control samples, TRT: treated samples, NA: not analysed

Wheat

Flutriafol was applied to winter wheat at eight trials in North France and the UK (Northern Europe) and at eight trials in South France and Spain (Southern Europe). Eight trials were conducted for decline curve studies and the other eight for harvest studies. In all trials, 2 foliar applications of flutriafol (125 g/L SC formulation) were made at a rate of nominally 0.125 kg ai/ha with a 14 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.0 mg/kg. The recoveries of flutriafol from wheat grains fortified at 0.01 mg/kg were 80–97%, at 0.10 mg/kg were 80–95%, at 0.50 mg/kg were 99–101%, and at 1.0 mg/kg were 92–97%. The limit of quantification (LOQ) was 0.01 mg/kg.

Table 66 Flutriafol residues on wheat grains from supervised trials in Europe

Wheat country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	GS	no.			
GAP, Estonia *GAP, Lithuania* *GAP, Spain*	*SC* *SC* *SC*	*0.125* *0.125* *0.125*	 *300* *600*		 *2* *a*	 *30* *b*		
Northern Europe								
North France, 2002 (Courtot)	SC	0.125 0.125	300 300	45 - 47 61	2	42	0.02	Mende, 2003, 1048 FLU Storage interval: 156-248 days
North France, 2002 (Charger)	SC	0.123 0.125	294 299	45 - 47 59	2	42	0.04	
North France, 2002 (Rudal)	SC	0.122 0.124	293 297	49 59	2	35 42	< 0.01 < 0.01	
UK, 2002 (Consort)	SC	0.121 0.125	291 301	37 - 39 51 - 57	2	86	< 0.01	
North France, 2003 (Galibier)	SC	0.124 0.125	298 299	47 59	2	49	0.02	Jones, 2004, 1182 FLU Storage interval: 77-153 days
North France, 2003 (Apache)	SC	0.123 0.123	296 296	39 - 41 55	2	53	0.01	
North France, 2003 (Cezanne)	SC	0.125 0.124	301 298	43 59	2	55	0.02	

Wheat country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	GS	no.			
UK, 2003 (Claire)	SC	0.126 0.120	303 289	37 55	2	68	< 0.01	
Southern Europe								
South France, 2002 (Apache)	SC	0.125 0.125	300 299	51 55	2	42	0.02	Mende, 2003, 1047 FLU Storage interval: 203-265 days
South France, 2002 (Soissons)	SC	0.125 0.125	300 299	49 55	2	42	< 0.01	
Spain, 2002 (Cartaya)	SC	0.124 0.125	297 300	47 - 49 55 - 57	2	35 42	0.04 < 0.01	
Spain, 2002 (Cartaya)	SC	0.125 0.126	300 302	45 - 47 55	2	35 42	0.01 < 0.01	
South France, 2003 (Soissons)	SC	0.124 0.130	298 313	41 59	2	42	0.01	Jones, 2004, 1183 FLU Storage interval: 135-153 days
Spain, 2003 (Jabato)	SC	0.126 0.126	303 302	45 58 - 59	2	36	0.02	
Spain, 2003 (Marius)	SC	0.125 0.126	301 303	43 - 45 58 - 59	2	35	0.1	
South France, 2004 (Apache)	SC	0.124 0.127	297 304	43 65	2	42	< 0.01	Jones, 2004, 1184 FLU Storage interval: 51-63 days

[a] not necessary

[b] Between the end of stem elongation and flowering (BBCH: 37–61)

Twenty residue trials were carried out in USA, in Arkansas, Idaho, Illinois, Kansas, Minnesota, Montana, North Dakota, Nebraska, Oklahoma, South Dakota, Texas and Virginia. All trials received one seed treatment with 50 g/L SC at a rate of 30.1–49.6 mg ai/kg and four foliar applications of flutriafol 125 g/L SC at a rate of 0.124–0.134 kg ai/ha. Applications timing were 7 days prior to forage harvest (first application), 0 days before forage harvest (second application), 22 days prior to hay cutting (third application) and 7 days after third application (forth application). At a trial site in IL, a second treated plot was applied at 3X rate of 0.318–0.355 mg ai/kg to produce processing grain samples. At the KS and MN decline trials, decline samples were collected as follows: forage: 0, 1, 5, 10 and 14 days after second foliar application, hay: 0, 5, 10, 15 and 21 days after fourth foliar application, grain and straw: 5 days prior to and 0, 5 and 10 days after normal harvest maturity.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 5.0 mg/kg and with triazole metabolite at levels ranging from 0.01 to 1.2 mg/kg. The concurrent recoveries of analytes from wheat grains were 77–110% for flutriafol, 77–107% for 1,2,4-Triazole (0.01 and 0.5 mg/kg), 70–117% for Triazole alanine (0.01 to 1.2 mg/kg), and 70–116% for Triazole acetic acid (0.01 and 0.5 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in untreated and treated samples.

Table 67 Flutriafol and triazole metabolites residues on wheat grains from supervised trials in USA

Wheat country, year (variety)	Application				PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid		
							UTC	TRT	UTC	TRT	
GAP, USA	*SC*	*0.128*	*> 94*	*2*	*30*						
USA/ AR, 2009	SC	Treated Seed 36.0 mg/kg		1+4	29	0.09, 0.07 mean 0.08	0.02	0.06, 0.07	< 0.01	0.02, 0.02	Jones, 2010

Wheat country, year (variety)	Application				PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid		
							UTC	TRT	UTC	TRT	
(Progeny 10832)		0.129 0.129 0.129 0.129	310 310 309 309								1811 FLU Storage interval: Flutriafol 26-292 days Triazole metabolites 30-350 days
USA/ID, 2009 (Brundage)	SC	Treated Seed 36.9 mg/kg		1+4	70	0.05, 0.03 mean 0.04	0.02	0.28, 0.29	< 0.01	0.07, 0.09	
		0.129 0.129 0.128 0.127	311 301 307 308								
USA/IL, 2009 (Roane)	SC	Treated Seed 38.4 mg/kg		1+4	42	0.02, 0.02 mean 0.02	0.03	0.32, 0.39	0.01	0.09, 0.09	
		0.129 0.129 0.127 0.129	318 321 319 355								
		Treated Seed 38.4 mg/kg		1+4	42	0.10, 0.07 mean 0.09		1.1, 0.81		0.22, 0.20	
		0.387 0.383 0.381 0.387	319 318 319 354								
USA/KS, 2009 (Overley)	SC	Treated Seed 38.8 mg/kg		1+4	42	0.01, 0.01 mean 0.01	0.02	0.08 0.08	< 0.01	0.03 0.03	
		0.134 0.125 0.126 0.130	313 292 293 301								
USA/KS, 2009 (Fuller)	SC	Treated Seed 35.8 mg/kg		1+4	32	0.01, 0.02 mean 0.02	< 0.01	0.30, 0.29	< 0.01	0.08, 0.08	
		0.127 0.124 0.132 0.126	296 286 307 302								
USA/KS, 2009 (Overley)	SC	Treated Seed 38.8 mg/kg		1+4	42	< 0.01, < 0.01 mean < 0.01	0.01	0.21, 0.20	< 0.01	0.09, 0.08	
		0.128 0.129 0.131 0.128	299 302 305 297								
USA/KS, 2009 (Fuller)	SC	Treated Seed 38.8 mg/kg		1+4	39	0.02, 0.01 mean 0.02		0.16, 0.18		0.05, 0.06	
		0.130 0.130 0.125 0.132	307 303 295 299		44	0.02, 0.02 mean 0.02	< 0.01	0.13, 0.14	< 0.01	0.03, 0.04	
					49	0.01, 0.01 mean 0.01		0.17, 0.14		0.05, 0.04	
					54	0.01, 0.01 mean 0.01		0.12, 0.16		0.03, 0.05	
USA/MN, 2009 (Oklee)	SC	Treated Seed 49.6 mg/kg		1+4	32	< 0.01, < 0.01 mean < 0.01		0.10, 0.10		0.03, 0.02	
		0.128 0.128 0.128 0.128	328 329 328 328								
					37	< 0.01, < 0.01 mean < 0.01	0.03	0.09, 0.10	0.01	0.02, 0.03	

Wheat country, year (variety)	Application				PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid		
							UTC	TRT	UTC	TRT	
					42	< 0.01, < 0.01 mean < 0.01		0.09, 0.09		0.02, 0.02	
					48	< 0.01, < 0.01 mean < 0.01		0.09, 0.10		0.02, 0.03	
USA/MT, 2009 (Choteau)	SC	Treated Seed 30.1 mg/kg 0.128 326 0.121 298 0.130 324 0.128 332		1+4	48	0.03, 0.02 mean 0.03	< 0.01	0.11, 0.09	< 0.01	0.02, 0.02	
USA/ND, 2009 (Kelby)	SC	Treated Seed 38.1 mg/kg 0.128 328 0.128 327 0.128 328 0.126 326		1+4	38	< 0.01, < 0.01 mean < 0.01	0.06	0.18, 0.18	0.02	0.05, 0.05	
USA/ND, 2009 (Faller)	SC	Treated Seed 41.0 mg/kg 0.130 286 0.129 283 0.129 282 0.129 283		1+4	38	< 0.01, < 0.01 mean < 0.01	0.01	0.05, 0.05	< 0.01	0.03, 0.03	
USA/NE, 2009 (Certified Jagalene)	SC	Treated Seed 40.4 mg/kg 0.128 322 0.125 329 0.126 318 0.129 325		1+4	41	0.04, 0.02 mean 0.02	0.04	0.22, 0.29	0.03	0.08, 0.11	
USA/NE, 2009 (Certified Jagalene)	SC	Treated Seed 40.4 mg/kg 0.127 319 0.126 331 0.127 321 0.128 323		1+4	44	< 0.01, < 0.01 mean < 0.01	0.04	0.29, 0.35	0.01	0.06, 0.09	
USA/OK, 2009 (Jagger)	SC	Treated Seed 42.2 mg/kg 0.127 322 0.130 346 0.134 353 0.124 327		1+4	45	0.06, 0.04 mean 0.05	0.06	0.55, 0.51	0.03	0.10, 0.09	
USA/OK, 2009 (Jagger)	SC	Treated Seed 42.2 mg/kg 0.128 335 0.126 344 0.129 356 0.131 346		1+4	48	0.03, 0.04 mean 0.03	0.04	0.67, 0.66	0.02	0.18, 0.18	
USA/SD, 2009 (Harding)	SC	Treated Seed 38.0 mg/kg 0.127 317 0.129 366 0.127 336 0.128 351		1+4	60	0.01, 0.01 mean 0.01	0.05	0.11, 0.11	0.02	0.02, 0.02	
USA/SD, 2009	SC	Treated Seed 40.8 mg/kg		1+4	47	< 0.01, < 0.01	0.03	0.12, 0.15	0.01	0.06, 0.07	

Wheat country, year (variety)	Application				PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid		
							UTC	TRT	UTC	TRT	
(Briggs HRS)		0.130 0.128 0.127 0.129	296 319 291 334			mean < 0.01					
USA/TX, 2009 (Fannin)	SC	Treated Seed 39.8 mg/kg		1+4	37	0.01, 0.01 mean 0.01	0.09	0.45, 0.43	0.04	0.23, 0.22	
		0.128 0.128 0.127 0.129	300 296 287 294								
USA/TX, 2009 (TAM105)	SC	Treated Seed 42.7 mg/kg		1+4	43	0.01, 0.02 mean 0.02	< 0.01	0.08, 0.11	< 0.01	0.05, 0.05	
		0.128 0.128 0.125 0.126	331 330 322 325								
USA/VA, 2009 (Coker 9436)	SC	Treated Seed 39.2 mg/kg		1+4	31	0.06, 0.06 mean 0.06	0.09	0.23, 0.24	0.05	0.07, 0.07	
		0.128 0.130 0.130 0.131	336 337 339 341								

UTC: untreated control samples, TRT: treated samples, NA: not analysed

Peanut

Thirteen residue trials were carried out in USA, in Georgia, Florida, Oklahoma, Texas and Virginia. All trials received 5 applications of flutriafol 125g/L SC at a rate of 0.118–0.128 kg ai/ha. Applications were made with a 14 days interval. At a trial site in FL, a second treated plot was applied at 5× rate of 0.605–0.615 mg ai/kg for generating processing samples.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 0.50 mg/kg and with triazole metabolite at levels ranging from 0.01 to 3.6 mg/kg. The concurrent recoveries of analytes from peanut nutmeats were 94–115% for flutriafol, 71–89% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 62–123% for Triazole alanine (0.04 to 3.6 mg/kg), and 82–105% for Triazole acetic acid (0.01 and 0.10 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in nutmeat. Triazole acetic acid residues of nutmeat were < 0.01 mg/kg in untreated samples.

Table 68 Flutriafol and triazole metabolites residues on peanut nutmeat from supervised trials in USA

Peanut country, year (variety)	Application				PHI Days	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
GAP, USA	*SC*	*0.128*	*> 94*	*4*	*7*					
USA/VA, 2007 (Georgia Green)	SC	0.122 0.121 0.122 0.118 0.120	304 303 303 303 303	5	7	0.01, 0.01 mean <u>0.01</u>	0.26	0.48, 0.62	< 0.01, < 0.01	Rice, 2008 1605 FLU Storage interval: Flutriafol 49-153days
USA/VA, 2007 (Georgia Green)	SC	0.119 0.122 0.120 0.125	289 289 289 289	5	7	< 0.01, < 0.01 mean <u>< 0.01</u>	0.26	0.58, 0.60	< 0.01, 0.01	

Peanut country, year (variety)	Application				PHI Days	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
		0.125	289							Triazole metabolites 99-216 days
USA/GA, 2007 (Georgia 02C)	SC	0.125 0.123 0.122 0.123 0.125	262 262 262 262 262	5	8	0.04, 0.04 mean <u>0.04</u>	0.09	0.26, 0.25	< 0.01, < 0.01	
USA/GA, 2007 (Georgia Green)	SC	0.125 0.122 0.123 0.124 0.123	262 262 262 262 262	5	8	0.04, 0.04 mean <u>0.04</u>	0.10	0.26, 0.23	< 0.01, < 0.01	
USA/GA, 2007 (C-99R)	SC	0.128 0.122 0.122 0.123 0.123	262 262 262 262 262	5	7	0.03, 0.04 mean <u>0.03</u>	0.07	0.18, 0.24	< 0.01, < 0.01	
USA/GA, 2007 (Georgia Green)	SC	0.124 0.123 0.122 0.123 0.123	262 262 262 262 262	5	1 8 14 21 28	0.04, 0.03 mean 0.03 0.04, 0.02 mean <u>0.03</u> 0.02, 0.02 mean 0.02 0.02, 0.02 mean 0.02 0.02, 0.02 mean 0.02	0.10 0.10 0.07 NA NA	0.31, 0.24 0.30, 0.24 0.15, 0.15 0.18, 0.18 0.12, 0.14	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	
USA/GA, 2007 (GA-02)	SC	0.118 0.120 0.118 0.120 0.119	188 187 188 183 189	5	7	0.02, 0.03 mean <u>0.02</u>	0.65	0.60, 0.58	< 0.01, < 0.01	
USA/GA, 2007 (GA-02)	SC	0.118 0.120 0.118 0.120 0.119	188 187 188 183 189	5	7	0.02, 0.02 mean <u>0.02</u>	0.67	0.71, 0.75	< 0.01, < 0.01	
USA/FL, 2007 (GA-Green)	SC	0.120 0.120 0.118 0.121 0.119	187 187 188 183 189	5	7	0.01, 0.01 mean <u>0.01</u>	0.39	0.78, 0.50	< 0.01, < 0.01	
USA/OK, 2007 (Olin)	SC	0.121 0.122 0.123 0.124 0.125	225 225 229 227 228	5	6	0.02, 0.02 mean <u>0.02</u>	0.10	0.41, 0.41	< 0.01, < 0.01	
		0.622 0.605 0.608 0.615 0.612	225 225 229 227 228	5	6	0.19, 0.19 mean 0.19	0.09	1.8, 1.5	0.01, 0.01	
USA/TX, 2007 (TAM SPAN)	SC	0.124 0.123 0.123 0.123 0.122	238 234 234 234 234	5	7	< 0.01, < 0.01 mean <u>< 0.01</u>	0.01	0.08, 0.06	< 0.01, < 0.01	

Peanut country, year (variety)	Application				PHI Days	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.		Flutriafol	Triazole alanine		Triazole acetic acid	
							UTC	TRT		
USA/TX, 2007 (Tamrun OL-1)	SC	0.123 0.123 0.121 0.122 0.123	235 235 231 232 235	5	1 7 15 22 28	0.08, 0.06 mean 0.07 0.07, 0.05 mean 0.06 0.04, 0.07 mean 0.05 0.09, 0.07 mean <u>0.08</u> 0.06, 0.06 mean 0.06	0.25 NA NA NA NA	1.8, 2.0 2.4, 2.2 2.2, 2.1 2.0, 2.1 2.4, 2.6	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	
USA/GA, 2007 (GA-Green)	SC	0.118 0.120 0.118 0.120 0.119	188 187 188 183 189	5	7	0.02, 0.02 mean <u>0.02</u>	NA	NA	NA	

UTC: untreated control samples, TRT: treated samples, NA: not analysed

Coffee beans

Four field trails were conducted on coffee from 2001 to 2002 in Brazil using the SC formulation containing 125 g/L flutriafol. In all the trials, Initial application was made to the soil using undiluted product equivalent to 0.69 kg ai/ha at BBCH 55 followed by two foliar applications made to plants at 0.25 kg ai/ha at a 30 day interval at BBCH 78–82. In each trial, additional plot was treated with the application rates of 1.4 kg ai/ha for direct soil application and 0.5 kg ai/ha for the following two foliar applications, which were twice those of the critical GAP.

The recoveries of flutriafol from coffee beans fortified at 0.05 mg/kg were 81–95% and at 0.5 mg/kg were 80–113%.The storage period was maximally 7–9 months. No storage stability studies have been carried out on coffee beans.

Table 69 Flutriafol residues on coffee beans from supervised trials in Brazil

Coffee country, year (variety)	Application						Portion analysed	PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.				
GAP, Brazil	*SC*	*0.69* *0.25*	*12.5* *0.05*	*500*	*55*	3[a]		*30*[b]		
Brazil, 2001 (Catuai)	SC	0.69 0.25 0.25	12.5 0.05 0.05	500 500	55 78 80	3	Beans	0 25 30 45	0.07 0.07 <u>< 0.05</u> < 0.05	Ciscato and Gebara, 2003 989 FLU Storage interval: 226-272 months
		1.4 0.5 0.5	12.5 0.1 0.1	500 500	55 78 80	3	Beans	30 45	0.06 0.06 c < 0.05	
Brazil, 2001 (Mundo Novo)	SC	0.69 0.25 0.25	12.5 0.05 0.05	500 500	58 80 82	3	Beans	30	<u>< 0.05</u>	Trevizan and Baptista, 2003 975 FLU Storage interval: 224 days
		1.4 0.5 0.5	12.5 0.1 0.1	500 500	58 80 82	3	Beans	30	< 0.05 c < 0.05	
Brazil, 2001 (Catuai)	SC	0.69 0.25 0.25	12.5 0.05 0.05	500 500	55 78 80	3	Beans	30	<u>< 0.05</u>	Trevizan and Baptista, 2003 976 FLU Storage interval:
		1.4 0.5	12.5 0.1	500	55 78	3	Beans	30	< 0.05	

Coffee country, year (variety)	Application						Portion analysed	PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.				
		0.5	0.1	500	80				c < 0.05	231 days
Brazil, 2001 (Mundo Novo)	SC	0.69 0.25 0.25	12.5 0.05 0.05	 500 500	59 79 81	3	Beans	30	< 0.05	Trevizan and Baptista, 2003 977 FLU Storage interval: 224 days
		1.4 0.5 0.5	12.5 0.1 0.1	 500 500	59 79 81	3	Beans	30	< 0.05 c < 0.05	

[a] One at 0.69 kg ai/ha directly to soil using undiluted product before BBCH 55 and two at 0.25 kg ai/ha to plants at a 30 day interval

[b] The PHI of 30 days after final spray application to plants is equivalent to 120 days after direct application to soil

Five field trials were conducted on coffee during the 2009/2010 growing season in Brazil, Colombia, Guatemala and Vietnam using the SC formulation containing 125 g/L flutriafol. The first application was made at a rate of 0.138 g ai/plant to the soil at the base of each coffee plant at the BBCH 55 to 60 growth stage. The second and third applications were foliar applied 58–60 and 29–30 days before harvest at 0.25 kg ai/ha. The retreatment interval between first and second applications ranged from 123 to 202 days.

The analytical method was validated with analyses by spiking control samples with flutriafol and triazole metabolite at fortification levels ranging from 0.01 to 1.0 mg/kg. The concurrent recoveries of analytes from coffee beans were 74–112% for flutriafol, 72–95% for 1,2,4-Triazole, 85–115% for Triazole alanine, and 93–114% for Triazole acetic acid. The limit of quantification (LOQ) was 0.01 mg/kg.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in coffee beans.

Table 70 Flutriafol and triazole metabolites residues on coffee beans from supervised trials in Brazil, Colombia, Guatemala and Vietnam

Coffee country, year (variety)	Application					PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water,L/ha	GS	no		Flutriafol	Triazole alanine		Triazole acetic acid		
								UTC	TRT	UTC	TRT	
GAP, Brazil	*SC*	*0.69* *0.25*	500	*55*	*3*[a]	*30*						
Brazil, 2009 (Obata)	SC	0.14[b] 0.25 0.25	 485 514	55 77 81	3	29	0.08, 0.12 mean 0.10	0.09	0.18, 0.20	< 0.01	< 0.01, < 0.01	Carringer, 2011
						34	0.06, 0.08 mean 0.07		0.20, 0.19		< 0.01, < 0.01	Storage interval:
						39	0.08, 0.06 mean 0.07		0.40, 0.18		< 0.01, < 0.01	Flutriafol 14-59 days
						44	0.05, 0.07 mean 0.06		0.21, 0.28		< 0.01, < 0.01	Triazole metabolites 14-282 days
						49	0.08, 0.07 mean 0.07		0.34, 0.43		< 0.01, < 0.01	
						59	0.07, 0.09 mean		0.25, 0.28		< 0.01, < 0.01	

Coffee country, year (variety)	Application					PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water,L/ha	GS	no		Flutriafol	Triazole alanine		Triazole acetic acid		
								UTC	TRT	UTC	TRT	
							0.08					
Brazil, 2009 (Obata)	SC	0.14[b] 0.25 0.25	 494 513	55 77 81	3	29	0.04, 0.03 mean 0.03	< 0.01	0.03, 0.04	< 0.01	< 0.01, < 0.01	
						44	0.03, 0.04 mean 0.04		0.04, 0.07		< 0.01, < 0.01	
Colombia, 2010 (Castillo)	SC	0.14[b] 0.25 0.25	 515 503	55 75 85	3	30	0.01, 0.01 mean 0.01	< 0.01	0.06, 0.06	< 0.01	< 0.01, < 0.01	
						45	< 0.01, 0.01 mean 0.01		0.04, 0.04		< 0.01, < 0.01	
Guatemala, 2010 (Caturra)	SC	0.13[b] 0.26 0.25	 514 503	55 77 81	3	30	0.04, 0.03 mean 0.03	0.01	0.12, 0.09	< 0.01	< 0.01, < 0.01	
						44	0.04, 0.06 mean 0.05		0.17, 0.16		< 0.01, < 0.01	
Vietnam, 2010 (Catimor)	SC	0.14[b] 0.25 0.25	 513 517	60 79 88	3	29	0.10, 0.09 mean 0.09	0.35	0.90, 0.89	0.01	0.02, 0.03	
						44	0.03, 0.03 mean 0.03		0.84, 0.52		0.02, 0.02	

UTC: untreated control samples, TRT: treated samples

[a] One at 0.69 kg ai/ha directly to soil using undiluted product before BBCH 55 and two at 0.25 kg ai/ha to plants at a 30 day interval

[b] g ai/plant

Straw, fodder and forage of cereals

Wheat forage and straw

Flutriafol was applied to winter wheat at eight trials in North France and the UK (Northern Europe) and at eight trials in South France and Spain (Southern Europe). Eight trials were conducted for decline curve studies and the other eight for harvest studies. In all trials, 2 foliar applications of flutriafol (125 g/L SC formulation) were made at a rate of nominally 0.125 kg ai/ha with a 14 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 10 mg/kg. The concurrent recoveries of flutriafol from whole plant fortified ranged from 0.01 to 10 mg/kg were 75–99%. The recoveries from wheat straw fortified ranged from 0.01 to 5.0 mg/kg were 80–100%. The recoveries from wheat ears fortified at levels of 0.01 and 0.10 mg/kg were 71–91%. The recoveries from residual plant fortified ranged from

0.01 to 0.30 mg/kg were 89–103%. The recoveries from wheat stems fortified at levels of 0.01 and 0.50 mg/kg were 94–101%. The limit of quantification (LOQ) was 0.01 mg/kg.

Table 71 Flutriafol residues on wheat forage and straw from supervised trials in Europe

Wheat country, year (variety)	Application					Portion analysed	PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	GS	no.				
GAP, Estonia	*SC*	*0.125*							
GAP, Lithuania	*SC*	*0.125*	*300*		*2*		*30*		
GAP, Spain	*SC*	*0.125*	*600*		*a*		*b*		
Northern Europe									
North France, 2002 (Courtot)	SC	0.125 0.125	300 300	45-47 61	2	Whole plant Straw	0 7 14 21 35 42	1.5 0.42 0.35 0.22 <u>0.17</u> 0.41	Mende, 2003, 1048 FLU
North France, 2002 (Charger)	SC	0.123 0.125	294 299	45-47 59	2	Whole plant Straw	0 7 14 21 35 42	1.5 0.53 0.36 0.24 <u>0.16</u> 0.44	
North France, 2002 (Rudal)	SC	0.122 0.124	293 297	49 59	2	Whole plant Straw	0 7 14 21 35 42	1.4 0.34 0.27 0.18 <u>0.44</u> 0.35	
UK, 2002 (Consort)	SC	0.121 0.125	291 301	37-39 51-57	2	Whole plant Ears Residual plant Straw	0 7 14 21 35 42 56 56 86	1.5 0.42 0.28 0.22 <u>0.16</u> 0.13 0.01 0.11 0.36	
North France, 2003 (Galibier)	SC	0.124 0.125	298 299	47 59	2	Whole plant Ears Stems Straw	0 42 42 49	3.8 0.32 0.42 1.4	Jones, 2004, 1182 FLU
North France, 2003 (Apache)	SC	0.123 0.123	296 296	39-41 55	2	Whole plant Ears Stems Straw	0 42 42 53	2.3 0.14 0.28 0.48	
North France, 2003 (Cezanne)	SC	0.125 0.124	301 298	43 59	2	Whole plant Ears Stems Straw	0 42 42 55	2.5 0.35 0.36 2.4	
UK, 2003 (Claire)	SC	0.126 0.120	303 289	37 55	2	Whole plant Ears Stems Straw	0 42 42 68	2.5 0.31 0.02 0.28	
Southern Europe									
South France, 2002 (Apache)	SC	0.125 0.125	300 299	51 55	2	Whole plant Straw	0 7 14 21 35 42	1.0 0.42 0.15 0.14 < 0.01 <u>0.15</u>	Mende, 2003, 1047 FLU

Wheat country, year (variety)	Application					Portion analysed	PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	GS	no.				
South France, 2002 (Soissons)	SC	0.125 0.125	300 299	49 55	2	Whole plant Straw	0 7 14 21 35 42	1.1 0.39 0.21 0.10 0.12 0.35	
Spain, 2002 (Cartaya)	SC	0.124 0.125	297 300	47-49 55-57	2	Whole plant Straw	0 7 14 21 35 42	2.8 1.8 0.82 0.56 1.5 0.86	
Spain, 2002 (Cartaya)	SC	0.125 0.126	300 302	45-47 55	2	Whole plant Straw	0 7 14 21 35 42	1.4 0.74 0.48 0.46 0.55 0.49	
South France, 2003 (Soissons)	SC	0.124 0.130	298 313	41 59	2	Whole plant Straw	0 42	3.8 1.9	Jones, 2004, 1183 FLU
Spain, 2003 (Jabato)	SC	0.126 0.126	303 302	45 58-59	2	Whole plant Straw	0 36	2.9 4.1	
Spain, 2003 (Marius)	SC	0.125 0.126	301 303	43-45 58-59	2	Whole plant Straw	0 35	5.7 3.6	
South France, 2004 (Apache)	SC	0.124 0.127	297 304	43 65	2	Whole plant Straw	0 42	2.3 1.4	Jones, 2004, 1184 FLU

[a] not necessary

[b] Between the end of stem elongation and flowering (BBCH: 37–61)

Twenty residue trials were carried out in USA, in Arkansas, Idaho, Illinois, Kansas, Minnesota, Montana, North Dakota, Nebraska, Oklahoma, South Dakota, Texas and Virginia. All trials received one seed treatment with 50 g/L SC at a rate of 30.1–49.6 mg ai/kg and four foliar applications of flutriafol 125 g/L SC at a rate of 0.124–0.134 kg ai/ha. Applications timing were 7 days prior to forage harvest (first application), 0 days before forage harvest (second application), 22 days prior to hay cutting (third application) and 7 days after third application (forth application). At the KS and MN decline trials, decline samples were collected as follows: forage: 0, 1, 5, 10 and 14 days after second foliar application, hay: 0, 5, 10, 15 and 21 days after fourth foliar application, grain and straw: 5 days prior to and 0, 5 and 10 days after normal harvest maturity.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 25 mg/kg and with triazole metabolite at levels of 0.01 and 0.5 mg/kg. The concurrent recoveries of analytes from wheat forage were 77–117% for flutriafol (0.01 to 25 mg/kg), 71–92% for 1,2,4-Triazole, 75–109% for Triazole alanine, and 74–108% for Triazole acetic acid. The recoveries from wheat hay were 75–113% for flutriafol (0.01 to 15 mg/kg), 72–100% for 1,2,4-Triazole, 77–106% for Triazole alanine, and 75–119% for Triazole acetic acid. The recoveries from wheat straw were 75–106% for flutriafol (0.01 to 10 mg/kg), 71–94% for 1,2,4-Triazole, 70–102% for Triazole alanine, and 75–114% for Triazole acetic acid. The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of 1,2,4-Triazole were found above the LOQ in untreated and treated samples.

Table 72 Flutriafol and triazole metabolites residues on wheat forage, hay and straw from supervised trials in USA

Wheat country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		Triazole acetic acid		
								UTC	TRT	UTC	TRT	
GAP, USA	*SC*	*0.128*	*> 94*	*2*		*30*						
USA/ AR, 2009 (Progeny 10832)	SC	Treated Seed 36.0 mg/kg 0.129 0.129 0.129 0.129	310 310 309 309	1+4	Forage Hay Straw	0[a] 16 29	3.9, 4.3 mean 4.1 3.9, 4.4 mean 4.1 1.1, 1.3 mean 1.2	0.02 < 0.01 < 0.01	0.01, 0.01 0.02, 0.02 0.01, < 0.01	< 0.01 0.02 < 0.01	< 0.01,< 0.01 0.02, 0.02 < 0.01, < 0.01	Jones, 2010 1811 FLU
USA/ID, 2009 (Brundage)	SC	Treated Seed 36.9 mg/kg 0.129 0.129 0.128 0.127	311 301 307 308	1+4	Forage Hay Straw	0[a] 27 70	5.7, 5.6 mean 5.7 0.62, 0.59 mean 0.60 0.61, 0.66 mean 0.63	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.12, 0.14 0.02, 0.02	< 0.01 < 0.01 < 0.01	< 0.01, < 0.01 0.08, 0.08 0.03, 0.04	
USA/IL, 2009 (Roane)	SC	Treated Seed 38.4 mg/kg 0.129 0.129 0.127 0.129	318 321 319 355	1+4	Forage Hay Straw	0[a] 20 42	5.5, 4.9 mean 5.2 0.91, 1.0 mean 0.96 1.0, 1.1 mean 1.1	< 0.01 0.01 < 0.01	0.02, 0.02 0.10, 0.14 0.12, 0.14	< 0.01 0.01 0.01	< 0.01, < 0.01 0.06, 0.07 0.06, 0.07	
USA/KS, 2009 (Overley)	SC	Treated Seed 38.8 mg/kg 0.134 0.125 0.126 0.130	313 292 293 301	1+4	Forage Hay Straw	0[a] 19 42	12, 14 mean 13 2.8, 2.8 mean 2.8 0.91, 0.79 mean 0.85	< 0.01 0.03 < 0.01	< 0.01, < 0.01 0.07, 0.07 0.01, 0.01	< 0.01 0.03 0.01	< 0.01, < 0.01 0.05, 0.04 0.02, 0.02	
USA/KS, 2009 (Fuller)	SC	Treated Seed 35.8 mg/kg 0.127 0.124 0.132 0.126	296 286 307 302	1+4	Forage Hay Straw	0[a] 19 32	13, 8.4 mean 11 1.6, 1.6 mean 1.6 0.47, 0.57 mean 0.52	< 0.01 < 0.01 < 0.01	< 0.01, < 0.01 0.28, 0.17 0.18, 0.16	< 0.01 < 0.01 < 0.01	< 0.01, < 0.01 0.08, 0.06 0.09, 0.10	
USA/KS, 2009 (Overley)	SC	Treated Seed 38.8 mg/kg 0.128 0.129 0.131 0.128	299 302 305 297	1+4	Forage Hay Straw	0[a] 19 42	17, 15 mean 16 1.6, 1.5 mean 1.6 0.41, 0.36 mean 0.38	< 0.01 < 0.01 < 0.01	< 0.01, < 0.01 0.09, 0.15 0.02, 0.01	< 0.01 < 0.01 < 0.01	< 0.01, < 0.01 0.08, 0.11 0.04, 0.04	

Wheat country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		Triazole acetic acid		
								UTC	TRT	UTC	TRT	
USA/KS, 2009 (Fuller)	SC	Treated Seed 38.8 mg/kg		1+2	Forage	0[a]	8.3, 8.5 mean 8.4	< 0.01	< 0.01, < 0.01	< 0.01	< 0.01, < 0.01	
		0.130	307								< 0.01, < 0.01	
		0.130	303			1	2.0, 2.0 mean 2.0		< 0.01, < 0.01		< 0.01, < 0.01	
		0.125	295									
		0.132	299			5	1.6, 1.2 mean 1.4		< 0.01, < 0.01		< 0.01, < 0.01	
											< 0.01, < 0.01	
						10	1.0, 1.0 mean 1.0		< 0.01, < 0.01		0.03, 0.03	
						14	0.62, 0.65 mean 0.64		< 0.01, < 0.01		0.02, 0.03	
											0.04, 0.04	
				1+4	Hay	3	15, 18 mean 17		0.05, 0.06		0.03, 0.03	
						8	7.8, 6.0 mean 6.9		0.04, 0.04		0.03, 0.03	
											0.04, 0.02	
						11	3.8, 4.3 mean 4.0		0.07, 0.07		0.02, 0.03	
						21	2.1, 2.0 mean 2.0	< 0.01	0.05, 0.05	< 0.01	0.02, 0.03	
											0.01, 0.02	
						23	1.9, 1.8 mean 1.9		0.09, 0.06			
				1+4	Straw	39	0.42, 0.50 mean 0.46		0.06, 0.04			
						44	0.66, 0.48 mean 0.57	< 0.01	0.02, 0.02	< 0.01		
						49	0.39, 0.43 mean 0.41		0.02, 0.02			
						54	0.47, 0.39 mean 0.43		0.01, 0.01			
USA/MN, 2009 (Oklee)	SC	Treated Seed 49.6 mg/kg		1+2	Forage	0[a]	8.6, 6.5 mean 7.6	0.01	0.02, 0.02	< 0.01	< 0.01, < 0.01	
		0.128	328								< 0.01, < 0.01	
		0.128	329			1	1.8, 1.5 mean 1.7		0.02, 0.02		< 0.01, < 0.01	
		0.128	328									
		0.128	328			5	0.51, 0.46 mean 0.49		0.01, 0.02		< 0.01, < 0.01	
											< 0.01, < 0.01	
						10	0.15, 0.10 mean 0.12		0.01, 0.01		0.02, 0.02	
						14	0.12, 0.13 mean 0.12		< 0.01, < 0.01		0.03, 0.03	
											0.02, 0.03	
				1+4	Hay	2	14, 13 mean 14		0.02, 0.02		0.01, 0.02	
						7	3.3, 3.5 mean 3.4		0.05, 0.04		0.02, 0.01	
											0.01, 0.02	

Wheat country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		Triazole acetic acid		
								UTC	TRT	UTC	TRT	
						12	2.4, 2.4 mean 2.4		0.04, 0.06		0.02, 0.02	
						18	1.3, 1.5 mean 1.4	< 0.01	< 0.01, 0.03	0.01	0.02, 0.02	
						23	1.3, 1.5 mean 1.4		0.03, 0.03		0.02, 0.02	
				1+4	Straw	32	0.29, 0.36 mean 0.32		< 0.01, < 0.01			
						37	0.40, 0.41 mean 0.40	< 0.01	< 0.01, < 0.01	0.01		
						42	0.39, 0.41 mean 0.40		< 0.01, < 0.01			
						48	0.30, 0.27 mean 0.27		< 0.01, < 0.01			
USA/MT, 2009 (Choteau)	SC	Treated Seed 30.1 mg/kg		1+4	Forage	0[a]	6.1, 4.2 mean 5.1	< 0.01	< 0.01, < 0.01	< 0.01	< 0.01, < 0.01	
		0.128	326								0.02, 0.02	
		0.121	298		Hay	20	4.6, 3.8 mean 4.2	< 0.01	0.03, 0.05	< 0.01	< 0.01, < 0.01	
		0.130	324									
		0.128	332									
					Straw	48	1.1, 0.14 mean 0.63	< 0.01	0.01, 0.01	< 0.01		
USA/ND, 2009 (Kelby)	SC	Treated Seed 38.1 mg/kg		1+4	Forage	0[a]	5.7, 5.5 mean 5.6	0.02	0.04, 0.04	< 0.01	< 0.01, < 0.01	
		0.128	328								0.04, 0.03	
		0.128	327		Hay	17	1.0, 1.0 mean 1.0	0.04	0.08, 0.08	0.01	0.04, 0.04	
		0.128	328									
		0.126	326									
					Straw	38	0.15, 0.19 mean 0.17	< 0.01	0.06, 0.06	0.01		
USA/ND, 2009 (Faller)	SC	Treated Seed 41.0 mg/kg		1+4	Forage	0[a]	8.8, 9.5 mean 9.2	< 0.01	0.01, 0.01	< 0.01	< 0.01, < 0.01	
		0.130	286								0.02, 0.03	
		0.129	283		Hay	16	3.4, 3.3 mean 3.3	0.02	0.04, 0.04	< 0.01	0.01, 0.01	
		0.129	282									
		0.129	283									
					Straw	38	0.53, 0.61 mean 0.57	< 0.01	< 0.01, < 0.01	< 0.01		
USA/NE, 2009 (Certified Jagalene)	SC	Treated Seed 40.4 mg/kg		1+4	Forage	0[a]	11, 11 mean 11	0.02	0.03, 0.03	< 0.01	< 0.01, < 0.01	
		0.128	322								0.07, 0.08	
		0.125	329		Hay	21	1.4, 1.7 mean 1.5	0.02	0.09, 0.13	0.03	0.07, 0.07	
		0.126	318									
		0.129	325									
					Straw	41	0.67, 0.65 mean 0.66	< 0.01	0.04, 0.03	0.02		
USA/NE, 2009 (Certified Jagalene)	SC	Treated Seed 40.4 mg/kg		1+4	Forage	a	8.4, 8.7 mean 8.6	< 0.01	0.01, 0.01	< 0.01	< 0.01, < 0.01	
		0.127	319								0.07, 0.08	
		0.126	331		Hay	21	1.9, 2.2 mean 2.1	0.01	0.12, 0.12	< 0.01	0.06, 0.08	
		0.127	321									
		0.128	323									
					Straw	44	0.47, 0.41 mean 0.44	< 0.01	0.06, 0.07	< 0.01		

Wheat country, year (variety)	Application				Portion analysed	PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol	Triazole alanine		Triazole acetic acid		
								UTC	TRT	UTC	TRT	
USA/OK, 2009 (Jagger)	SC	Treated Seed 42.2 mg/kg		1+4	Forage	0[a]	7.5, 7.1 mean 7.3	0.02	0.02, 0.02	< 0.01	< 0.01, < 0.01	
		0.127	322								0.07, 0.07	
		0.130	346		Hay	20	2.1, 2.0 mean 2.1	0.01	0.14, 0.13	0.02	0.06, 0.08	
		0.134	353									
		0.124	327									
					Straw	45	1.7, 2.1 mean 1.9	< 0.01	0.09, 0.11	0.02		
USA/OK, 2009 (Jagger)	SC	Treated Seed 42.2 mg/kg		1+4	Forage	0[a]	3.9, 7.0 mean 5.5	0.01	0.02, 0.02	< 0.01	< 0.01, < 0.01	
		0.128	335								0.16, 0.18	
		0.126	344		Hay	20	2.9, 2.6 mean 2.8	0.02	0.30, 0.33	0.02	0.07, 0.07	
		0.129	356									
		0.131	346									
					Straw	48	2.8, 2.5 mean 2.7	< 0.01	0.06, 0.05	< 0.01		
USA/SD, 2009 (Harding)	SC	Treated Seed 38.0 mg/kg		1+4	Forage	0[a]	3.0, 5.5 mean 4.2	0.03	0.02, 0.02	< 0.01	< 0.01, < 0.01	
		0.127	317								0.02, 0.02	
		0.129	366		Hay	21	2.1, 1.9 mean 2.0	0.02	0.02, 0.02	0.03	0.02, 0.02	
		0.127	336									
		0.128	351									
					Straw	60	0.50, 0.57 mean 0.53	< 0.01	0.02, 0.03	0.01		
USA/SD, 2009 (Briggs HRS)	SC	Treated Seed 40.8 mg/kg		1+4	Forage	0[a]	7.7, 11 mean 9.3	< 0.01	0.01, 0.02	< 0.01	< 0.01, < 0.01	
		0.130	296								0.09, 0.08	
		0.128	319		Hay	19	1.3, 1.2 mean 1.3	0.01	0.11, 0.09	0.01	0.03, 0.04	
		0.127	291									
		0.129	334									
					Straw	47	0.11, 0.08 mean 0.10	< 0.01	0.02, 0.02	< 0.01		
USA/TX, 2009 (Fannin)	SC	Treated Seed 39.8 mg/kg		1+4	Forage	0[a]	20, 18 mean 19	0.01	0.01, 0.02	*< 0.01*	< 0.01, < 0.01	
		0.128	300								0.20, 0.21	
		0.128	296		Hay	20	2.2, 2.7 mean 2.4	0.05	0.21, 0.25	*0.04*	0.05, 0.10	
		0.127	287									
		0.129	294									
					Straw	37	0.84, 1.7 mean 1.3	< 0.01	0.04, 0.08	*0.01*		
USA/TX, 2009 (TAM105)	SC	Treated Seed 42.7 mg/kg		1+4	Forage	0[a]	11, 14 mean 13	< 0.01	< 0.01, < 0.01	< 0.01	< 0.01, < 0.01	
		0.128	331								0.07, 0.06	
		0.128	330		Hay	21	12, 9.6 mean 9.6	< 0.01	0.06, 0.04	< 0.01	0.02, 0.02	
		0.125	322									
		0.126	325									
					Straw	43	8.6, 8.5 mean 8.5	< 0.01	< 0.01, < 0.01	< 0.01		
USA/VA, 2009 (Coker 9436)	SC	Treated Seed 39.2 mg/kg		1+4	Forage	0[a]	4.3, 3.7 mean 4.0	0.04	0.05, 0.05	0.01	0.01, 0.02	
		0.128	336								0.05, 0.05	
		0.130	337		Hay	20	2.9, 3.0 mean 2.9	0.02	0.09, 0.10	0.03	0.06, 0.04	
		0.130	339									
		0.131	341									
					Straw	31	0.75, 1.6 mean 1.2	< 0.01	0.03, 0.02	0.02		

UTC: untreated control samples, TRT: treated samples, NA: not analysed

[a] Forage samples were collected as soon as spray had dried after the second foliar application.

Miscellaneous fodder and forage crops

Peanut fodder

Thirteen residue trials were carried out in USA, in Georgia, Florida, Oklahoma, Texas and Virginia. All trials received 5 applications of flutriafol 125g/L SC at a rate of 0.118–0.128 kg ai/ha. Applications were made with a 14 days interval.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 20 mg/kg and with triazole metabolite at levels ranging from 0.01 to 0.8 mg/kg. The concurrent recoveries of analytes from peanut hay were 88–119% for flutriafol, 73–94% for 1,2,4-Triazole, 60–115% for Triazole alanine, and 71–84% for Triazole acetic acid. The limit of quantification (LOQ) was 0.01 mg/kg.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. Low residues of 1,2,4-Triazole (0.02 mg/kg) were found in hay.

Table 73 Flutriafol and triazole metabolites residues on peanut hay from supervised trials in USA

Peanut country, year (variety)	Application				PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no		Flutriafol	1,2,4-Triazole	Triazole alanine		Triazole acetic acid	
								UTC	TRT		
GAP, USA	*SC*	*0.128*	*> 94*	*4*	*7*						
USA/VA, 2007 (Georgia Green)	SC	0.122 0.121 0.122 0.118 0.120	304 303 303 303 303	5	7	4.5, 4.1 mean 4.3	< 0.01, < 0.01	0.02	0.07, 0.05	0.06, 0.04	Rice, 2008 1605 FLU
USA/VA, 2007 (Georgia Green)	SC	0.119 0.122 0.120 0.125 0.125	289 289 289 289 289	5	7	3.3, 3.0 mean 3.1	< 0.01, < 0.01	0.02	0.03, 0.02	0.04, 0.04	
USA/GA, 2007 (Georgia 02C)	SC	0.125 0.123 0.122 0.123 0.125	262 262 262 262 262	5	8	10.2, 7.49 mean 8.8	< 0.01, < 0.01	0.01	0.10, 0.07	0.02, 0.02	
USA/GA, 2007 (Georgia Green)	SC	0.125 0.122 0.123 0.124 0.123	262 262 262 262 262	5	8	6.5, 8.8 mean 7.7	< 0.01, < 0.01	0.02	0.11, 0.11	0.02, 0.02	
USA/GA, 2007 (C-99R)	SC	0.128 0.122 0.122 0.123 0.123	262 262 262 262 262	5	7	8.1, 6.6 mean 7.3	< 0.01, < 0.01	0.02	0.08, 0.06	0.02, 0.03	
USA/GA, 2007 (Georgia Green)	SC	0.124 0.123 0.122 0.123 0.123	262 262 262 262 262	5	1 8 14 21 28	5.8, 8.5 mean 7.2 8.1, 7.5 mean 7.8 9.1, 8.8 mean 8.9 2.4, 1.2 mean 1.8 2.2, 1.3 mean 1.8	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01 NA NA	0.05, 0.03 0.07, 0.04 0.05, 0.04 0.04, 0.03 0.02, 0.02	0.03, 0.02 0.03, 0.03 0.03, 0.03 0.03, 0.03 0.02, 0.02	

Peanut country, year (variety)	Application				PHI Days	Residues, mg/kg					Ref
	Form	kg ai/ha	water, L/ha	no		Flutriafol	1,2,4-Triazole	Triazole alanine		Triazole acetic acid	
								UTC	TRT		
USA/GA, 2007 (GA-02)	SC	0.118 0.120 0.118 0.120 0.119	188 187 188 183 189	5	7	1.6, 1.8 mean 1.7	< 0.01, < 0.01	0.06	0.10, 0.08	0.07, 0.07	
USA/GA, 2007 (GA-02)	SC	0.118 0.120 0.118 0.120 0.119	188 187 188 183 189	5	7	2.0, 3.2 mean 2.6	< 0.01, < 0.01	0.06	0.11, 0.11	0.07, 0.07	
USA/FL, 2007 (GA-Green)	SC	0.120 0.120 0.118 0.121 0.119	187 187 188 183 189	5	7	2.4, 1.8 mean 2.1	< 0.01, < 0.01	0.08	0.10, 0.10	0.11, 0.10	
USA/OK, 2007 (Olin)	SC	0.121 0.122 0.123 0.124 0.125	225 225 229 227 228	5	6	2.0, 2.0 mean 2.0	< 0.01, < 0.01	< 0.01	0.03, 0.05	< 0.01, 0.01	
USA/TX, 2007 (TAM SPAN)	SC	0.124 0.123 0.123 0.123 0.122	238 234 234 234 234	5	7	0.85, 0.63 mean 0.74	< 0.01, < 0.01	< 0.01	< 0.01, 0.01	< 0.01, < 0.01	
USA/TX, 2007 (Tamrun OL-1)	SC	0.123 0.123 0.121 0.122 0.123	235 235 231 232 235	5	1 7 15 22 28	1.8, 1.3 mean 1.5 1.2, 1.8 mean 1.5 0.75, 1.1 mean 0.93 0.41, 0.44 mean 0.43 0.73, 0.91 mean 0.82	< 0.01, < 0.01 0.01, 0.02 0.02, 0.02 < 0.01, < 0.01 < 0.01, 0.01	< 0.01 NA NA NA NA	0.16, 0.20 0.17, 0.19 0.31, 0.29 0.37, 0.37 0.31, 0.30	0.05, 0.05 0.05, 0.05 0.06, 0.05 0.06, 0.06 0.06, 0.07	
USA/GA, 2007 (GA-Green)	SC	0.118 0.120 0.118 0.120 0.119	188 187 188 183 189	5	7	2.7, 2.3 mean 2.5	NA	NA	NA	NA	

UTC: untreated control samples, TRT: treated samples, NA: not analysed

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on the fate of flutriafol residues during the processing of apples, grapes, soya bean seeds, wheat grains and peanut nutmeat.

Apple, grape, soya bean, wheat and peanut of the crops that the Meeting received information on supervised field trials may be processed prior to consumption. Processing factors have been calculated for flutriafol residues in apple, grape, soya bean, wheat and peanut.

Apple

The Meeting received a processing study for apple fruits which were conducted in USA, in New York. The trials received 6 applications of flutriafol 125g/L SC at the application rate of 0.12 kg ai/ha with a 14 days interval. Additional plot was treated with the first three applications at the nominal application rate of 0.12 kg ai/ha and the last three applications at the 2× rate of 0.24 kg ai/ha.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 0.20 mg/kg and with triazole metabolite at levels ranging from 0.01 to 0.50 mg/kg. The concurrent recoveries of analytes from apple juice were 71–86% for flutriafol, 97–98% for 1,2,4-Triazole, 92–98% for Triazole alanine, and 99–117% for Triazole acetic acid respectively. The recoveries from apple wet pomace were 84–100% for flutriafol, 88–92% for 1,2,4-Triazole, 78–96% for Triazole alanine, and 112–115% for Triazole acetic acid. The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Table 74 Flutriafol and triazole metabolites residues in processed commodities of apples from supervised trials

Apples country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol		Triazole alanine, mg/kg	Triazole acetic acid, mg/kg	
							mg/kg	PF			
USA/NY, 2006 (Ida Red)	SC	0.12	939-953	6	14	Fruit	0.08		0.03	< 0.01	Willard, 2007 1471 FLU
						Juice	0.04	0.50	0.02	< 0.01	
						Wet pomace	0.15	1.9	0.03	< 0.01	
						Dry pomace[a]	0.80	10	0.14	0.04	
		0.12 and 0.24	933-942	6	14	Fruit	0.11		0.03	< 0.01	
						Juice	0.05	0.45	0.02	< 0.01	
						Wet pomace	0.21	1.9	0.03	< 0.01	
						Dry pomace[b]	0.93	8.5	0.12	< 0.01	

[a] Dry pomace calculated from wet pomace residue and 81.0% moisture.

[b] Dry pomace calculated from wet pomace residue and 77.4% moisture.

Grapes

Processing trial was established in typical grape growing areas in USA, in California (CA). At the site in CA, a second treated plot was applied at 2× rate of 0.256 kg ai/ha for generating processing samples.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 2.0 mg/kg and with triazole metabolite at levels ranging from 0.01 to 0.25 mg/kg. The concurrent recoveries of analytes from sun-dried grapes were 78–112% for flutriafol, 91–102% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 93–97% for Triazole alanine (0.02 and 0.20 mg/kg), and 91–99% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from raisins were 96–100% for flutriafol, 80–84% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 90–98% for Triazole alanine (0.02 and 0.20 mg/kg), and 85–98% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from grape juice were 93–98% for flutriafol, 61–87% for Triazole (0.01 and 0.10 mg/kg), 71–96% for Triazole alanine (0.025 and 0.25 mg/kg), and 94–107% for Triazole acetic acid (0.01 and 0.10 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Table 75 Flutriafol and triazole metabolites residues in processed commodities of grapes from supervised trials

Grapes country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol		Triazole alanine, mg/kg	Triazole acetic acid, mg/kg	
							mg/kg	PF			
USA/CA, 2007 (Thompson Seedless)	SC	0.256	1164-1186	7	14	Fruit	0.34, 0.45 mean 0.40		0.04, 0.04	< 0.01, < 0.01	Rice, 2008 1606 FLU
						Sun-dried Grapes	1.4, 0.79 mean 1.1	2.8	0.09, 0.06	0.01, < 0.01	
						Raisins	1.0, 1.1 mean 1.1	2.8	0.09, 0.09	0.01, 0.01	
						Juice	0.26, 0.24 mean 0.25	0.63	0.03, 0.02	< 0.01, < 0.01	

Sweet peppers

Processing trials were conducted in Spain. From four trials in 2004, the preserved samples were obtained from treated plot samples at 3 and 7 days after last application.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 0.1 mg/kg. The recoveries of flutriafol from sweet peppers fortified at 0.01 mg/kg were 83–90% and at 0.1 mg/kg were 86–99%. The limit of quantification (LOQ) was 0.01 mg/kg.

Table 76 Flutriafol residues in processed commodity of sweet peppers from supervised trials

Sweet peppers country, year (variety)	Application					PHI Days	Residues, mg/kg		Processing factor	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		Raw	Preserved		
Spain, 2004 (Retama)	SC	0.19 0.19 0.19	0.019	1000 1000 985	3	3 7	0.19 0.09	0.14 0.10	0.74 1.1	Almeria, 2005 1268 FLU 1269 FLU
Spain, 2004 (California Vardenas)	SC	0.19 0.19 0.19	0.019	1010 985 990	3	3 7	0.21 0.19	0.27 0.15	1.3 0.79	
Spain, 2004 (California Vardenas)	SC	0.19 0.19 0.19	0.019	1015 1000 995	3	3 7	0.27 0.19	0.20 0.26	0.74 1.4	
Spain, 2004 (Aifos)	SC	0.19 0.19 0.18	0.019	1010 1010 970	3	3 7	0.36 0.28	0.24 0.16	0.67 0.57	

Soya bean

A processing trial was carried out in the USA, in Louisiana (LA). The trial received 3 applications of flutriafol 125g/L SC. Applications were made with a 14 days interval. At a trial site in LA, a treated plot was applied at 5× rate of 0.308, 0.314 and 0.621 mg ai/kg for generating processing samples.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 5.0 mg/kg and with triazole metabolite at levels ranging from 0.01 to 1.0 mg/kg. The concurrent recoveries of analytes from soya bean meal were 96–115% for flutriafol (0.05 and 0.10 mg/kg), 80–88% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 83–88% for Triazole alanine (0.5 and 1.0 mg/kg), and 95–99% for Triazole acetic acid (0.05 and 0.50 mg/kg). The recoveries from hulls were 82–106% for flutriafol (0.01 and 0.10 mg/kg), 79–93% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 73–89% for Triazole alanine (0.10 and 1.0 mg/kg), and 100–101% for

Triazole acetic acid (0.02 and 0.20 mg/kg). The recoveries from refined oil were 98–111% for flutriafol (0.01 and 0.10 mg/kg), 87–90% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 102–106% for Triazole alanine (0.01 and 0.10 mg/kg), and 103–107% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from aspirated grain fraction were 106–117% for flutriafol (0.50 and 5.0 mg/kg), 89–90% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 94–97% for Triazole alanine (0.2 and 1.0 mg/kg), and 105–107% for Triazole acetic acid (0.10 and 1.0 mg/kg).The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Table 77 Flutriafol and triazole metabolites residues in processed commodities of soya bean seeds from supervised trials

Soya bean country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol		Triazole alanine, mg/kg	Triazole acetic acid, mg/kg	
							mg/kg	PF			
USA/LA, 2005 (DP5634)	SC	0.308 0.314 0.621	143 153 148	3	21	Seed	0.30, 0.29 mean 0.29		0.18, 0.19	< 0.01, < 0.01	Rice, 2007 1468 FLU
						Meal	0.40, 0.38 mean 0.39	1.3	0.29, 0.29	0.13, 0.12	
						Hulls	0.34, 0.21 mean 0.28	0.97	0.13, 0.20	< 0.01, 0.01	
						Refined oil	0.38, 0.36 mean 0.37	1.3	< 0.01, < 0.01	< 0.01, < 0.01	
						Aspirated grain fraction	< 0.50 (3) mean < 0.50	1.7	< 0.01, < 0.01	< 0.01, < 0.01	

Residues below LOQ were calculated as LOQ value.

Wheat

Processing studies were carried out in USA, in Illinois (IL). At a trial site in IL, a treated plot was applied at 3× rate of 0.318–0.355 mg ai/kg to produce processing grain samples.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels of 0.01 and 5.0 mg/kg, and with triazole metabolite at levels ranging from 0.01 to 3.4 mg/kg. The concurrent recoveries of analytes from wheat aspirated grain fraction were 93–106% for flutriafol, 86–103% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 70–76% for Triazole alanine (0.06 and 0.50 mg/kg), and 89–92% for Triazole acetic acid (0.06 and 0.50 mg/kg). The recoveries from wheat bran were 88% for flutriafol, 78–85% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 70–77% for Triazole alanine (0.20 to 3.4 mg/kg), and 87–89% for Triazole acetic acid (0.04 and 0.50 mg/kg). The recoveries from wheat flour were 86–93% for flutriafol, 72–85% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 81–85% for Triazole alanine (0.03 and 0.50 mg/kg), and 75–79% for Triazole acetic acid (0.03 and 0.50 mg/kg). The recoveries from wheat middlings were 88% for flutriafol, 88–91% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 74–86% for Triazole alanine (0.08 to 1.0 mg/kg), and 88% for Triazole acetic acid (0.04 and 0.50 mg/kg). The recoveries from wheat shorts were 88–93% for flutriafol, 84–86% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 70–78% for Triazole alanine (0.2 to 1.8 mg/kg), and 77–79% for Triazole acetic acid (0.04 and 0.50 mg/kg). The recoveries from wheat germ were 103–106% for flutriafol, 89–92% for 1,2,4-Triazole (0.01 and 0.50 mg/kg), 73–83% for Triazole alanine (0.2 to 3.4 mg/kg), 84–91% for Triazole acetic acid (0.04 and 0.50 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg for all analytes.

Table 78 Flutriafol and triazole metabolites residues in processed commodities of wheat grains from supervised trials

Wheat country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Flutriafol		Triazole alanine, mg/kg	Triazole acetic acid, mg/kg	
							mg/kg	PF			
USA/IL, 2009 (Roane)	SC	Treated Seed 38.4 mg/kg		1+4	42	Grain	0.02, 0.02 mean 0.02		0.32, 0.39	0.09, 0.09	Jones, 2010 1811 FLU
		0.129 0.129 0.127 0.129	318 321 319 355			Aspirated grain fraction	0.26, 0.26 mean 0.26	13	0.17, 0.18	0.13, 0.13	
		Treated Seed 38.4 mg/kg		1+4	42	Grain	0.09		0.84	0.16	
		0.387 0.383 0.381 0.387	319 318 319 354			Bran	0.20, 0.19 mean 0.19	2.1	2.3, 3.3	0.30, 0.38	
						Flour	0.03, 0.03 mean 0.03	0.33	0.24, 0.22	0.11, 0.11	
						Middlings	0.07, 0.08 mean 0.08	0.89	0.81, 0.89	0.24, 0.24	
						Shorts	0.11, 0.12 mean 0.12	1.3	1.3, 1.1 2.9, 2.8	0.30, 0.20	
						Germ	0.25, 0.26 mean 0.25	2.8		0.34, 0.34	

Peanut

Thirteen residue trials were carried out in USA, in Georgia, Florida, Oklahoma, Texas and Virginia. All trials received 5 applications of flutriafol 125g/L SC at a rate of 0.118–0.128 kg ai/ha. Applications were made with a 14 days interval. At a trial site in FL, a second treated plot was applied at 5× rate of 0.605–0.615 mg ai/kg for generating processing samples.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels of 0.01 and 0.50 mg/kg and with triazole metabolite at levels ranging from 0.01 to 3.8 mg/kg. The concurrent recoveries of analytes from peanut meal were 115% for flutriafol, 94–96% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 79–111% for Triazole alanine (0.20 to 3.8 mg/kg), and 86–95% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from refined oil were 101–114% for flutriafol, 75–95% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 71–95% for Triazole alanine (0.01 and 0.10 mg/kg), and 87–90% for Triazole acetic acid (0.01 and 0.10 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of T were found above the LOQ in processed commodities.

Table 79 Flutriafol and triazole metabolites residues in processed commodities of peanut nutmeats from supervised trials

Peanut country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Fultriafol		Triazole alanine, mg/kg	Triazole acetic acid, mg/kg	
							mg/kg	PF			
USA/OK, 2007 (Olin)	SC	0.622 0.605 0.608 0.615 0.612	225 225 229 227 228	5	6	Nutmeat	0.19, 0.19 mean 0.19		1.8, 1.5	0.01, 0.01	Rice, 2008 1605 FLU

Peanut country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Fultriafol		Triazole alanine, mg/kg	Triazole acetic acid, mg/kg	
							mg/kg	PF			
						Meal	0.10, 0.20 mean 0.15	0.79	1.9, 2.7	0.02, 0.03	
						Refined Oil	0.25, 0.27 mean 0.26	1.4	< 0.01, < 0.01	< 0.01, < 0.01	

Coffee

Five field trials were conducted on coffee during the 2009/2010 growing season in Brazil, Colombia, Guatemala and Vietnam using the SC formulation containing 125 g/L flutriafol. At the Colombia test site, an additional treated plot was established for the processing phase. This plot received three 5× rate applications at 0.69 g ai/plant to the soil at the base of each coffee plant at the BBCH 55 to 60 growth stage and at 1.25 kg ai/ha with foliar applications 58–60 and 29–30 days before harvest.

The analytical method was validated with analyses by spiking control samples with flutriafol at fortification levels ranging from 0.01 to 1.0 mg/kg and with triazole metabolite at fortification levels ranging from 0.01 to 0.50 mg/kg. The concurrent recoveries of analytes from cleaned green beans were 82–106% for flutriafol, 107–108% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 74–82% for Triazole alanine (0.01 and 0.10 mg/kg), and 86–97% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from roasted coffee beans were 82–83% for flutriafol, 83–102% for 1,2,4-Triazole (0.01 to 0.25 mg/kg), 101–120% for Triazole alanine (0.01 and 0.10 mg/kg), and 109–117% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from spent coffee grounds were 87–92% for flutriafol, 120% for 1,2,4-Triazole (0.01 and 0.10 mg/kg), 80–91% for Triazole alanine (0.01 and 0.10 mg/kg), and 100–110% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from liquor extract were 78–80% for flutriafol, 95–117% for 1,2,4-Triazole (0.01 to 0.35 mg/kg), 91–109% for Triazole alanine (0.01 and 0.10 mg/kg), and 70–104% for Triazole acetic acid (0.01 and 0.10 mg/kg). The recoveries from instant coffee were 85–89% for flutriafol, 113–120% for 1,2,4-Triazole (0.05 and 0.50 mg/kg), 76–101% for Triazole alanine (0.01 and 0.10 mg/kg), and 71–97% for Triazole acetic acid (0.01 and 0.10 mg/kg). The limit of quantification (LOQ) was 0.01 mg/kg.

Residue data also included residues on metabolites 1,2,4-Triazole, Triazole alanine and Triazole acetic acid. No residues of Triazole acetic acid were found above the LOQ in processed commodities from coffee beans.

Table 80 Flutriafol and triazole metabolites residues in processed commodities of coffee beans from supervised trials

Coffee country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Fultriafol		1,2,4-Triazole, mg/kg	Triazole alanine, mg/kg	
							mg/kg	PF			
Colombia, 2010 (Castillo)	SC	0.695[a] 1.24 1.23	514 504	3	30	Green coffee beans	0.21, 0.18 mean 0.19		< 0.01, < 0.01	0.05, 0.03	Carringer, 2011
						Cleaned green beans	0.22, 0.17 mean 0.19	1.0	< 0.01, < 0.01	0.04, 0.03	
						Roasted coffee beans	0.17, 0.18 mean 0.18	0.95	< 0.01, < 0.01	< 0.01, < 0.01	
						Spent coffee	0.02, 0.02	0.11	< 0.01, < 0.01	< 0.01, < 0.01	

Coffee country, year (variety)	Application				PHI Days	Commodity	Residues				Ref
	Form	kg ai/ha	water, L/ha	no.			Fultriafol		1,2,4-Triazole, mg/kg	Triazole alanine, mg/kg	
							mg/kg	PF			
						grounds	mean 0.02				
						Liqour extract	0.17, 0.16 mean 0.16	0.84	0.02, 0.02	< 0.01, < 0.01	
						Instant coffee	0.48, 0.58 mean 0.53	2.8	0.07, 0.08	0.02, 0.02	

[a] g ai/plant

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received lactating dairy cow and laying hen feeding studies.

Lactating dairy cow

Flutriafol was administered orally (within gelatin capsules containing only ethyl cellulose) with a balling gun to lactating Holstein dairy cows for 29 consecutive days (Willard, 2008: 1566 FLU). A total of 10 cows were used in the study. The treatment groups were Group I (control, 1 cow), II (1×; 0.5 ppm flutriafol based on the diet dry weight, 3 cows), III (3×; 1.5 ppm, 3 cows), and IV (10×; 5.0 ppm, 3 cows). Milk was collected from each cow twice daily and the weight of the milk was recorded. A day's milk production was defined as the PM milk, collected following the morning dosing, combined with the AM milk, collected just prior to the next morning's dosing. Pooled milk samples (AM combined with PM milk based on the proportion of the total milk produced at each milking) were collected for analysis from each animal on each of the following study days: 0 (pre-dosing), 3, 7, 10, 14, 17, 21, 24, 26 and 28. Each cow was sacrificed within 24 hours after the final dosing (study day 29). At termination, samples of muscle (collected from the thigh and loin), liver, kidney and fat were collected from each cow. Each composite sample was homogenized in the presence of dry ice in a blender. Each processed sample was divided into two equivalent subsamples, weighed, and placed back into freezer storage until shipment or disposal. The residues of flutriafol and triazole metabolites [1,2,4-Triazole, Triazole alanine, Triazole acetic acid] were determined using two unique methods.

No flutriafol residues were detected in any milk samples collected from the untreated control cow or any of the samples collected from the 10× dose group cows. None of the other milk samples from the 1× or 3× dose group were analysed. None of the milk samples collected from the untreated control cow or from the 10× dose group cows had residues of any of the triazole metabolites at level > LOQ (0.01 mg/kg). None of the other milk samples from the 1× or 3× dose group were analysed.

No flutriafol residues were detected in any untreated control animal samples of muscle, liver, kidney or fat. No flutriafol residues were detected in the muscle, kidney or fat samples from any of the animals in the 10 × dose group or the 1× dose group. There were flutriafol residues > LOQ in the liver samples from all the animals from 1×, 3× and 10× dosing groups. None of the tissue samples collected from the 1× and 10× dose groups had residues of any of the triazole metabolites at level > LOQ (0.01 mg/kg).

Freshly-fortified control samples were analysed with each analytical set to monitor the procedural recovery of flutriafol and triazole metabolites. Concurrent procedural recoveries of flutriafol were 73–103% for milk, 83–110% for liver, 99–107% for kidney, 79–119% for muscle and 71–106% for fat. Those of 1,2,4-triazole were 81–104% for milk, 75–107% for liver, 84–92% for kidney, 79–95% for muscle and 84–103% for fat. Those of triazole alanine were 81–124% for milk,

78–89% for liver, 83–109% for kidnet, 71–93% for muscle and 84–103% for fat. Those of triazole acetic acid were 72–109% for milk, 71–85% for liver, 71–80% for kidney, 82–105% for muscle and 75–91% for fat. The LOQ for flutriafol and each of the triazole metabolites in all matrices was 0.01 mg/kg. The results of the flutriafol and triazole metabolites analyses in the milk, liver, kidney, muscle and fat samples are presented in Table 81.

Table 81 Residues of flutriafol and triazole metabolites in tissues and milk

Dose (ppm dw feed)	Analyte	Residue, mg/kg				
		Milk*	Liver	Muscle	Kidney	Fat
Control	Flutriafol	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	1,2,4-Triazole	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Triazole alanine	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	Triazole acetic acid	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
0.5	Flutriafol	na	< 0.01, 0.02, 0.04	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	1,2,4-Triazole	na	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole alanine	na	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole acetic acid	na	< 0.01 (3)	< 0.01	< 0.01 (3)	< 0.01 (3)
1.5	Flutriafol	na	0.09, 0.09, 0.10	na	na	na
	1,2,4-Triazole	na	na	na	na	na
	Triazole alanine	na	na	na	na	na
	Triazole acetic acid	na	na	na	na	na
5.0	Flutriafol	< 0.01 (3)	0.23, 0.23, 0.39	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	1,2,4-Triazole	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole alanine	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole acetic acid	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)

*Results taken from analysis at 0, 3, 7, 10, 14, 17, 21, 24, 26 and 29 days for 10 × (5.0 ppm) dose group.

na: not analysed

Laying hen

Flutriafol was administered orally (within gelatin capsules containing only lactose) with a balling gun to laying hens (*Gallus domesticus*, Hy-Line 36) for 29 consecutive days (Willard, 2008: 1567 FLU). A total of 40 hens were used in the study. The treatment groups were Group I (control), II (1 ×; 0.5 ppm flutriafol based on the diet dry weight), III (3 ×; 1.5 ppm), and IV (10 ×; 5.0 ppm) for 10 hens per treatment group. Eggs were collected from each hen twice daily and the number of eggs was recorded. Eggs were collected for sample analysis on study days 0, 3, 7, 10, 14, 17, 21, 24, 26 and 28. For these samples, at each collection event, all eggs from the control group were pooled. The eggs collected from the treated groups (Group II and IV) were pooled by subgroup (A, B, C). The egg yolk and white were put into a container and thoroughly mixed. The mixture was divided into two equivalent subsamples (a, b), weighed, and placed into freezer storage until shipment or disposal. Each hen was sacrificed within 24 hours after the final dosing (study day 29). At termination, samples of muscle (collected from the legs and breast), liver and abdominal fat were collected from each hen. The tissue from each hen was weighed, cubed, and pooled by treatment Group-subgroup and placed into labelled bags. These bags were placed into freezer storage until processing. Each composite sample was homogenized in the presence of dry ice in a blender. Each processed sample was divided into two equivalent subsamples, weighed, and placed back into freezer storage until shipment or disposal. The residues of flutriafol and triazole metabolites [1,2,4-Triazole, Triazole alanine, Triazole acetic acid] were determined using two unique methods.

No flutriafol residues were detected in any untreated control group egg samples. Initially, the 10× dose group samples were analysed. After it was found that the samples from this dose group had flutriafol residue (> LOQ), the samples from the 1× dose group were analysed. The highest flutriafol residue was 0.04 mg/kg from a 10 × dose group sample collected at study day 17. None of the egg samples from the 1× dose group had flutriafol residues > LOQ (0.01 mg/kg). None of the egg samples collected from the 10× dose group had residues of any of triazole metabolites at level > LOQ (0.01 mg/kg). None of the egg samples analysed from 1× dose group (day 0, 7, 14, 21 and 28) had residues of any of triazole metabolites at level > LOQ.

Freshly-fortified control samples were analysed with each analytical set to monitor the procedural recovery of flutriafol and triazole metabolites. Concurrent procedural recoveries for eggs were 72–95% for flutriafol, 71–92% for 1,2,4-triazole, 81–113% for triazole alanine, 73–89% for triazole acetic acid. The LOQ for flutriafol and each of the triazole metabolites in all matrices was 0.01 mg/kg. The results of the flutriafol and triazole metabolites analyses in the egg samples are presented in Table 82.

Table 82 Residues of flutriafol and triazole metabolites in eggs

Dose (ppm dw feed)	Study day	Residue, mg/kg			
		Flutriafol	1,2,4-Triazole	Triazole alanine	Triazole acetic acid
Control	0	< 0.01	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01	< 0.01
	7	< 0.01	< 0.01	< 0.01	< 0.01
	10	< 0.01	na	na	na
	14	< 0.01	< 0.01	< 0.01	< 0.01
	17	na	na	na	na
	21	< 0.01	< 0.01	< 0.01	< 0.01
	24	< 0.01	na	na	na
	28	< 0.01	< 0.01	< 0.01	< 0.01
0.5	0	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	3	na	na	na	na
	7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	10	na	na	na	na
	14	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	17	na	na	na	na
	21	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	24	na	na	na	na
	28	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
1.5		na	na	na	na
5.0	0	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	3	0.02 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	7	0.03 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	10	0.03, 0.03, 0.02	na	na	na
	14	0.03, 0.03, 0.04	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	17	0.03, 0.03, 0.04	na	na	na
	21	0.02, 0.03, 0.03	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	24	0.03 (3)	na	na	na
	28	0.03 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)

No flutriafol residues were detected inany untreated control group samples of muscle, liver or fat. Initially, the 10× dose group samples were analysed. After it was found that the samples from this dose group had significant flutriafol residue (> LOQ), the samples from the 1× dose group were analysed. The highest flutriafol residue in liver sample from the 10× dose group was 0.10 mg/kg. The highest flutriafol residue in fat sample from the 10× dose group was 0.07 mg/kg. None of the tissue samples from the 1× dose group had flutriafol residues > LOQ (0.01 mg/kg).

Freshly-fortified control samples were analysed with each analytical set to monitor the procedural recovery of flutriafol and triazole metabolites. Concurrent procedural recoveries of flutriafol were 71–109% for liver, 83–99% for muscle and 66–83% for fat. Those of 1,2,4-triazole were 72–85% for liver, 75–101% for muscle and 84–104% for fat. Those of triazole alanine were 78–115% for liver, 84–104% for muscle and 90–97% for fat. Those of triazole acetic acid were 73–77% for liver, 74–92% for muscle and 70–89% for fat. The LOQ for flutriafol and each of the triazole metabolites in all matrices was 0.01 mg/kg. The results of the flutriafol and triazole metabolites analyses in the liver, muscle and fat samples are presented in Table 83.

Table 83 Residues of flutriafol and triazole metabolites in tissues

Dose (ppm dw feed)	Analyte	Residue, mg/kg		
		Liver	Muscle	Fat
Control	Flutriafol	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	1,2,4-Triazole	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole alanine	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole acetic acid	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
0.5	Flutriafol	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	1,2,4-Triazole	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole alanine	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole acetic acid	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
1.5	Flutriafol	na	na	na
	1,2,4-Triazole	na	na	na
	Triazole alanine	na	na	na
	Triazole acetic acid	na	na	na
5.0	Flutriafol	0.10, 0.07, 0.03	< 0.01 (3)	0.06, 0.07, 0.05
	1,2,4-Triazole	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole alanine	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
	Triazole acetic acid	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)

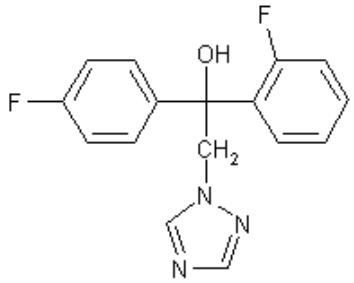
APPRAISAL

Residue and analytical aspects of flutriafol were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2011 JMPR by the Forty-second Session of the CCPR (ALINORM 10/33/24)

Flutriafol is a triazole fungicide used in many crops for control of a broad spectrum of leaf and cereal ear diseases, particularly embryo borne diseases, e.g., bunts and smuts. The Meeting received information on identity, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fates of residues in processing.

(*RS*)-2,4′-difluoro-α-(1*H*-1,2,4-triazol-1-ylmethyl)benzhydryl alcohol

Structural formula:



Flutriafol is a 1:1 mixture of the enantiomers.

In this appraisal, the following abbreviated names were used for metabolites.

M1B	4-hydroxyflutriafol
M1D	4-hydroxy-5-methoxyflutriafol
M2B	flutriafol-(trans)-dihydrodiol
M5	Mixture of two isomeric hydroxyflutriafol derivatives
R5a	Hexose conjugated flutriafol
C6	Defluorinated flutriafol
1,2,4-Triazole	1*H*-1,2,4-triazole
Triazole alanine	1,2,4-triazoyl-3-alanine

Triazole acetic acid	1*H*-1,2,4-triazol-1-ylacetic acid

Animal metabolism

The Meeting received results of animal metabolism studies with flutriafol in rats, lactating cows and laying hens. The metabolism and distribution of flutriafol in animals were investigated using [^{14}C-carbinol] and [^{14}C-triazole]-labelled flutriafol.

In rats, flutriafol was extensively absorbed following a single oral administration of 5 or 250 mg/kg dose. Bile was shown to be the major route of elimination of administered radioactivity excreted via this route over three days. Excretion of the dose was rapid at both dose levels and urinary excretion was the major route, accounting for 50–68% daily dose in the repeat dose animals and 61–68% for the single dose animals. The remaining radioactivity was excreted in the faeces (29–55%) with less than 6% dose remaining in the tissues 168 hours after the final repeat dose was administered and less than 0.8% 168 hours after the single dose. Only a minor amount of cleavage of the triazole moiety from the flutriafol molecule occurred. Furthermore, no metabolism was detected in the triazole group or in the 4-fluorophenyl ring of molecule. All identified metabolites were shown to be hydroxylated derivatives of the 2-fluorophenyl ring. Three of the major urinary metabolites were identified as a 3,4-(cis)- and the two M2B metabolites of flutriafol. The bulk of remaining urinary radioactivity was attributable to glucuronide conjugates, the two main aglycones were identified as M1B and M1D. Metabolism in rats was summarized and evaluated by the WHO panel of the JMPR in 2011.

A lactating cow were dosed with [^{14}C-triazole]-flutriafol at 40 mg/animal/day, equivalent to approximately 2 ppm in the diet for 7 consecutive days. Gelatine capsules containing ^{14}C-triazole flutriafol absorbed onto powdered maize were introduced directly into the stomach via a stomach tube. Most of the administered radioactivity was excreted in the urine and faeces (45 and 33% of the dose).

Radioactive residues in muscle and fat (subcutaneous, omental and perirenal) were insignificant (< 0.01 mg/kg equivalent to flutriafol). The liver, kidney and heart contained residues of 0.29, 0.061 and 0.011 mg/kg equivalent to flutriafol, respectively. A total of 0.1% of the radioactivity administered to the cow was collected in the milk over the 7 day dosing period. The radioactive residue in the milk gradually increased to 0.007 mg/L (flutriafol equivalent) by Day 4 of the study, and maintained this level until the end of the study.

A small amount of parent flutriafol and M1B were contained in the milk. No compound in this fraction accounted for more than 6% of TRR in the milk. Most of the radioactivity in the milk (75%) was associated with polar, water soluble metabolites which were converted to oraganosoluble fractions by extensive hydrolysis conditions. Parent flutriafol was identified (29% TRR) and no individual compound accounted for more than 10% of TRR in the liver. However, M1B was detected (23% TRR) and no individual compound accounted for more than 10% of TRR in the kidney. The radioactive residues were too low to identify the compound in muscle and fat.

Laying hens were orally dosed with [^{14}C-triazole]- or [^{14}C-carbinol]-flutriafol at doses equivalent to 13.9 or 11.6 ppm in the feed for 7 consecutive days. Most of the administered doses (89.7% for triazole label and 91.2% for the carbinol label) were recovered in the excreta at sacrifice. The radioactive residues in egg appeared to reach plateau concentrations by the end of the study, and ranged from 0.134 mg/kg (carbinol label) to 0.204 mg/kg (triazole label). The TRR values were the highest for liver (0.36–0.41 mg/kg), followed by muscle (0.01–0.06 mg/kg) and fat (0.02–0.04 mg/kg).

Parent flutriafol was the most abundant component of the residue (carbinol label: 0.088–0.119 mg/kg, 65.7–74.8% TRR, triazole label: 0.099–0.103 mg/kg, 48.3–50.5% TRR) in the eggs. 1,2,4-Triazole was also detected in the eggs (0.056–0.060 mg/kg, 27.5–29.3% TRR) and muscle (0.048 mg/kg, 75.0% TRR). Flutriafol was detected in liver at low concentrations (0.007–0.013 mg/kg, 1.9–3.2% TRR) and 1,2,4-Triazole was present in liver at 0.057 mg/kg (13.9% TRR).

Flutriafol was found in fat (0.012–0.028 mg/kg, 75.0–80.0% TRR) but no flutriafol was detected in muscle.

In the lactating cow and laying hen studies, flutriafol was metabolized to several metabolites. All metabolites detected in the metabolism of the lactating cow were also found in the metabolism of rats. The major metabolic processes in laying hens were the binding of flutriafol to liver proteins, the formation of hydroxyl flutriafol derivatives (fluorophenyl moiety), and the formation of free 1,2,4-Triazole.

Plant metabolism

The Meeting received plant metabolism studies performed on apples, sugar beet, cereals (wheat and barley) and oilseed rape using [^{14}C-triazole]- or [^{14}C-carbinol]-flutriafol.

In an apple metabolism study, apple trees were treated once at a rate of 0.12 kg ai/ha. The application rate was equivalent to 1 × the single application rate for apples. Samples of fruit and foliage were taken at a typical harvest time, 64 days after application. TRR values in apple fruits from the two radiolabelled forms of the test substance were similar with most of the radioactivity (72–78% TRR, equivalent to 0.030–0.051 mg/kg) extracted using acetonitrile or acetonitrile/water (1:1, v/v). Unextracted radioactivity comprised 18 to 23% TRR (0.009–0.012 mg/kg). Flutriafol comprised 50 to 56% TRR (0.023–0.032 mg/kg). The total unknowns comprised 22% TRR (0.013 mg/kg) in the triazole labelled sample and 9% TRR (0.003 mg/kg) in the carbinol labelled sample. The largest individual unknowns comprised only 5% and 3% TRR (0.003 and 0.001 mg/kg) respectively.

In attempt to confirm the presence of trace levels of triazole alanine and triazole acetic acid in apple fruit, the foliage from the [^{14}C-triazole]-flutriafol application was analysed since this contained higher radioactive residues. The results showed that flutriafol comprised 48% TRR (2.00 mg/kg). Triazole alanine was present at low level in the foliage extract (0.13 mg/kg) and comparison of chromatograms from fruit and foliage confirmed that triazole alanine was present in the fruit extracts at trace levels. As with the fruit extracts, the presence of trace levels of Triazole acetic acid could not be confirmed. The radioactive residue was predominantly flutriafol. The triazole alanine metabolite is known to oxidise to triazole acetic acid in plants, however it did not appear that triazole acetic acid was present in apple extracts. The free 1,2,4-Triazole metabolite was not detected in the fruit or foliage samples.

In a sugar beet metabolism study, sugar beets grown in containers outdoors were sprayed at a rate of 0.125 kg ai/ha. Samples of foliage and beet (root) were taken within three hours after application and at 16 and 21 days after treatment (DAT). The TRRs in foliage samples taken just after application were 1.37 and 1.27 mg/kg for the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. No significant residue was observed in the root. In the 16 DAT sample, TRRs were 0.342 and 0.381 mg/kg in the foliage for [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. The TRR of the root from the 16 DAT samples was ≤ 0.005 mg/kg in both labelled forms. At harvest (21 DAT), the TRRs were 0.747 and 0.596 mg/kg in the foliage, and 0.009 and 0.005 mg/kg in the root for [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. Flutriafol was the major residue in foliage samples, accounting for 69.2 and 70.8% TRR (0.412 and 0.519 mg/kg) at harvest, no other radioactive metabolite at harvest represented more than 5.4 and 5.0% TRR (0.033 and 0.038 mg/kg). There had been no cleavage of the flutriafol molecule that resulted in separation of the [^{14}C-carbinol] and [^{14}C-triazole] radiolabel positions. One metabolite of flutriafol in foliage extracts of sugar beet was identified as a hexose conjugate of flutriafol. This component represented a maximum of 0.026 mg/kg, 4.4% TRR at harvest (21 DAT).

In a cereal metabolism study, wheat and barley plants, grown both in a greenhouse and in the field, were treated at a rates of 0.081–0.105 kg ai/ha. In the greenhouse, both plants were treated with ^{14}C-flutriafol just prior ear emergence. In the field, ^{14}C-triazole-labelled flutriafol was applied to wheat just before ear emergence and to barley just after ear emergence. ^{14}C-carbinol-labelled flutriafol was applied just before ear emergence to barley and just after ear emergence to wheat.

At maturity in the greenhouse study no residues of flutriafol (< 0.005 mg/kg) were detected in the grain. Following application of ^{14}C-triazole-labelled flutriafol, triazole alanine accounted for 40–48% of the TRR in the grain (0.08 and 0.04 mg/kg in barley and wheat respectively) and triazole acetic acid was also characterised as a significant metabolite (0.04 and < 0.01 mg/kg in barley and wheat respectively). TRRs in barley and wheat grain following ^{14}C-carbinol-labelled flutriafol applications were considerably lower (0.02 and 0.01 mg/kg respectively) than those of ^{14}C-triazole-labelled flutriafol applications (0.41 and 0.18 mg/kg respectively). In barley straw, flutriafol was the major radioactive component, and accounted for 63% of the TRR (1.3 mg/kg).

At maturity in the field study, the TRR in the wheat grain, following the ^{14}C-triazole-labelled flutriafol application, was 0.05 mg/kg and no flutriafol was detected (< 0.0002 mg/kg). Triazole alanine and triazole acetic acid accounted for 58 and 26% of this residue, respectively. The TRR in barley grain was 0.10 mg/kg. Flutriafol, triazole alanine and triazole acetic acid accounted for 24, 8 and 5% of this residue respectively. The TRRs in the grain in both plants following ^{14}C-carbinol-labelled flutriafol applications were less than 0.01 mg/kg.

Radioactive residues in the straw ranged from 0.12 to 0.72 mg/kg. Flutriafol was the major component (57% in wheat straw from ^{14}C-triazole-labelled flutriafol treatment). Radioactive residues in the straw were of a similar level for greenhouse and field-grown plants. Flutriafol was the dominant component of the radioactive residue. Triazole alanine and Triazole acetic acid were not detected in the straw. Some (16–40%) of the radioactive residue was not extractable.

In an oilseed rape metabolism study, oilseed rape plants grown in containers outdoors were sprayed at a rate of 0.125 kg ai/ha. The plants were treated at the early pod set growth stage, i.e., approximately 10% of the potential pods had formed. Plants were sampled just after treatment (0 DAT), at three intermediate stages (7, 14 and 21 DAT) and at harvest (42 DAT). The TRR and the nature of the radioactive residue were investigated in the whole plant taken just after application, in the separated pods and remaining plant at the intermediate sampling (14 DAT) and in the separated seeds and remaining plant at harvest.

The TRR in the forage samples (whole plant) taken just after treatment (0 DAT) were 0.782 and 1.50 mg/kg for the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. At the pod development stage (14 DAT), TRRs were 0.751 and 0.779 mg/kg in the pods, and 1.17 and 1.60 mg/kg in the remaining plant. At harvest (42 DAT), TRRs were 1.32 and 0.729 mg/kg in the seeds, and 0.246 and 0.355 mg/kg in the remaining plant ([^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively).

In the seeds at 42 DAT, flutriafol was the only radioactive component in the hexane extracts, accounting for 31.5 and 27.2% TRR (0.415 and 0.198 mg/kg) for the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively. Flutriafol was the major radioactive component in the subsequent solvent/water extracts of the seeds, accounting for 29.8 and 27.4% TRR (0.392 and 0.200 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively). Flutriafol was present in the enzyme extracts, in the weak base extracts and in the strong acid and strong base extracts. In total, flutriafol accounted for 6.3 and 7.3% TRR (0.082 and 0.054 mg/kg) in the acid and base extracts of seeds for the [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively.

Components R5a accounted for 3.8% TRR (0.028 mg/kg) in seeds of plants treated with [^{14}C-carbinol] flutriafol, and for 3.8% TRR (0.050 mg/kg) in seeds of plants treated with [^{14}C-triazole] flutriafol. Component C6, which was present in the strong acid and strong base extracts only, accounted for a total of 3.0 and 2.9% TRR (0.039 and 0.021 mg/kg, [^{14}C-triazole] and [^{14}C-carbinol] radiolabels, respectively). Unidentified radioactive components in these seed extracts did not individually represent more than 10% TRR.

In the forage samples taken at 0 DAT, and in the remaining plant samples at 14 DAT and 42 DAT, most of the residues were extractable by acetonitrile/water. In the pods and seeds, significant residues were released only by successive additional enzyme and acid/base treatments. Flutriafol was found both in the initial acetonitrile/water extracts and in the further enzyme/acid/base extracts, suggesting some binding to the plant matrix. A hexose conjugate of flutriafol was released by

acetonitrile/water, while the other identified residue, a defluorinated flutriafol, was found only in the further extracts. All extractable residues contained both the carbinol carbon and triazole ring radiolabel centres indicating that no cleavage of the flutriafol molecule had occurred.

In the plant metabolism studies on apples, sugar beet, cereals (wheat and barley) and oilseed rape, flutriafol is the major component of the residues found in apples, sugar beet (forage), cereals (straw) and oilseed rape (seed and remaining plant). In wheat grain, the major components of the residues are triazole alanine and triazole acetic acid. In barley grain, flutriafol was the major component with low levels of 0.002 and 0.02 mg/kg. Radioactivity in sugar beet root was too low to characterize.

Environmental fate in soil

The Meeting received information on aerobic soil metabolism, soil photolysis and on rotational crops.

Aerobic soil metabolism and degradation has been studied in six different soils at a nominal average temperature of 20 °C for 252 days. Flutriafol is slowly degraded in laboratory incubated soils. Approximately 85% remained after 252 days in a loamy sand and a sandy clay loam.

The photodegradation of flutriafol was investigated on a sandy loam soil. After 7 days photochemical reactor irradiation, 60–67% of the total radioactivity applied to the soil plates was characterized as flutriafol. 1,2,4-Triazole and 2,4'-difluorobenzophenone accounted for 2–3% of the radioactive residues. After 30 days irradiation in natural sunlight, 75–82% of the applied radioactivity was characterized as flutriafol. The photodegradation products formed were similar to those seen after artificial irradiation and accounted for 7–10% of the applied radioactivity. Flutriafol appears to be relatively photochemically stable on a dry soil surface.

In a confined rotational crop study, wheat, peas, sugar beet and oilseed rape were planted at 30, 120 and 365 days after a soil application of [^{14}C-triazole] and [^{14}C-carbinol]-flutriafol at 0.125 kg ai/ha. The major components found were flutriafol, triazole alanine and triazole acetic acid. Small levels of M1B (1.5–2.5% TRR) were found in the wheat straw and sugar beet foliage.

Another confined rotational crop study was conducted using [^{14}C-triazole] and [^{14}C-carbinol] - flutriafol with lettuce, radish, and wheat planted at 30, 120 and 365 days after application to soil at 0.26 kg ai/ha. Residues in crops planted 120 DAT to soil had similar TRR values to those found in crops planted 30 DAT. Radish tops had higher residues than the corresponding radish roots samples, indicating that flutriafol and its metabolites were translocated within the xylem of the plant tissue. The TRR in the vegetative portions of wheat increased with maturity, in the order forage < hay < straw. Flutriafol was a significant component of the residue in all treated crop samples, ranging from barely detectable in wheat grain (0.003 to 0.009 mg/kg) to a high of 0.416 mg/kg in one straw sample. Triazole lactic acid and triazole alanine were detected in various commodities, present at more than 10% TRR for any individual commodity. Triazole alanine was detected as a major compound in wheat grains, and triazole acetic acid was also found at > 10% TRR.

Residues in field rotational crops were studied at sites in the UK. A foliar spray was applied at four sites in the first year at 0.188 kg ai/ha and in each subsequent year at 0.25 kg ai/ha. After five consecutive years of application, with a total rate applied of 1.188 kg ai/ha, sugar beet, fodder beet, potato, carrot and spring barley, as rotational crops, were sown/planted on the test sites in the sixth growth season and grown to maturity. Sugar beet roots, fodder beet roots, potatoes and carrots from each of the sites contained non-detectable or low residues of flutriafol, i.e., < 0.01–0.02 mg/kg. Foliage samples of sugar beet and fodder beet also contained non-detectable or low residues, < 0.01–0.08 mg/kg. Grain from the spring barley contained a low residue of flutriafol, 0.05 mg/kg. Residues of flutriafol in the spring barley straw samples were 0.38 mg/kg.

The additional field rotational crop studies were carried out using the plots treated at the highest rate (4.0 kg ai/ha) at three trial sites in the UK. The field studies were designed to provide samples of potato, sunflower, maize, spring barley, spring wheat, oilseed rape, pea, cabbage, carrot and sugar beet grown in soil with artificially high levels of aged flutriafol residues, close to or in excess of the maximum predicted concentration in soil after 26 years continuous application of the

maximum label rate (0.156 kg ai/ha) of field crops. The soil at the trial sites used had been treated in 1988 with a single application of flutriafol at a nominal rate of 4.0 kg ai/ha. The rotational crops were sown in 1991 and grown to maturity. Representative samples of the crops were taken at harvest and analysed for residues of flutriafol and its major metabolites triazole alanine and triazole acetic acid. No residues (< 0.05 mg/kg) of flutriafol were detected in the potato tuber, sunflower seeds, maize grains, rape seeds and pea seeds. Residues of flutriafol were found at low concentrations in the grain of spring barley (< 0.03–0.07 mg/kg), spring wheat (< 0.03–0.03 mg/kg), cabbage heads (< 0.05–0.12 mg/kg), carrot roots (< 0.05–0.13 mg/kg), sugar beet roots (< 0.01–0.03 mg/kg), maize straw (0.16–0.31 mg/kg) and sugar beet foliage (0.03–0.42 mg/kg). Residues in the spring barley straw (0.24–1.5 mg/kg), the spring wheat straw (0.29–2.5 mg/kg) and pea haulm (0.28–3.8 mg/kg) were found at higher levels of flutriafol. The samples of spring wheat grain (0.28–3.0 mg/kg), rape seeds (0.59–17 mg/kg) and pea seeds (0.15–7.7 mg/kg) contained high residues of triazole alanine.

Residues of flutriafol and triazole alanine may be found in rotational crops.

Analytical methods

The Meeting received description and validation data for analytical methods for residues of parent flutriafol and triazole metabolites (1,2,4-Triazole, triazole alanine and triazole acetic acid) in raw agricultural commodities, processed commodities, feed commodities and animal commodities. In most of the methods for determination of flutriafol, homogenized samples were extracted with acetonitrile/water (for plant materials) and acetonitrile (for animal commodities), and the extract was cleaned up with liquid–liquid partition followed by column chromatography using SPE (for plant materials) or gel permeation chromatography (for animal materials). Residues were determined by gas chromatography with NPD or MSD, or HPLC with MS/MS. The methods of analysis for a range of substrates were validated with LOQs of the 0.01 mg/kg for flutriafol.

The multiresidue method DFG Method S19 (modified version) with GC employing TSD, NPD or MSD was validated for flutriafol in plant materials. LOQs were 0.01–0.05 mg/kg for flutriafol.

The Meeting received LC-MS/MS method of analysis for triazole metabolites in plant and animal materials. The method was specific for each metabolite. The method was validated with an LOQ of 0.01 mg/kg for all analytes.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the freezer storage stability of flutriafol residues in plants (apple, grape, cabbage, sugar beet root, pea seed, soya bean, barley grain, wheat and oilseed rape), processed commodities (apple juice, soya bean meal and refined oil) and animal commodities (milk, eggs, muscle and fat).

The Meeting also received information on the freezer storage stability of triazole metabolites in apple (fruit and juice), milk, eggs, muscle and fat.

Storage stability results indicate that flutriafol residues were stable for at least 4 months in animal commodities, for at least 5 months in soya bean seed, for at least 12 months in apple, barley grains and coffee beans, for at least 23 months in grapes, for at least 24 months in cabbage and oilseed rape, and for at least 25 months in wheat (grains and straw), pea seed, sugar beet root. The results also indicate that triazole metabolite residues were stable for at least 4 months in apple fruits and juice, and for at least 5 months in animal commodities.

The periods of storage stability studies cover the sample storage intervals of residue trials.

Definition of the residue

In the lactating cow metabolism study, TRRs in liver (0.29 mg/kg), kidney (0.06 mg/kg) and heart (0.01 mg/kg) were higher than those in milk and other tissues (< 0.01 mg/kg). Flutriafol is the major component of the residue in liver (29% TRR). However, M1B is the major component of the residue in kidney (23% TRR) but at low level. In the laying hen metabolism study, TRRs were the highest for

liver (0.36–0.41 mg/kg), followed by muscle (0.01–0.06 mg/kg) and fat (0.02–0.04 mg/kg). Flutriafol is the major residue component in eggs and fat (48–80% TRR).

1,2,4-Triazole was found in muscle (75.0% TRR) and liver (13.9% TRR) of laying hens. However, according to farm animal feeding studies, concentrations of 1,2,4-Triazole, triazole alanine and triazole acetic acid were < 0.01 mg/kg.

The Meeting decided that parent flutriafol is a suitable analyte for enforcement purposes and dietary risk assessment in animal commodities.

The octanol/water coefficient (log P_{ow}) of flutriafol is 2.3 at 20 °C. In the lactating cow metabolism and feeding studies, flutriafol residues were < 0.01 mg/kg in muscle and fat. However, in the laying hen metabolism and feeding studies, flutriafol residues in fat were at least 3 times higher than those in muscle. The Meeting considered flutriafol was fat soluble.

The plant metabolism studies of flutriafol were conducted with fruit (apple), root vegetables (sugar beet), cereals (wheat and barley) and oilseeds (rape). Each study was conducted with both triazole- and carbinol-radiolabelled flutriafol. Parent flutriafol was always the major component (24–71% TRR) in all matrices except wheat grains at harvest. Triazole alanine and triazole acetic acid accounted for 58 and 28% of TRR (0.05 mg/kg) in wheat grains. No other radioactive components in the extracts from plant matrices were individually present at more than 10% TRR.

The 2007 JMPR noted that 1,2,4-Triazole, triazole acetic acid and triazole alanine may be derived from several sources. In a situation in which the metabolites arise from multiple triazole fungicides, they cannot be included in the residue definition. Since the metabolite cannot be linked to a specific triazole fungicide, they would have to be evaluated on their own.

Field trials conducted in the USA indicated that triazole alanine and triazole acetic acid were found in plant matrices. However, 1,2,4-Triazole was not detected in samples (except one trial) above LOQ. These findings agree with the information obtained from the metabolism studies. The relatively low level of the residues in food commodities and the low toxicity of triazole alanine and triazole acetic acid do not justify their inclusion for dietary risk assessment.

The Meeting decided that parent flutriafol is a suitable analyte for enforcement purposes and dietary risk assessment in plant commodities.

The Meeting recommended the following residue definition:

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

The Meeting considers the residue is fat soluble

Results of supervised trials on crops

The Meeting received supervised trial data for the foliar application of flutriafol on apples, grapes, bananas, sweet peppers, soya beans, wheat, peanuts and coffee. Residue trial data was made available from European countries, the countries of Latin America and the USA.

Labels were available from Argentina, Australia, Belarus, Brazil, Chile, Columbia, Croatia, Estonia, Italy, Kazakhstan, Lithuania, Malaysia, Mexico, Moldova, Romania, Russia, South Africa, Spain, Taiwan, the Ukraine and the USA describing the registered uses of flutriafol.

The OECD calculator was used as a tool to assist in the estimation of maximum residue levels from the selected residue data set obtained from the supervised residue trials. As a first step, the Meeting reviewed the trial conditions and other relevant factors related to each data set to arrive at a best estimate of the maximum residue level using expert judgement. Then the OECD calculator was employed. If the statistical calculation spreadsheet suggested a different value, a brief explanation of the deviation was supplied.

Pome fruits

Data were available from supervised trials on apples in European countries and the USA.

In Italy, flutriafol is registered for apples at two foliar applications of 0.030 kg ai/ha with a PHI of 21 days. However, the residue trials on apples conducted in Greece, Italy, France and Spain did not match the GAP of Italy.

The GAP on pome fruit of the USA is a maximum four foliar applications at a maximum rate of 0.12 kg ai/ha with a PHI of 14 days. Flutriafol residues in apples from trials in the USA matching GAP were (n = 16): 0.03, 0.04, 0.05 (3), 0.06 (3), 0.08 (2), 0.09, 0.10 (2), 0.12 (2) and 0.16 mg/kg.

Based on the trials for apples in the USA, the Meeting estimated a maximum residue level, an STMR value and an HR value for flutriafol in pome fruit of 0.3, 0.07 and 0.16 mg/kg respectively.

Grapes

Data were available from supervised trials on grapes from a number of European countries and the USA.

In the Ukraine, flutriafol is registered for use on grapes at two foliar applications of 0.025 kg ai/ha with a PHI of 45 days. In Romania, flutriafol is registered for use on grapes at six foliar applications of 0.038 kg ai/ha with a PHI of 30 days. However, the residue trials on grapes conducted in Greece, Italy, France and Spain did not match the GAP of the Ukraine or that of Romania.

The GAP on grapes of the USA allows a maximum six foliar applications at a maximum rate of 0.073–0.091 kg ai/ha with a PHI of 14 days. The Meeting concluded that the additional spray would have little influence on the final residue. The Meeting decided to use the principle of proportionality, as described in the JMPR Report 2010, to estimate a maximum residue level, an STMR value and an HR value for grapes.

Residues in grapes from the US trials at 0.128 kg ai/ha were (n = 13): 0.12, 0.21 (2), 0.25, 0.28, 0.30 (2), 0.31, 0.35, 0.37, 0.43, 0.61 and 0.86 mg/kg. Scaled residues in grapes were (scaling factor 0.71; 0.128➔0.091 kg ai/ha): 0.09, 0.15 (2), 0.18, 0.20, 0.21 (2), 0.22, 0.25, 0.26, 0.31, 0.43 and 0.61 mg/kg.

Based on the scaled residues in grapes, the Meeting estimated a maximum residue level, an STMR value and an HR value of 0.8, 0.21 and 0.61 mg/kg, respectively.

Banana

Data were available from supervised trials on bananas from a number of South American countries.

In Columbia, flutriafol is registered for use as a foliar application to bananas at a rate of 0.1 kg ai/ha with a PHI of 0 days. The number of applications is not specified on the registered label of Columbia. The results from unbagged bananas in each trial were used to estimate a maximum residue level, an STMR and an HR value for banana. In all trials conducted, residue concentrations in bagged banana were never higher than those in unbagged banana. Residues in whole fruit of unbagged banana from trials matching GAP of Columbia were (n = 12): 0.01, 0.02 (2), 0.05, 0.07 (2), 0.09, 0.10 (2), 0.14 and 0.17 (2) mg/kg.

Based on the residues in whole fruit of unbagged banana, the Meeting estimated a maximum residue level for flutriafol in banana of 0.3 mg/kg.

Residues in unbagged banana pulp from trials matching the GAP of Columbia were (n = 12): 0.01, 0.02, 0.04 (2), 0.05 (4), 0.07, 0.08 (2) and 0.09 mg/kg.

Based on the residues in unbagged banana pulp, the Meeting estimated an STMR and an HR values for flutriafol in banana of 0.05 and 0.09 mg/kg respectively.

Sweet peppers

Data were available from supervised trials on sweet peppers in Spain.

In Spain, flutriafol is registered for use of three foliar applications on sweet peppers at a rate of 0.019 kg ai/hL with a PHI of 1day. Because residues decline was slow, the Meeting decided to use

the available 0 day PHI data. Residues in sweet peppers from trials in greenhouse approximately matching GAP of Spain were (n = 8): 0.15, 0.19, 0.24, 0.26, 0.29, 0.32 and 0.41 (2) mg/kg.

Based on the trials for sweet peppers in Spain, the Meeting estimated a maximum residue level, an STMR and HR values for flutriafol in sweet peppers of 1, 0.28 and 0.41 mg/kg, respectively.

The normal Meeting procedure is to round the value to the nearest units for maximum residue levels. Rounding up the value of 0.9 mg/kg obtained from the OECD calculator results in 1 mg/kg, which coincides with the recommendation of the current Meeting.

Soya bean

Data were available from supervised trials on soya bean in Brazil and the USA.

In Brazil, flutriafol is registered for use on soya bean at two foliar applications of 0.125 kg ai/ha with a PHI of 28 days. Residues in soya bean seeds from trials matching Brazilian GAP were (n = 3): < 0.05 (3) mg/kg. However, the numbers of trials on soya bean, matching Brazilian GAP, were considered insufficient to estimate a maximum residue level for the commodity.

Trials from the USA on soya bean were reported for the foliar application of a SC formulation (GAP: three foliar applications of a maximum rate of 0.128 kg ai/ha, 0.256 kg ai/ha per season, PHI of 21 day). Flutriafol residues in soya bean seeds from trials in the USA matching GAP were (n = 20): < 0.01, 0.01, 0.02 (3), 0.03, 0.04 (3), 0.05, 0.06 (2), 0.07, 0.08 (3), 0.11, 0.17, 0.19 and 0.30 mg/kg.

Based on the trials on soya beans in the USA, the Meeting estimated a maximum residue level and an STMR value for flutriafol in soya bean seeds of 0.4 and 0.055 mg/kg, respectively.

Wheat

Data were available from supervised trials on wheat in European countries and the USA.

In Lithuania, flutriafol is registered for use on wheat at two foliar applications of 0.125 kg ai/ha with a PHI of 30 days. Residue in wheat grains from trial in Northern France and UK matching GAP of Lithuania was < 0.01 mg/kg. However, the trials for wheat matching the GAP of Lithuania were insufficient to estimate a maximum residue level for the commodity.

In Spain, flutriafol is registered for use on wheat at the foliar application of 0.125 kg ai/ha between the end of stem elongation and flowering. The frequency of application is not specified in the registration label. Residues in wheat grains from trials in South France and Spain matching the GAP of Spain were (n = 8): < 0.01 (2), 0.01 (2), 0.02 (2), 0.04 and 0.1 mg/kg.

Trials from the USA on wheat were reported for the foliar application of a SC formulation (GAP: two foliar applications of a maximum rate of 0.128 kg ai/ha, PHI of 30 day). However, flutriafol residue trials on wheat in the USA did not match the GAP of the USA.

Based on the trials for wheat in Southern France and Spain, the Meeting estimated a maximum residue level, an STMR value for flutriafol in wheat grains of 0.15 and 0.015 mg/kg respectively.

Peanuts

Data were available from supervised trials on peanuts from the USA.

Trials from the USA on peanuts were reported for the foliar application of a SC formulation (GAP: four foliar applications of a maximum rate of 0.128 kg ai/ha, PHI of 7 day). Flutriafol residues in peanut nutmeat from trials in the USA matching GAP were (n = 13): < 0.01 (2), 0.01 (2), 0.02 (4), 0.03 (2), 0.04 (2) and 0.08 mg/kg.

Based on the trials for peanuts from the USA, the Meeting estimated a maximum residue level and an STMR value for flutriafol in peanut nutmeats of 0.15 and 0.02 mg/kg respectively.

The OECD calculator estimated a maximum residue level of 0.1 mg/kg. However, the Meeting recommended 0.15 mg/kg because the result of the OECD calculator was too close to the highest residue value.

Coffee beans

Data were available from supervised trials on coffee in Brazil, Columbia, Guatemala and Vietnam.

Trials from Brazil on coffee were reported for the foliar application of a SC formulation. (GAP: initial soil application of a rate of 0.69 kg ai/ha followed by two foliar applications of a rate of 0.25 kg ai/ha, PHI of 30 day). Flutriafol residues in coffee beans from trials in Brazil, Columbia and Guatemala matching GAP of Brazil were (n = 8): 0.01, 0.04, < 0.05 (4), 0.05 and 0.10 mg/kg.

Based on the trials for coffee in Brazil, Columbia and Guatemala, the Meeting estimated a maximum residue level and an STMR value for flutriafol in coffee beans of 0.15 and 0.05 mg/kg respectively.

Animal feedstuffs

Wheat forage and straw

Data were available from supervised trials on wheat in European countries and the USA.

In Lithuania, flutriafol is registered for use on wheat at two foliar applications of 0.125 kg ai/ha with a PHI of 30 days. Residues in wheat whole plant from trial in Northern France and UK matching GAP of Lithuania were (n = 3): 0.16 (2) and 0.17 mg/kg. Residues in wheat straw from trials in Northern France and UK matching GAP of Lithuania were 0.44 mg/kg. However, the trials for wheat matching the GAP of Lithuania were insufficient to estimate a maximum residue level for the commodity.

In Spain, flutriafol is registered for use on wheat at the foliar application of 0.125 kg ai/ha between the end of stem elongation and flowering. The frequency of application and the timing of harvest for forage are not specified in the registration label. Residues in wheat straw from trials in Southern France and Spain matching GAP of Spain were (n = 8): 0.15, 0.35, 0.55, 1.4, 1.5, 1.9, 3.6 and 4.1 mg/kg (fresh weight basis).

Based on the residues in wheat straw from trials in Southern France and Spain, the Meeting estimated a maximum residue level, a median residue value and a highest residue value for flutriafol in wheat straw and fodder, dry of 8, 1.45 and 4.1 mg/kg respectively.

Trials from the USA on wheat were reported for the foliar application of a SC formulation (GAP: two foliar applications of a maximum rate of 0.128 kg ai/ha, PHI of 0 day for wheat forage). Residues in wheat forage from trials in the USA matching GAP were (n = 20): 4.0, 4.1, 4.2, 5.1, 5.2, 5.5, 5.6, 5.7, 7.3, 7.6, 8.4, 8.6, 9.2, 9.3, 11 (2), 13 (2), 16 and 19 mg/kg (fresh weight basis).

Based on the residues in wheat forage from trials in the USA, the Meeting estimated a median residue value and a highest residue value for flutriafol in wheat forage of 8.0 and 19 mg/kg respectively.

Peanut fodder

Data were available from supervised residue trials on peanut in the USA

Trials from the USA on peanut were reported for the foliar application of a SC formulation (GAP: four foliar applications of a maximum rate of 0.128 kg ai/ha, PHI of 7 day). Flutriafol residues in peanut hay from trials in the USA matching GAP were (n = 13): 0.74, 1.5, 1.7, 2.0, 2.1, 2.5, 2.6, 3.1, 4.3, 7.3, 7.7, 8.8 and 8.9 mg/kg (fresh weight basis).

Based on the trials for peanut in the USA, the Meeting estimated a maximum residue level, a median residue value and a highest residue value for flutriafol in peanut fodder of 20, 2.6 and 8.9 mg/kg respectively.

Rotational crops

The Meeting noted that residues may occur in rotational crops. However, available field rotational crop studies were not adequate to propose maximum residue levels to cover rotational crops.

Fate of residues during processing

The fate of flutriafol residues has been examined in apple, grapes, soya bean seeds, wheat grains and peanut nutmeats processing studies. Based on the results of processing studies conducted in the USA, processing factors were calculated for apples, grapes, sweet peppers, soya bean, wheat and peanut. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P and HR-P for food and feed

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
Apple	Juice	0.50, 0.45	0.48	0.07	0.034	0.16	
	Wet pomace	1.9, 1.9	1.9		0.13		
	Dry pomace	10, 8.5	9.3		0.65		1.5
Grape	Juice	0.63	0.63	0.21	0.13	0.61	
	Raisin	2.8	2.8		0.59		1.7
Sweet pepper	Preserved	0.57, 0.67, 0.74 (2), 0.79, 1.1, 1.3, 1.4	0.77	0.28	0.22	0.41	0.32
Soya bean	Meal	1.3	1.3	0.055	0.072		
	Refined oil	1.3	1.3		0.072		
	Hulls	0.97	0.97		0.053		
	Aspirated grain fraction	1.7	1.7		0.094		
Wheat	Bran	2.1	2.1	0.015	0.032		
	Flour	0.33	0.33		0.005		
	Germ	2.8	2.8		0.042		
	Aspirated grain fraction	13	13		0.20		
Peanut	Meal	0.79	0.79	0.02	0.016		
	Refined oil	1.4	1.4		0.028		
Coffee	Roasted coffee beans	0.95	0.95	0.05	0.048		

[a] Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

The Meeting estimated a maximum residue level of 2 mg/kg (0.8 × 2.8 = 2.2 mg/kg) for raisins, 0.3 mg/kg (0.15 × 2.1 = 0.315 mg/kg) for wheat bran by the processing factor.

On the basis of the STMR and HR for sweet peppers and default dehydration factor of 10, the Meeting estimated at an STMR value and an HR value for dried chili peppers of 2.7 and 4.1 mg/kg respectively. Based on the maximum residue level of sweet peppers, the Meeting recommended a maximum residue level of 10 mg/kg for chili peppers (dry).

Residue in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of flutriafol in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual 2009. Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides levels in feed suitable for estimating maximum residue levels, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed in a dry weight basis.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Appendix IX of the FAO manual. The calculations were made according to the animal diets from US-Canada, EU, Australia and Japan in the Table (Appendix IX of the FAO manual).

Livestock dietary burden, flutriafol, ppm of dry matter diet								
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.50	0.19	15	6.5	76[a]	32[b]	0.064	0.064
Dairy cattle	17	6.9	15	6.5	50	20[c]	0.055	0.055
Poultry-broiler	0.041	0.041	0.050	0.050	0.039	0.039	0.029	0.029
Poultry-layer	0.041	0.041	7.6[d]	3.2[e]	0.038	0.038	0.024	0.024

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

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

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

[d] Highest maximum poultry dietary burden suitable for maximum residue level estimates for poultry meat and eggs

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

Farm animal feeding studies

The Meeting received a lactating dairy cow and a laying hen feeding studies, which provided information on likely residues resulting in animal commodities, milk and eggs from flutriafol residues in the animal diet.

Lactating dairy cows

Holstein dairy cows were dosed with flutriafol for 29 days at the equivalent of 0.5, 1.5 and 5.0 ppm in the diet. Residues of flutriafol were below the LOQ (0.01 mg/kg) in whole milk at the 5.0 ppm feeding level. Kidney, muscle and fat contained no residue (< 0.01 mg/kg) of flutriafol at 0.5 and 5.0 ppm feeding levels. Liver contained flutriafol residues of < 0.01–0.04 mg/kg at the 0.5 ppm feeding level, 0.09–0.10 mg/kg at the 1.5 ppm level and 0.23–0.39 mg/kg at the 5.0 ppm level respectively.

Laying hens

Laying hens were dosed with flutriafol for 29 days at the equivalent of 0.5, 1.5 and 5.0 ppm in the diet. Residues of flutriafol were below the LOQ (0.01 mg/kg) in eggs at the 0.5 ppm feeding level. At the 5.0 ppm level, flutriafol residues in eggs were 0.02–0.04 mg/kg from day 3 to day 28. Muscle contained no residue (< 0.01 mg/kg) of flutriafol at 0.5 and 5.0 ppm feeding levels. Liver contained no residues (< 0.01 mg/kg) of flutriafol at 0.5 feeding level and 0.03–0.10 mg/kg at the 5.0 ppm level. Fat contained no residues (< 0.01 mg/kg) of flutriafol at 0.5 feeding level and 0.05–0.07 mg/kg at the 5.0 ppm level.

Animal commodities maximum residue levels

For MRL estimation, the residue in animal commodities is flutriafol.

The maximum dietary burden for beef and dairy cattle is 76 ppm and is much higher than the highest dose level in the feeding study of 5.0 ppm. In a feeding study, in which flutriafol equivalent to 5.0 ppm in the diet was dosed to lactating cows for 29 consecutive days, no flutriafol residues were detected in kidney, muscle, fat and milk (< 0.01 mg/kg). The Meeting considered it is not applicable to extrapolate the residues in kidney, liver, muscle, fat and milk at a dietary burden of 76 ppm from the results of the feeding study.

The Meeting could not estimate a maximum residue level of mammalian meat, mammalian edible offal and milk.

The maximum dietary burden for broiler and layer poultry is 7.6 and is higher than the highest dose level in the feeding study of 5.0 ppm. In a feeding study, in which flutriafol equivalent to 5.0 ppm in the diet was dosed to laying hens for 29 consecutive days, no flutriafol residues were detected

in muscle (< 0.01 mg/kg). The Meeting considered it is not applicable to extrapolate the residues in muscle at a dietary burden of 7.6 ppm from the results of the feeding study.

The Meeting could not estimate a maximum residue level of poultry meat, poultry edible offal and eggs.

FURTHER WORK OR INFORMATION

Desirable

Field rotational crop studies suitable for estimation of maximum residue levels for rotational crops.

Animal feeding studies covering the estimated maximum dietary burden of farm animals.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Plant and Animal commodities:

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

The residue is fat soluble.

Commodity		Recommended MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New		
FI 0327	Banana	0.3	0.05	0.09
SB 0716	Coffee beans	0.15	0.05	
DF 0269	Dried grapes (= Currants, Raisins and Sultanas)	2	0.59	1.7
FB 0269	Grapes	0.8	0.21	0.61
SO 0697	Peanut	0.15	0.02	
AL 0697	Peanut fodder	20	2.6	8.9
VO 0445	Peppers, Sweet (including pimento or pimiento)	1	0.28	0.41
VO 0444	Peppers, Chili, dried	10	2.7	4.1
FP 0009	Pome fruits	0.3	0.07	0.16
VD 0541	Soya bean (dry)	0.4	0.055	
GC 0654	Wheat	0.15	0.015	
CM 0654	Wheat bran, unprocessed	0.3	0.032	
AS 0654	Wheat straw and fodder, dry	8	1.45	4.1
AB 0226	Apple dry pomace		0.65	
JF 0226	Apple juice		0.034	
AB 1230	Apple wet pomace		0.13	
SB 0716	Coffee beans, roasted		0.048	
JF 0269	Grape juice		0.13	
AB 0697	Peanut meal		0.016	
OR 0697	Peanut oil, edible		0.028	
	Peppers, Sweet, preserved		0.22	0.32
CF 0541	Soya bean aspirated grain fraction		0.094	
AB 0541	Soya bean hulls		0.053	
AB 1265	Soya bean meal		0.072	
OR 0541	Soya bean refined oil		0.072	
CF 0654	Wheat aspirated grain fraction		0.20	
AF 0654	Wheat forage		8.0	19
CF 1211	Wheat flour		0.005	
CF 1210	Wheat germ		0.042	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of flutriafol were calculated for the 13 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (see Annex 3 of the 2011 JMPR Report). The ADI is 0–0.01 mg/kg bw and the calculated IEDIs were 0–7% of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term intakes of residues of flutriafol, resulting from the uses considered by current JMPR, are unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of flutriafol were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (see Annex 4 of the 2011 JMPR Report). The ARfD is 0.05 mg/kg bw and the calculated IESTIs were a maximum of 50% of the ARfD. The Meeting concluded that the short-term intake of residues of flutriafol, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institution, Report reference
1358 FLU	Comb, A. L.	2006	Flutriafol Pure Melting Temperature and Boiling Temperature Huntingdon Life Sciences Ltd., GBR Study No.: CHV0146/063780 Unpublished report, CHA Doc. No.: 1358 FLU
1097 FLU	Tognucci, A.	2003	Determination of the Relative Density of Flutriafol Purified Active Substance RCC Ltd., Itingen, CHE Study No.: 847681 Unpublished report, CHA Doc. No.: 1097 FLU
1513 FLU	Piasentini de Campos, L. F.	2007	Vapor Pressure of Flutriafol Tecnico DK BIOAGRI Laboratórios Ltda., BRA Study No.: RF-0265.007.186.06 Unpublished report, CHA Doc. No.: 1513 FLU
1359 FLU	Kusk, T.	2006	Determination of the Long Term Storage Stability of Flutriafol Technical (dry), Batch No. UPL Bx 1 (2001) in commercial Packaging Cheminova A/S Study No.: PYC 120 Unpublished report, CHA Doc. No.: 1359 FLU
1367 FLU	Søndergaard, L.	2006	Statement of Odor of Flutriafol Technical Wet Paste Cheminova A/S Study No.: CHA 100007 Unpublished report, CHA Doc. No.: 1367 FLU
1103 FLU	Tognucci, A.	2004	Determination of the Water Solubility of Flutriafol Purified Active Substance RCC Ltd., Itingen, CHE Study No.: 847682 Unpublished report, CHA Doc. No.: 1103 FLU
1104 FLU	Tognucci, A.	2004	Determination of the Solubility of Flutriafol Technical in Organic Solvents RCC Ltd., Itingen, CHE Study No.: 847683 Unpublished report, CHA Doc. No.: 1104 FLU
1511 FLU	Piasentini de Campos, L. F.	2007	Partition Coefficient (N-octanol/water) of Flutriafol Tecnico DK BIOAGRI Laboratórios Ltda., BRA Study No.: RF-0265.014.178.06 Unpublished report, CHA Doc. No.: 1511 FLU
213 FLU	Hawkins, D.R., Elsom, L.F. and Jackson, R.	1987	PP450 - A Hydrolysis Study in Aqueous Solution at Ph 5,7 and 9 Huntingdon Research Centre Study No.: HRC/ISN '136/87510 Unpublished report, CHA Doc. No.: 213 FLU
212 FLU	Snow, A.D. and	1982	PP450 : Hydrolysis in Aqueous Solution

Code	Author	Year	Title, Institution, Report reference
	Cavell, B.D.		ICI Plant Protection Division Jealott's Hill Research Station Study No.: RJ0281B Unpublished report, CHA Doc. No.: 212 FLU
215 FLU	Moffatt, F.	1994	Flutriafol : Environmental Half-Life and Quantum Yield for Direct Phototransformation in Aqueous Solution Zeneca Agrochemicals, Jealott's Hill Research Station Study No.: RJ1581B Unpublished report, CHA Doc. No.: 215 FLU
214 FLU	Skidmore, M.W.	1987	Flutriafol : A Photolysis Study in Aqueous Solution at pH7 ICI Plant Protection Division, Jealott's Hill Study No.: PP450AG03 Report No.: RJ0607B Unpublished report, CHA Doc. No.: 214 FLU
29 FLU	Jones, B.K., Ladd, R.M., Choo, C.K.. and Soames, A.R.	1982	PP450: Excretion and Tissue Retention of a Single Oral Dose (5 mg/kg) in the Rat ICI, Central Toxicology Laboratory Study No.: CTL/P/751 Report No.: UR0140, UR0149 Unpublished report, CHA Doc. No.: 29 FLU
30 FLU	Jones, B.K. and Ladd, R.M.,	1986	PP450: Flutriafol : Biotransformation in the Rat ICI, Central Toxicology Laboratory Study No.: CTL/P/867 Report No.: UR0140, UR0149 Unpublished report, CHA Doc. No.: 30 FLU
1293 FLU	Millais, A.J.	2004	[^{14}C]-Flutriafol. Metabolism in Rats after single and repeated Doses Amended Final Report Huntingdon Life Sciences Ltd. Study No.: CHV '081/024050 Unpublished report, CHA Doc. No.: 1293 FLU
85 FLU	Hignett, R.R. and Cavell, B.D.	1985	Flutriafol : Quantification and Characterisation of Radioactive Residues in Milk and Tissues of a Cow After Dosing with ^{14}C-Flutriafol ICI Plant Protection Division, Jealott's Hill Study No.: PP450AA01 ; ISN/021 Report No.: RJ0416B Unpublished report, CHA Doc. No.: 85 FLU
992 FLU	Litchfield, M.H.	2003	Flutriafol: Metabolism in the Lactating Cow–A Re-Assessment of ICI Plant Protection Division, Jealott's Hill Study No. RJ0416B. Flutriafol: Quantification and Characterisation of Radioactive Residues in Milk and Tissues of a Cow after Dosing with ^{14}C -Flutrifol Melrose Consultancy, GBR Unpublished report, CHA Doc. No.: 992 FLU
1464 FLU	Dohn, D. R., Bowler, D. T. and Estigoy, L. L.	2006	The Metabolism of [^{14}C] Flutriafol in Laying Hens PTRL West, Inc. (USA) / Wildlife International, Ltd. (USA) Study No.: '1348W Report No.: '1348W-1 Unpublished report, CHA Doc. No.: 1464 FLU
1625 FLU	Brice, A.	2007	[^{14}C]-Flutriafol: Metabolism in Apple Covance Laboratories Ltd., GBR Study No.: '0676/038 Report No.: '0676/038-D2149 Unpublished report, CHA Doc. No.: 1625 FLU
1080 FLU	Aikens, P.J.	2003	^{14}C-Flutriafol Metabolism in Sugar Beet Huntingdon Life Sciences Ltd. Study No.: CHV/080 Unpublished report, CHA Doc. No.: 1080 FLU
80 FLU	MacNeil, R.M., Snow, A., Bell, E.G., Goddard, C. and Cavell, B.D.	1982	PP450 : Characterisation of the Radioactive Residue in Grain and Straw Following Foliar Application of 14C-PP450 to Wheat and Barley ICI Plant Protection Division Study No.: RJ0300B Unpublished report, CHA Doc. No.: 80 FLU
1079 FLU	Shaw, D.	2003	^{14}C -Flutriafol Metabolism in Oilseed Rape Huntingdon Life Sciences Ltd. Study No.: CHV '079/032256 Unpublished report, CHA Doc. No.: 1079 FLU
173 FLU	Vincent, J., Harvey, B.R., Arnold, D.J. and	1982	PP450 : Degradation in Laboratory Incubated Soils ICI Plant Protection Division, Jealotts Hill

Code	Author	Year	Title, Institution, Report reference
	Hill, I.R.		Study No.: RJ0287B Unpublished report, CHA Doc. No.: 173 FLU
172 FLU	Snow, A.D., Cavell, B.D.	1982	PP450 : Photolysis on a Soil Surface ICI Plant Protection Division, Jealotts Hill Study No.: RJ0289B Unpublished report, CHA Doc. No.: 172 FLU
82 FLU	Skidmore, M.	1988	Flutriafol - An Investigation Into the Nature of Radioactive Residues Taken Up By Rotated Crops Grown in Soil Treated with 14C-Flutriafol (Technical Letter No 88JH077/1) ICI Plant Protection Division, Jealotts Hill Study No.: RIC1360 Unpublished report, CHA Doc. No.: 82 FLU
1355 FLU	Skidmore, M.	1999	Full details on Characterisation of Metabolites in the Submitted Study, SC 9679/8 for all Crop Studies including further Information on the Comparison of Metabolite Distribution before and after storage. If this is not available then the Study must be Repeated to show the Metabolism and Distribution of Flutriafol in Rotational Crops Zeneca Agrochemicals Jealott's Hill Research Station, GBR Study No.: 90000244 Unpublished report, CHA Doc. No.: 1355 FLU
1624 FLU	Dohn, D. R., Mohajeri, S. and Estigoy, L. E.	2009	A Confined Rotational Crop Study with [^{14}C] Flutriafol using Radish, Lettuce, and Wheat at 30 and 120 Day Plant-back Intervals PTRL West, Inc., USA Study No.: '1535W Report No.: '1535W-1 Unpublished report, CHA Doc. No.: 1624 FLU
1622 FLU	Dohn, D. R., Mohajeri, S. and Estigoy, L. L.	2009	A Confined Rotational Crop Study with [^{14}C] Flutriafol using Radish, Lettuce, and Wheat at a 365 Day Plant-back Interval PTRL West, Inc., USA Study No.: '1554W Report No.: '1554W-1 Unpublished report, CHA Doc. No.: 1622 FLU
209 FLU	Dick, J.P.	1988	Flutriafol : Residues in Soil and Crops From Rotational Crop Studies in the UK During 1987 ICI Agrochemicals, Jealott's Hill Research Station Study No.: M4778B Unpublished report, CHA Doc. No.: 209 FLU
83 FLU	Wheals, I.B.	1993a	Flutriafol : Residues of Flutriafol and Its major Metabolites in Potato, Sunflower and Maize grown on Soil with Aged Flutriafol Residues in the UK during 1991 ICI Agrochemicals, Jealott's Hill Research Station Study No.: RJ1270B Unpublished report, CHA Doc. No.: 83 FLU
84 FLU	Wheals, I.B.	1993b	Flutriafol : Residues of Flutriafol and Its major Metabolites in Barley, Wheat, Oilseed Rape, Carrot, Cabbage and Pea grown on Soil with Aged Flutriafol Residues in the UK during 1991 ICI Agrochemicals, Jealott's Hill Research Station Study No.: RJ1252B Unpublished report, CHA Doc. No.: 84 FLU
453 FLU	Wheals, I.B.	1993c	Flutriafol: Residues in Soil and Sugarbeet From Accelerated Carryover Trials Carried Out in the UK 1988-1990 (Series 1). (Final Report) ICI Agrochemicals, Jealott's Hill Research Station Study No.: '88JH058 Report No.: RJ1223B Unpublished report, CHA Doc. No.: 453 FLU
1377 FLU	Watson, G. and Stevenson, K.	2006	Final Report on Project AD/10029/CN : Study to Determine the Stability of Flutriafol Residues in Apples following Frozen Storage at ca. -18ºC for 0, 3, 6 & 12 months : 2005-2006 Agrisearch LTD, GBR Study No.: AD/10029/CN Unpublished report, CHA Doc. No.: 1377 FLU
1471 FLU	Willard, T.R.	2007	Magnitude of the Residue of Flutriafol and Three Triazole Metabolites in Apple Raw Agricultural and Processed Commodities American Agricultural Services, Inc. (USA) / ACDS Research, Inc. (USA) / Morse Laboratories, Inc. (USA) Study No.: AA060705

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			Unpublished report, CHA Doc. No.: 1471 FLU
935 FLU	Greig, I.	2005	Technical Letter Agrisearch UK Limited, GBR Study No.: L270/IG/RT Report No.: - Unpublished report, CHA Doc. No.: 935 FLU
1468 FLU	Rice, F.	2007	Magnitude of the Residues of Flutriafol and Three Metabolites in/on Raw Agricultural Commodities of Soya bean Following Application of an SC Formulation of Flutriafol ABC Laboratories, Inc. (USA) Study No.: 49535 Unpublished report, CHA Doc. No.: 1468 FLU
73 FLU	Swaine, H.	1982	PP450 : Grain Storage Stability : Stability of PP450 Residues in Grain Stored Deep Frozen for One Year ICI Plant Protection Division, GBR Study No.: PP450BC01 Report No.: PP450B014 Unpublished report, CHA Doc. No.: 73 FLU
1126 FLU	Mende, P.	2004	Determination of the Storage Stability of Flutriafol in Wheat Plant, Straw and Grain at approximately -20°C Arbeitsgemeinschaft GAB Biotechnologie GmbH, GAB Analytik GmbH Study No.: '20031018/01-RSS Unpublished report, CHA Doc. No.: 1126 FLU
75 FLU	Benet, F.	1993	Storage Stability in Deep Frozen Wheat Straw Samples ICI Protection de l'Agriculture Study No.: 92 PA 005 Unpublished report, CHA Doc. No.: 75 FLU
79 FLU	Benet, F.	1993	Flutriafol : Storage Stability in Deep Frozen Oil Seed Rape Samples ICI Protection de l'Agriculture Study No.: 92 PA 003 Unpublished report, CHA Doc. No.: 79 FLU
169 FLU	Kwiatkowski, A.S., Robinson, N.J.	1995	Triazolyl Acetic Acid : Storage Stability in Wheat Grain and Straw, Oil Seed Rape, Cabbage, Pea Seed and Sugar Beet Root when Stored Deep Frozen for up to Twenty Five Months Zeneca Agrochemicals, Jealott's Hill Research Station Study No.: RJ1932B Unpublished report, CHA Doc. No.: 169 FLU
1566 FLU	Willard, T. R.	2008	Magnitude of the Residue of Flutriafol and Three Triazole Metabolites in Tissues of and Milk from Lactating Dairy Cows American Agricultural Services, Inc., USA Study No.: AA070702 Unpublished report, CHA Doc. No.: 1566 FLU
1567 FLU	Willard, T. R.	2008	Magnitude of the Residue of Flutriafol and Three Triazole Metabolites in Tissues of and Eggs from Laying Hens American Agricultural Services, Inc., USA Study No.: AA070703 Unpublished report, CHA Doc. No.: 1567 FLU
1294 FLU	Jones, G.	2005	Final Report on Project AF/7358/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Apple resulting from sequential Applications of CHA 1320 and sampled at Intervals, in Northern France, Southern France, Italy and Spain Agrisearch UK Limited, GBR Study No.: AF/7358/CN Unpublished report, CHA Doc. No.: 1294 FLU
1295 FLU	Jones, G.	2005	Final Report on Project AF/8139/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Apple resulting from sequential Applications of Flutriafol 250 g/l SC and sampled at Harvest, in Northern France, Southern France, Italy and Spain Agrisearch UK Limited, GBR Study No.: AF/8139/CN Unpublished report, CHA Doc. No.: 1295 FLU
1329 FLU	Jones, G.	2006	Final Report on Project AF/10040/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Apple resulting from Sequential Applications of Flutriafol 250 g/l SC and sampled at Intervals, in Greece

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			Agrisearch UK Limited, GBR Study No.: AF/10040/CN Unpublished report, CHA Doc. No.: 1329 FLU
1478 FLU	Jones, G.	2007	Final Report on Project AF/11021/CN : To Determine the Magnitude of Flutriafol Residues in the Raw Agricultural Commodity Apple resulting from three applications of Flutriafol 250 g/l SC and Sampled at Harvest in Greece Eurofins Agrisearch, GBR Study No.: AF/11021/CN Unpublished report, CHA Doc. No.: 1478 FLU
1299 FLU	Jones, G.	2005	Final Report on Project AF/7357/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Grape resulting from two Applications of Flutriafol 250 g/l SC and sampled at Intervals, in Northern France, Southern France, Italy and Southern Spain Agrisearch UK Limited, GBR Study No.: AF/7357/CN Unpublished report, CHA Doc. No.: 1299 FLU
1296 FLU	Jones, G.	2005	Final Report on Project AF/8140/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Grape resulting from two Applications of Flutriafol 250 g/l SC and sampled at Harvest, in Northern France, Southern France, Italy and Spain Agrisearch UK Limited, GBR Study No.: AF/8140/CN Unpublished report, CHA Doc. No.: 1296 FLU
1330 FLU	Jones, G.	2006	Final Report on Project AF/10041/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Grape from Sequential Applications of Flutriafol 250 g/l SC and sampled at Intervals, in Greece Agrisearch UK Limited, GBR Study No.: AF/10041/CN Unpublished report, CHA Doc. No.: 1330 FLU
1396 FLU	Jones, G.	2007	Final Report on Project AF/11020/CN : To determine the Magnitude of Flutriafol Residues in the raw agricultural Commodity Grape from two Applications of Flutriafol 250 g/l SC and sampled at Harvest, in Greece Eurofins Agrisearch UK Limited, GBR Study No.: AF/11020/CN Unpublished report, CHA Doc. No.: 1396 FLU
1606 FLU	Rice, F.	2008	Magnitude of the Residues of Flutriafol and Three Triazole Metabolites in/on Raw Agricultural and Processed Commodities of Grape Following Application of a 125 g/l SC Formulation of Flutriafol ABC Laboratories, Inc., USA Study No.: 62136 Unpublished report, CHA Doc. No.: 1606 FLU
1626 FLU	Willard, T.R.	2009	Magnitude of the Residue of Flutriafol and Three Triazole Metabolites in Banana Fruit American Agricultural Services, Inc. / Morse Laboratories, Inc., USA Study No.: AASI: AA080701 ; Morse: ML08-1425-CVA Unpublished report, CHA Doc. No.: 1626 FLU
1264 FLU	Benet, F. L.	2004	Determination of Residues of Flutriafol in Sweet Peppers LARP, Laboratorio de Análisis de Residuos de Plaguicidas, Universitat Jaume I, ESP Study No.: '040-03 Unpublished report, CHA Doc. No.: 1264 FLU
1265 FLU	Gimeno, C.	2004	Magnitude of Residues in Sweet Pepper following Three Applications with IMPACT 25 SC TrialCamp S.L.L, ESP Study No.: TRC03-15 Unpublished report, CHA Doc. No.: 1265 FLU
1268 FLU	Benet, F. L.	2005	Determination of Residues of Flutriafol in Sweet Peppers (2004) LARP, Laboratorio de Análisis de Residuos de Plaguicidas, Universitat Jaume I, ESP Study No.: '063-04 Unpublished report, CHA Doc. No.: 1268 FLU
1269 FLU	Gimeno, C.	2005	Magnitude of Residues in Sweet Peppers following Three Applications with IMPACT 25 SC (Flutriafol) TrialCamp S.L.L, ESP Study No.: TRC04-26 Unpublished report, CHA Doc. No.: 1269 FLU

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978 FLU	Ciscato, C.H.P. and Gebara, A.B.	2002	Study Report of Impact (Flutriafol) Fungicide Residues at Soya beans I.B/Lab. Resíduos Pesticidas, (IB LRP) Instituto Biológico, Laboratório de Resíduos de Pesticidas, BRA. (English translation performed by Cheminova A/S) Study No.: R/02/RCHE '332/Plantec Report No.: '94/02 Unpublished report, CHA Doc. No.: 978 FLU (English)
979 FLU	Ciscato, C.H.P. and Gebara, A.B.	2002	Study Report of Impact (Flutriafol) Fungicide Residues at Soya beans I.B/Lab. Resíduos Pesticidas, (IB LRP) Instituto Biológico, Laboratório de Resíduos de Pesticidas, BRA. (English translation performed by Cheminova A/S) Study No.: R/02/RCHE '333/Plantec Report No.: '95/02 Unpublished report, CHA Doc. No.: 979 FLU (English)
980 FLU	Ciscato, C.H.P. and Gebara, A.B.	2002	Study Report of Impact (Flutriafol) Fungicide Residues at Soya beans I.B/Lab. Resíduos Pesticidas, (IB LRP) Instituto Biológico, Laboratório de Resíduos de Pesticidas, BRA. (English translation performed by Cheminova A/S) Study No.: R/02/RCHE '334/Plantec Report No.: '96/02 Unpublished report, CHA Doc. No.: 980 FLU (English)
1048 FLU	Mende, P.	2003	Decline of Flutriafol Residues in the raw agricultural Commodity Winter Wheat resulting from two Applications of CHA 1310-03 (a SC Formulation containing 125 g/L Flutriafol) in Northern France and the UK during 2002 GAB Biotechnologie GmbH & IFU Umweltanalytik GmbH, DEU / Agrisearch UK Ltd., GBR Study No.: '20031018/01-RCE, AF/6604/CN Unpublished report, CHA Doc. No.: 1048 FLU
1182 FLU	Jones, G.	2004	Final Report on Project AF/7468/CN : Flutriafol Residues in the raw agricultural commodity Winter Wheat resulting from two Applications of CHA 1310-03 (a SC Formulation containing 125 g/l Flutriafol) in Northern France and the UK during 2003 Agrisearch UK Ltd., GBR Study No.: AF/7468/CN Unpublished report, CHA Doc. No.: 1182 FLU
1047 FLU	Mende, P.	2003	Decline of Flutriafol Residues in the raw agricultural Commodity Winter Wheat resulting from two Applications of CHA 1310-03 (a SC Formulation containing 125 g/L Flutriafol) in Southern France and Spain during 2002 GAB Biotechnologie GmbH & IFU Umweltanalytik GmbH, DEU / Agrisearch UK Ltd., GBR Study No.: '20031018/02-RCE, AF/6605/CN Unpublished report, CHA Doc. No.: 1047 FLU
1183 FLU	Jones, G.	2004	Final Report on Project AF/7469/CN : Flutriafol Residues in the raw agricultural commodity Winter Wheat resulting from two Applications of CHA 1310-03 (a SC Formulation containing 125 g/l Flutriafol) in Southern France and Spain during 2003 Agrisearch UK Ltd., GBR Study No.: AF/7469/CN Unpublished report, CHA Doc. No.: 1183 FLU
1184 FLU	Jones, G.	2004	Final Report on Project AF/7751/CN : Flutriafol Residues in the raw agricultural commodity Winter Wheat resulting from two Applications of CHA 1310-03 (a SC Formulation containing 125 g/l Flutriafol) in Southern France during 2004 Agrisearch UK Ltd., GBR Study No.: AF/7751/CN Unpublished report, CHA Doc. No.: 1184 FLU
1811 FLU	Jones, G.	2010	Magnitude of the Residue of Flutriafol and Three Triazole Metabolites in Wheat Raw Agricultural and Processed Commodities Morse Laboratories, LLC, American Agricultural Services, Inc., USA Study No.: AA080706 Unpublished report, CHA Doc. No.: 1811 FLU
1605 FLU	Rice, F.	2008	Magnitude of the Residues of Flutriafol and Three Triazole Metabolites in/on Raw Agricultural and Processed Commodities of Peanut Following Application of a 125 g/l SC Formulation of Flutriafol ABC Laboratories, Inc., USA Study No.: 62135

Code	Author	Year	Title, Institution, Report reference
			Unpublished report, CHA Doc. No.: 1605 FLU
989 FLU	Ciscato, C.H.P.	2003	Study Report of Residues of the Fungicide IMPACT (Flutriafol) in Coffee Laboratório de Resíduos de Pesticidas, BRA Study No.: R/02/RCHE272/Plantec Unpublished report, CHA Doc. No.: 989 FLU (English)
975 FLU	de Baptista, G.C.	2003	Determination of Residues of Impact in Coffee Universidade de São Paulo — Escola Superior de Agronomia "Luiz de Queiroz" — Laboratório de Resíduos de Pesticidas. Study No.: R/02/RCHE274B/PLANTEC/MACHADO-MG Report No.: '70/4409/03 Unpublished report, CHA Doc. No.: 975 FLU (English)
976 FLU	de Baptista, G.C.	2003	Determination of Residues of Impact in Coffee Universidade de São Paulo — Escola Superior de Agronomia "Luiz de Queiroz" — Laboratório de Resíduos de Pesticidas. Study No.: R/02/RCHE272B/PLANTEC/ENGENHEIRO COELHO-SP Report No.: '70/4407/03 Unpublished report, CHA Doc. No.: 976 FLU (English)
977 FLU	de Baptista, G.C.	2003	Determination of Residues of Impact in Coffee Universidade de São Paulo — Escola Superior de Agronomia "Luiz de Queiroz" — Laboratório de Resíduos de Pesticidas. Study No.: R/02/RCHE273B/PLANTEC/RIBEIRÃO CORRENTE-SP Report No.: '70/4408/03 Unpublished report, CHA Doc. No.: 977 FLU (English)
	Carringer, S.J.	2011	Magnitude and Decline of Flutriafol and Metabolite Residues in/on Coffee Raw Agricultural and Processed Commodities Following Three Applications of Flutriafol 125 g/l SC—2009 Morse Laboratories, LLC, GLP Technologies Study No.: TCI-09-256 Unpublished report