TRIFLUMIZOLE (270)

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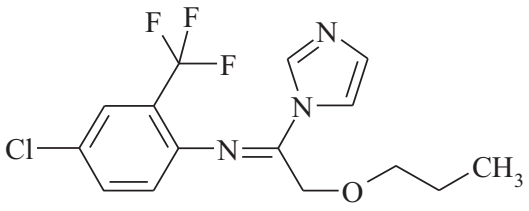
EXPLANATION

Triflumizole is a broad-spectrum foliar fungicide that controls a variety of fungal diseases in fruits and vegetables. It acts as a protectant and as an eradicant by preventing disease symptoms after infection has occurred. The anti-sporulant activity of triflumizole reduces spores after lesions become visible. Triflumizole belongs to the demethylation inhibitor (DMI) group of fungicides classified as Group 3 by the Fungicide Resistance Action Committee (FRAC). Triflumizole has protective and curative actions, and acts as an inhibitor of chitin biosynthesis. The product is mixed with water and applied as a foliar spray using ground equipment equipped for conventional spraying on crops.

Triflumizole was scheduled by the Forty-fourth Session of the CCPR for evaluation as a new compound by the 2013 JMPR. The Meeting received information on identity, animal and plant metabolism, environmental fates in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, dairy cattle feeding studies, and fates of residues in processing.

IDENTITY

Common name	Triflumizole
IUPAC	(E)-4-chloro-α,α,α-trifluoro-N-(1-imidazol-1-yl-2-propoxyethylidene)-o-toluidine
CAS	[N(E)]-4-chloro-N-[1-(1H-imidazol-1-yl)-2-propoxyethylidene]-2-(trifluoromethyl)benzenamine (formerly 1-[(1E)-1-[[4-chloro-2-(trifluoromethyl)phenyl]imino]-2-propoxyethyl]-1H-imidazole)
CAS Registry No	68694-11-1 (formerly 99387-89-0)
CIPAC No	730
Synonyms	NF-114, A815
Structural formula:	
Molecular formula	$C_{15}H_{15}ClF_3N_3O$
Molecular weight	347.5 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Result	Reference
Vapour pressure	$(5.2 \pm 0.4) \times 10^{-2}$ Pa at 20 ºC	Rijsbergen, 2002
Henry's Law Constant	1.71 Pa m^3 mol^{-1} at 20 ºC	Pierce, 1994
Solubility in water (g/L)	12.5 at 20 °C	IPCS Website www.inchem.org (9/2013)

Property	Result		Reference
Solubility in organic solvents at 25 ºC (g/L)	n-Octanol	605	Saito, 1990
	Acetonitrile	1187	
	Ethyl acetate	1486	
	Dichloromethane	3016	
Solubility in organic solvents at 20 ºC (g/L)	n-Hexane	17.6	Soeda, Nomura, 1988
	Methanol	496	
	Xylene	639	
	Acetone	1440	
	Chloroform	2220	
Partition coefficient (Log P_{ow})	5.06 (water at 20 ºC) 5.10 (buffer pH 7 at 20 ºC) 5.12 (buffer pH 8 at 20 ºC)		Higashida, Tanaka, 1998 Nomura, Nakashima, 1987
Hydrolysis rate at 25 ºC	pH buffer	DT_{50} (days)	Soeda, Shiotani, 1987a
	5	8.9	
	7	64.6	
	9	3.9	
Photolysis	DT_{50}=2.55 days at pH 7 at 25 °C		Soeda, Shiotani, 1987b
	$DT_{50=}$16.97 days in distilled water at 25 °C $DT_{50=}$8.04 days in natural water at 25 °C		Knight, 2002
Dissociation constant	pKa=3.7 at 25 ºC		Soeda, Shiotani, 1988

Formulations

Triflumizole is available in the following formulations:

- Suspension concentrate (SC) nominally containing 480 g/L active ingredient;
- Water soluble concentrate (WS) nominally containing 500 g/kg active ingredient;
- Emulsifiable concentrate (EC) nominally containing 150 g/L active ingredient.

The common trade names are Procure® 480SC, Procure® 50WS, Terraguard® SC, and Rocket® EC.

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of triflumizole was investigated in animals, plants, and environmental systems using test material universally labelled with ^{14}C in the phenyl ring (Figure 1). The major metabolites/degradation products from these studies are shown in the table below.

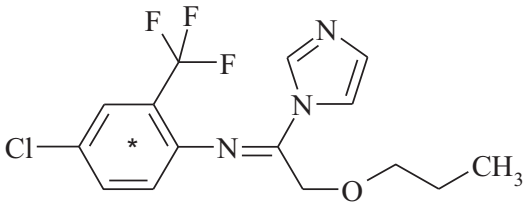
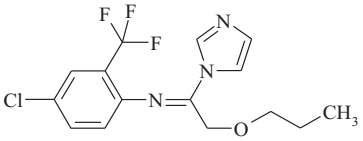
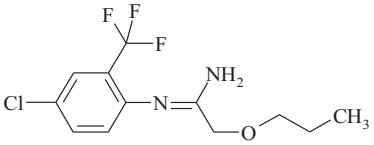
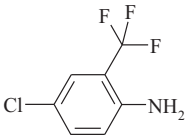
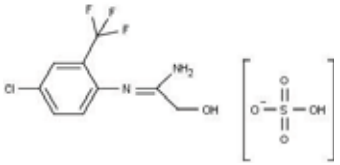
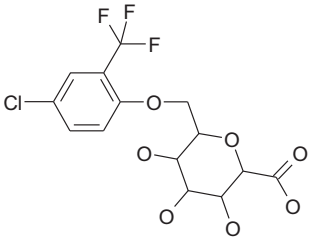
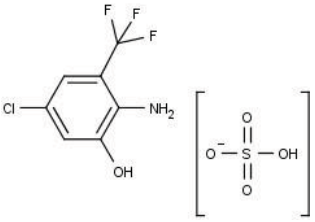
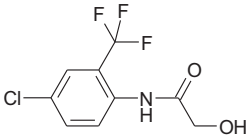
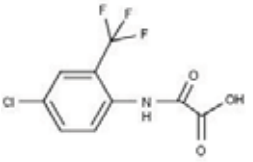
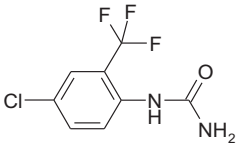
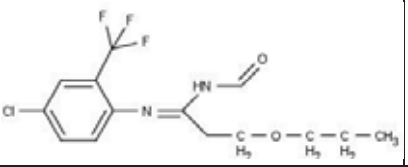
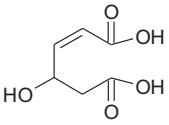
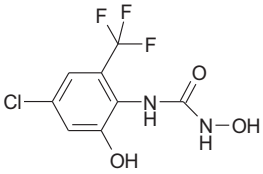
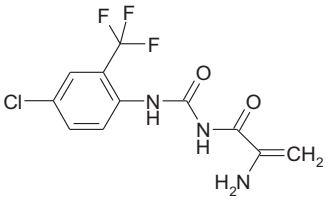
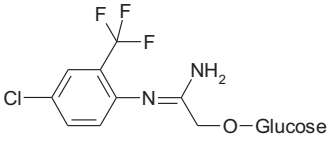
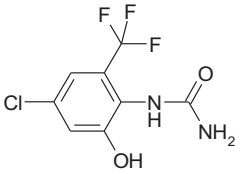
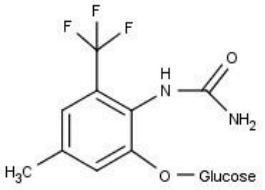


Figure 1 Test material for the studies using radiolabelled triflumizole

Chemicals identified as major components in the metabolic and degradation pathways of triflumizole

Identifier	Chemical Name	Chemical Structure	Test System
Triflumizole	(E)-4-chloro-α,α,α-trifluoro-N-(1-imidazol-1-yl-2-propoxyethylidene)-o-toluidine		Primary Crops
FM-6-1	N'-[4-chloro-2-(trifluoromethyl) phenyl]-2-propoxyethanimidamide		Primary Crops, Soil
FA-1-1	4-chloro-2-(trifluoromethyl)aniline		Egg and Milk
FM-8-1 (Free and sulphate conjugate)	N'-[4-chloro-2-(trifluoromethyl) phenyl]-2-hydroxyethanimidamide		Milk
FA-1-glucuronide	6-[4-chloro-2-(trifluoromethyl) phenoxymethyl]-3,4,5-trihydroxyoxane-2-carboxylic acid		Milk
FA-1-5 (Free and sulphate conjugate)	2-amino-5-chloro-3-(trifluoromethyl)phenol		Milk

Identifier	Chemical Name	Chemical Structure	Test System
FD-2-1	N-[4-chloro-2-(trifluoromethyl) phenyl]-2-hydroxyacetamide		Milk
FD-7-1	{[4-chloro-2-(trifluoromethyl) phenyl]carbamoyl}formic acid		Milk, Soil
FD-9-1 (IV)	[4-chloro-2-(trifluoromethyl) phenyl]urea		Rotational Radish Top Wheat Grain Wheat Hay Wheat Straw
FM-5-1	N-(1-{[4-chloro-2-(trifluoromethyl) phenyl]imino}-3-propoxypropyl)formamide		Egg
XIV	(2Z)-4-hydroxyhex-2-enedioic acid		Rotational Lettuce Radish Top
XXV	1-[4-chloro-2-hydroxy-6-(trifluoromethyl)phenyl]-3-hydroxyurea		Rotational Radish Root
XXVIII	3-(2-aminoprop-2-enoyl)-1-[4-chloro-2-(trifluoromethyl) phenyl]urea		Rotational Wheat Grain
XXIII	Glucose conjugate of FM-8-1		Rotational Lettuce

Identifier	Chemical Name	Chemical Structure	Test System
V	[4-chloro-2-hydroxy-6-(trifluoromethyl)phenyl]urea		Rotational Wheat Straw
XXVI	Glucose conjugate of V		Rotational Wheat Forage

Animal metabolism

The meeting received studies investigating the metabolism of triflumizole in rats, lactating goats and laying hens.

Rats

The metabolism of [^{14}C]triflumizole by rats was evaluated by the WHO Core Assessment Group.

Lactating Goats

The metabolism of [^{14}C]triflumizole by lactating goats was investigated by Knipe and Harned (1985), with a continuation by Knipe (1986). A single goat was orally dosed for 5 consecutive days with [^{14}C]triflumizole labelled in the phenyl ring. Dosing was twice daily by gelatin capsule at 500 µCi/dose, amounting to 14.3 mg/kg/day (280 ppm in feed). Milk, urine, and faeces were collected twice daily for 6 days, and tissue samples (muscle, fat, kidney and liver) were obtained at sacrifice, which was 6 days after the first dose.

Total radioactive residue (TRR) was determined by direct liquid scintillation counting (LSC) of urine and milk, and by combustion/LSC of faeces, blood, and tissues. Characterization of the metabolite profiles of the various matrices was accomplished by solvent extraction, HPLC fraction collection, enzymatic digestion, thin-layer chromatography, and comparison with known standards.

Levels of radiolabel material reached a plateau in milk within approximately 24 hours. Radioactivity in urine and faeces showed an initial rapid increase for the first 36–48 hours after dosing, followed by a gradual increase for the remainder of the study (Table1).

Radioactivity in tissues was generally low (Table 2). Liver contained the highest concentration of residue at approximately 11 mg/kg (0.4% of the cumulative dose). The stomach contents accounted for 8.63 ± 1.01 mg/kg triflumizole-equivalents; radioactivity in the small and large intestines was not determined. Of the radioactivity in the liver tissue, slightly more than half occurred as bound residue. Digestion with protease released approximately 70% of the bound radioactivity.

Characterization of the radioactivity showed that glucuronide- and sulphate-conjugated forms of metabolites FA-1-1 and FA-1-5 occur in urine, milk, liver and kidney; parent compound was not observed. Additional work, using an analytical system with higher resolution, was performed with milk and liver extracts. Those analyses (Table 3) showed major milk metabolites to be FA-1-5-sulphate, FA-1-5-glucaronide, and FM-8-1-sulphate. Minor residues in the milk extract were FD-6-1, FD-2-1, FD-7-1, and FM-6-1. Liver extracts were shown to contain primarily FA-1-5-sulphate and species yielding FA-1-1 after proteolytic digestion. Insufficient data were provided to derive quantitative residue levels for metabolites in milk.

Table1 Cumulative radiocarbon residues in excreta and milk from a goat orally administered [^{14}C]triflumizole

	% of Cumulative Dose [a, b]		
Hours after First Dosing	Urine	Faeces	Milk
4	6.7	< LOD	< LOD
8	11.3	< LOD	< LOD
12	24.4	0.3	0.07
24	36.0	5.3	0.15
36	33.8	8.0	0.15
48	43.0	11.4	0.15
60	41.8	13.4	0.15
72	49.7	16.5	0.16
84	48.4	16.7	0.16
96	53.7	17.7	0.18
108	53.1	17.9	0.16
120	56.0	19.9	0.18

[a] Cumulative dose is total administered dose up to indicated time point.

[b] Based upon 10 equal doses of 500 μCi

Table 2 Radiocarbon residues in tissues of a goat orally administered [^{14}C]triflumizole

Tissue	% TAR	Triflumizole Equivalents (mg/kg ± Std. Dev.)
Kidney	0.01	3.36 ± 0.13
Liver	0.40	11.26 ± 0.59
Muscle	0.18	0.30 ± 0.20
Fat	0.06	0.66 ± 0.05

Table 3 Identification of radioactivity in goat liver and milk following administration of [^{14}C]triflumizole

Metabolite	Matrix	% TRR	Triflumizole Equivalents (mg/kg [a])
FA-1-1	Liver (chloroform fraction)	24.4	2.75
FA-1-1	Liver (dried pellet)	54.8	6.17
FA-1-5-Sulphate	Liver (acetone fraction)	12.4	1.40
	Milk	29.1	–
FA-1-5-Glucuronide	Milk	11.8	–
FD-2-1	Milk	10.4	–
FD-6-1	Milk	5.5	–
FD-7-1	Milk	3.2	–
FM-6-1	Milk	5.2	–
FM-8-1-Sulphate	Milk	12.6	–

[a] Based on a total concentration of triflumizole-equivalent residue in liver of 11.26 mg/kg. Total triflumizole-equivalent residue in milk was not provided.

The proposed metabolic pathway of triflumizole in livestock is shown in Figure2

Laying hens

The metabolism of [^{14}C]triflumizole by laying hens was in investigated by Knipe and Lengen (1986) using triflumizole universally labelled in the phenyl ring. Five hens were dosed, orally, for five consecutive days. Three birds were dosed with 39 ppm in feed and two were dosed with 53 ppm in feed. Eggs and excreta were collected once daily during the dosing period. The birds were killed on the 6th day after the initial dosing and liver, kidneys, fat and muscle were collected for analysis.

Total radioactivity in excreta and tissues was determined by combustion and LSC; radioactivity in egg was determined by LSC directly and was on a whole-egg basis for the three low-

dose birds and separately for egg white and egg yolk for the two high-dose birds. Metabolic profiles of the collected matrices were investigated using solvent extraction, and, for skin-with-fat and liver samples, base or enzymatic digestion. Extracted residues were subjected to HPLC, TLC, and MS analysis, with comparison against known standards.

Analysis of total radioactivity indicates that the majority of the administered dose was excreted (Table 4 4) and that levels did not plateau in eggs within the dosing period (Table 5 5). By the fifth day of dosing, levels of radioactivity in the egg yolk were greater than those in the whites. That pattern may have been evident as early as Day 3 of dosing; however, insufficient samples were collected to make a firm conclusion. In tissues, total radioactivity was approximately an order of magnitude greater in liver and kidney than in fat or muscle (Table 6).

Table 4 Recovery of administered radioactivity in the excreta of chickens administered [^{14}C]triflumizole for 5 days

	μCi Excreted						
Chicken # [a]	Day 1	Day 2	Day 3	Day 4	Day 5	Total	% TAR
6 (control)	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
1	18.81	18.63	21.19	23.35	22.08	104.16	93.0
2	18.90	21.99	16.59	23.18	18.06	98.72	88.1
3	18.35	18.80	13.95	20.38	19.92	91.40	81.6
7	34.69	27.75	27.05	27.95	28.02	145.46	83.4
8	24.26	19.09	30.20	31.08	35.79	140.42	80.5

[a] The dose administered to Chickens l, 2, and 3 was 112 μCi (22.4 μCi/day for 5 days), while the dose administered to Chickens 7 and 8 was 174.5 μCi (34.90 μCi/day for 5 days).

Table 5 Radiocarbon residues in the eggs of chickens administered [^{14}C]triflumizole for 5 days

	Triflumizole equivalents (mg/kg)				
Chicken # [a]	Day 1	Day 2	Day 3	Day 4	Day 5
1	NS [b]	0.028	NS	NS	0.215
2 [c]	0.003	0.053	0.135	0.148	0.242 / 0.303
3	0.024	NS	NS	0.270	0.431
7 [c] Yolk White	0.006 0.009	0.197 0.111	1.080 0.072	NS	2.312 / 1.706 0.401 / 0.408
8 [c] Yolk White	0.002 0.011	0.095 0.081	NS	0.224 [d]	0.899 / 1.187 0.133 / 0.074

[a] Chickens 1, 2, and 3 received 39 mg/kg/day while chickens 7 and 8 received 53 mg/kg/day.

[b] No egg sample obtained.

[c] Chickens 2, 7 and 8 each gave two eggs per day.

[d] This egg was broken when received, so the analysis was performed on the whole egg.

Table 6 Tissue radiocarbon residues in chickens administered [^{14}C]triflumizole for 5 days

Chicken # [b]	Triflumizole Equivalents (mg/kg [a])				
	Liver	Kidney	Fat	Muscle (leg)	Muscle (breast)
1	1.107	0.677	0.067	0.047	0.049
2	0.989	0.537	0.050	0.017	0.026
3	1.232	0.600	0.058	0.026	0.029
7	– [c]	1.115	0.358	0.066	0.087
8	– [c]	0.781	0.312	0.073	0.054

[a] Values obtained for control tissues have been subtracted to obtain the tissues values given.

[b] Chickens 1, 2 and 3 received 39 mg/kg/day, while Chickens 7 and 8 received 53 mg/kg/day.

[c] The livers of these birds were used for metabolite identification and were not analysed for total radioactivity.

HPLC analysis of chicken excreta resulted in three peaks with higher levels of radioactivity at retention time fractions of 8–9 minutes, 24–26 minutes, and 27.5–29 minutes. The first peak was identified as FM-8-1-sulphate based on co-chromatography with an authenticated standard. The second peak was identified as FD-7-1 based on co-chromatography, stability to ß-glucuronidase and sulfatase hydrolysis, and cleavage to FA-1-1 after overnight incubation with HCl at 37 °C. The third peak was resistant to both ß-glucuronidase and sulfatase and was not further investigated. Based on retention time, the third peak may have been FM-6-1.

Analysis of egg yolk and egg white from a high-dose chicken (Chicken #7, Day 5) by HPLC and protease treatments resulted in identification of a number of metabolites (Table 7). The primary components were triflumizole, FD-6-1/FD-4-1 (white), and FA-1-1 (yolk).

Livers from Chickens 7 and 8 (composited) and from Chicken 2 were processed through either a Bligh-Dyer extraction protocol or exhaustive pentane extractions, respectively. For both extraction schemes, the majority of radioactivity in liver was associated with bound residues in the dried pellet (Table 8). HPLC analysis and proteolytic digestion indicate that chicken liver contained a single metabolite: Metabolite FA-1-1 or a metabolite converted to FA-1-1 during proteolytic digestion.

Table 7 Distribution of radioactivity in chicken egg (Chicken #7, day 5)

Total	Egg white (0.401 mg/kg) [a]		Egg yolk (2.312 mg/kg) [a]	
	mg/kg	% TRR	mg/kg	% TRR
Extractables	0.335	83.6 [b]	1.209	52.3 [b]
Triflumizole	0.054	13.4 [c]	0.085	3.7 [c]
FD-6-1/FD-4-1[d]	0.050	12.6 [c]	0.069	3.0 [c]
FM-5-1	0.016	4.1 [c]	–	–
FA-1-1	0.009 + (0.132) [e]	2.2 [d] + (33.05) [e]	(0.0511) [e]	(22.1) [e]
FA-1-5	0.008	1.9 [c]	–	–
FD-2-1	–	–	0.067	2.9[c]
FD-1-1/FM-2-1/FM-3-1[d]	0.008	1.9 [c]	–	–
Polar A	–	–	0.072	3.1 [c]
Polar B	–	–	0.064	2.8 [c]
Not identified [f]	0.058	14.4 [c]	0.210	9.1 [c]
Other	–	–	0.132	5.7 [g]
Losses	–	–	0.183	7.9 [h]
Unextractable	0.069	17.2	0.921	39.8

[a] Data from Table 55.

[b] Extractable prior to concentration and resultant losses of ^{14}C (attributable to FA-1-1).

[c] Percent of total after correction made for loss of ^{14}C after extract concentration

[d] Poor HPLC resolution of the standards did not allow further assignment of peaks in the egg samples.

[e] Evaporative losses of ^{14}C from the extract are most likely FA-1-1; based on that compound's known volatility and the HPLC analysis of the extract of acidic digestion of egg yolk. These residues were characterized by protease digestion (see text) and by acidic Bleidner digestion.

[f] Radioactivity not clearly distinguishable as a definite peak or too low to assign a definite peak identity.

[g] Other-5.7% of yolk was extractable with acetone but could not be separated from the sample matrix for HPLC analysis.

[h] Losses - Additional loss of material balance presumed to be volatiles as FA-1-1

Table 8 Distribution of radioactivity in chicken liver extracts

Fraction	% TRR
Bligh-Dyer Extraction (Chickens 7 and 8)	
Chloroform	15.4
Methanol/water	6.8
Acetone	7.8
Dried Pellet	70.0
Extensive Pentane Extractions (Chicken 2)	

Fraction	% TRR
Pentane	13.7
Acetonitrile	12.9
Dried Pellet	73.4

Summary of animal metabolism

The metabolism of ^{14}C labelled triflumizole has been studied in lactating goats and laying hens. In both studies, the majority (> 80%) of the administered triflumizole was excreted. Livers and kidneys of both species had the highest amount of the retained material. In livers, the predominant residue was FA-1-1. Triflumizole was observed as a major residue, at approximately 13% of the total residue, in egg white only.

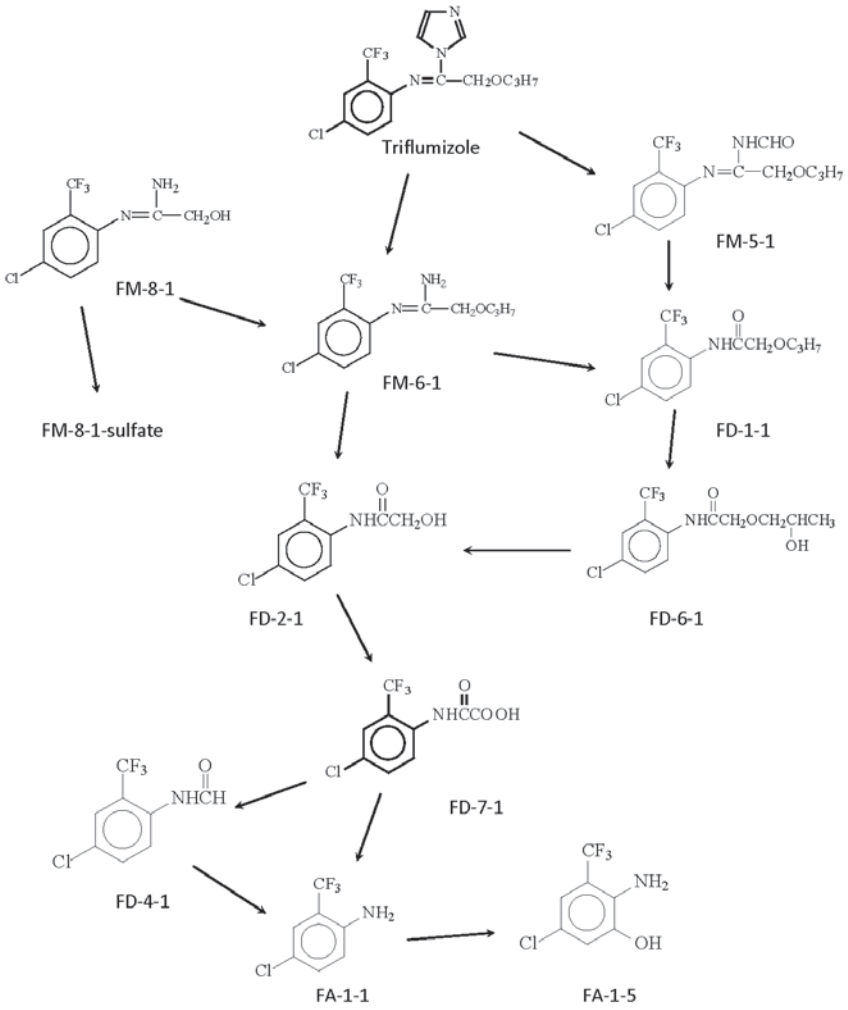


Figure 2 Proposed metabolic pathways of triflumizole in livestock

Plant metabolism

Plant metabolism studies were performed on cucumbers, apples, pears and grapes. Treatment was with triflumizole ^{14}C- universally labelled in the phenyl ring. Metabolites were identified using solvent partitioning and thin-layer chromatography with authentic standards.

Apples

The metabolism of triflumizole in apple trees grown in a greenhouse was investigated (Soeda and Hashimoto, 1987b). Triflumizole, uniformly labelled with ^{14}C in the phenyl ring, was applied as uniformly as possible to four leaves of a branch cluster containing 16–18 leaves. The application rate was approximately 0.127 mg triflumizole/4-leaf cluster. Branches were harvested and separated into treated leaves and upper and lower untreated leaves at 0, 1, 3, 7, 14, 21, 31, 60, and 90 DAT.

Treated leaves were surface washed with chloroform and then extracted three times with methanol:water (7:3, v/v) and the resulting extract was partitioned three times against chloroform. Radioactivity in the washes and extracts was determined by LSC. Radioactivity in the extracted fibrous material was determined by combustion followed by LSC. For samples other than treated leaves, the plant parts were each homogenized in a blender in the presence of dry ice and the radioactivity determined by combustion/LSC. Thin-layer chromatography was used to further analyse leaf washes as well as aqueous and chloroform extracts. Prior to TLC, the aqueous extracts were concentrated *in vacuo* to remove methanol and the residues transferred to 1-butanol. For TLC, the locations of radioactive compounds were determined by autoradiography and identification of residues was by comparison to authentic standards. Quantification of residues was made by collecting the silica gel from the TLC plates and counting by LSC. In the case of the metabolite FA-1-1, which is extremely volatile, TLC scraping and LSC was done immediately after two-dimensional co-chromatography with authentic standards, using UV light to locate the residue for scraping.

Radiocarbon residues in chloroform washes, extracts, and plant material are summarized below (Table 9). The data show that most of the radiocarbon residues were removed in the chloroform wash through the early stages of sampling and that there was very little translocation of radioactivity from treated to untreated leaves. The data also show a rapid decline in radiocarbon residues, with an initial half-life of approximately 4 days.

Table 9 Distribution of radiocarbon residues in apple leaves treated with [^{14}C] triflumizole

Plant Part or Fraction	0 DAT	1 DAT	3 DAT	7 DAT	14 DAT	21 DAT	31 DAT	60 DAT	90 DAT
% TAR →	98.6	80.6	60.8	34.1	17.3	18.1	19.2	15.5	9.0
Radiocarbon Residue (% TRR)									
Treated Leaves									
$CHCl_3$ Wash	100.0	94.4	88.0	69.5	45.3	38.8	38.9	25.2	10.6
MeOH:H_2O									
$CHCl_3$	– [a]	4.1	8.6	19.8	26.0	24.2	22.0	19.9	17.7
Aq. MeOH	–	0.9	2.0	6.5	14.9	19.3	24.2	28.7	39.3
PES	–	0.4	1.0	3.0	9.4	9.2	10.3	15.0	19.8
Subtotal	100.0	99.8	99.6	98.8	95.5	91.5	95.3	88.7	87.3
Untreated Leaves									
Upper Leaves	–	0.1	0.3	0.8	3.1	4.3	3.4	7.7	7.7
Lower Leaves	–	0.1	0.1	0.6	1.2	4.4	1.3	3.8	4.4
Subtotal	–	0.2	0.4	1.4	4.3	8.7	4.7	11.5	12.1
Total	100.0	100.0	99.9	100.2	99.8	100.2	100.1	100.2	99.4
Triflumizole equivalents (mg eq/kg)									
Treated Leaves	51.65	45.42	35.24	22.21	9.87	7.64	7.33	4.58	3.03
Upper Untreated Leaves	–	0.04	0.05	0.06	0.07	0.06	0.04	0.04	0.05
Lower Untreated Leaves	–	0.02	0.03	0.10	0.08	0.24	0.07	0.18	0.13

[a] Not determined.

The TLC analysis shows that the only major (> 10% TRR) identified compounds that appear consistently in the residue data are triflumizole and FM-6-1. As overall residues of radiocarbon decline with increasing time after treatment, FD-2-1 and unidentified metabolites ("Others" in Table 10) become more prominent in the residue profile in terms of %TRR.

Residues of treatment-related compounds in apple fruits were not reported in the study.

Table 10 Identification and distribution of radiocarbon residues in apple leaves treated with [^{14}C] triflumizole. Concentration is only shown if > 0.01 mg eq/kg

Plant Part or Fraction	0 DAT	1 DAT	3 DAT	7 DAT	14 DAT	21 DAT	31 DAT	60 DAT	90 DAT
% TAR→	98.57	80.44	60.53	33.68	16.52	16.57	18.3	13.75	7.86
Radiocarbon Residue (% TRR) [mg eq/kg]									
Chloroform Wash									
Triflumizole	92.8 [0.116]	40.9 [0.042]	21.4 [0.016]	9.5	3.9	3.0	1.5	< 0.01	< 0.01
FM-5-1	0.7	7.4	5.7	2.5	1.9	1.1	1.0	1.2	0.8
FM-6-1	1.4	22.9 [0.023]	29.0 [0.022]	17.9	7.3	6.0	3.6	1.7	0.6
FM-8-1	0.2	1.4	2.5	4.2	3.2	3.0	4.0	2.5	0.9
FD-1-1	1.0	2.4	2.5	2.4	1.6	1.4	1.6	1.2	0.5
FA-1-1	0.2	1.0	0.9	0.4	0.4	0.3	0.4	0.3	0.1
Others	3.9	18.6 [0.019]	26.4 [0.020]	33.5 [0.014]	29.2	27.6	28.6	21.5	9.2
Subtotal	100.0	94.6	88.4	70.4	47.4	42.4	40.8	28.4	12.1
Chloroform Extract									
Triflumizole	–[a]	1.3	2.0	0.9	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
FM-5-1	–	0.2	0.4	0.4	0.7	0.8	0.6	0.5	0.8
FM-6-1	–	1.0	2.3	9.4	7.7	5.1	1.4	0.7	0.5
FM-8-1	–	0.1	0.3	1.3	4.2	4.8	4.1	3.7	1.4
FD-1-1	–	0.2	0.3	0.4	0.4	0.4	0.6	0.6	0.4
FD-2-1	–	0.1	0.1	0.7	0.6	0.5	1.2	1.5	1.5
FD-6-1	–	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
FA-1-1	–	0.1	0.3	0.4	0.7	0.5	1.0	0.7	0.6
Others	–	1.1	2.8	6.5	12.9	14.2	13.4	14.8	15.0
Subtotal	–	4.1	8.6	20.1	27.2	26.4	23.1	22.4	20.2
Aqueous Methanol Extract									
FM-8-1	–	–	–	0.4	0.7	1.0	0.9	1.2	0.9
FD-7-1	–	–	–	1.0	2.3	3.1	3.6	4.4	4.8
M-1 (FD-6-1)[b]	–	–	–	< 0.01	0.4	0.5	0.8	0.9	1.3
M-2	–	–	–	0.2	0.5	0.7	0.6	0.7	1.3
M-3 (FD-2-1, FD-6-1)	–	–	–	0.9	1.9	2.6	2.2	2.4	4.2
M-4 (FA-1-1)	–	–	–	< 0.01	< 0.01	< 0.01	1.2	2.2	2.9
M-5	–	–	–	< 0.01	< 0.01	0.6	1.1	1.5	2.2
M-6	–	–	–	< 0.01	< 0.01	0.8	1.5	2.0	2.8
M-7	–	–	–	0.7	1.8	2.5	2.9	3.6	3.9
M-8 (FD-2-1)	–	–	–	0.6	1.6	2.7	3.1	3.6	6.9
M-9 (FD-2-1)	–	–	–	0.2	0.7	1.1	1.2	< 0.01	< 0.01
Others	–	–	–	2.5	5.6	5.4	6.4	10.0	13.9
Subtotal	–	0.9	2.0	6.5	15.6	21.1	25.4	32.4	45.0
PES	–	0.4	1.0	3.3	9.9	10.1	10.8	16.9	22.6
Sum of Chloroform Wash, Chloroform Extract, and Aqueous Methanol									
Triflumizole	92.8 [0.116]	42.2 [0.043]	23.4 [0.018]	10.4	3.9	3.0	1.5	< 0.01	< 0.01
FM-5-1	0.7	7.6	6.1	2.9	2.6	2.0	1.6	1.7	1.5
FM-6-1	1.4	23.9 [0.024]	31.3 [0.024]	27.3 [0.012]	15.0	11.0	4.9	2.4	1.2
FM-8-1	0.2	1.5	2.9	5.9	8.1	8.8	9.0	7.3	3.2
FD-1-1	1.0	2.6	2.9	2.9	2.0	1.8	2.2	1.7	0.9
FD-2-1[c]	–	0.1	0.1	2.3	4.8	7.0	7.7	7.5	12.6
FD-6-1[c]	–	–	–	–	0.4	0.5	2.2	2.0	2.8
FD-7-1	–	–	–	1.0	2.3	3.1	3.6	4.4	4.8
FA-1-1[c]	0.2	1.1	1.2	0.7	1.0	0.9	2.0	2.0	2.2
Others[d]	3.9	20.6 [0.021]	31.1 [0.024]	43.5 [0.019]	50.0 [0.010]	51.9 [0.011]	54.5 [0.013]	54.0	48.2
PES	–	0.4	1.0	3.0	9.9	10.1	10.8	16.9	22.6
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

[a] Not determined.

[b] Compound in parenthesis is an aglycone of the conjugate.

[c] Includes conjugate.

[d] Includes M-2, M-5, M-6, and M-7.

Pears

The metabolism of triflumizole in pear trees grown in a greenhouse was investigated (Soeda and Hashimoto, 1987a). Treatments were with triflumizole, uniformly labelled with ^{14}C in the phenyl ring, applied either as uniformly as possible to four leaves of a branch cluster containing 8–12 leaves or to the surface of a pear fruit. The leaf application rate was approximately 1 mg triflumizole/4-leaf cluster and the fruit application rate was approximately 0.166 mg triflumizole/fruit. Branches from leaf-treated trees were harvested at 0, 1, 3, 7, 14, 21, 31, 60, and 90 DAT and were divided into treated leaves, untreated leaves and fruit. Treated fruits were harvested 0, 1, 3, 7, and 14 DAT. After harvest, the treated leaves and fruits were surface washed with chloroform. The fruits were then peeled and sectioned into flesh and core. The leaves and fruit peel were separately homogenized and extracted with methanol:water (7:3, v/v). The methanol extract was diluted and partitioned with chloroform. Radioactivity in the extracts was determined directly by LSC. Radioactivity in fibrous residue (PES) was determined by combustion/LSC. The chloroform surface washes, the chloroform partition phases, and the water partition phases were concentrated. The water phases of leaf extracts were extracted with 1-butanol and the solvent was evaporated. All remainders were subjected to quantitative and qualitative 1-D TLC analysis using co-chromatography with known standards. The location of radioactivity on the TLC plates was determined by autoradiography and the activity in ^{14}C spots was determined by LSC of the scrapings derived from the TLC plates. The nature of the aglycones in the combined water phases of all leaf samples from 7 DAT onwards was investigated by means of column chromatography and subsequent treatment of the water-soluble metabolites with ß-glucosidase, cellulase, acid- or alkali pectinase. The reaction products were also subjected to 1-D TLC. In the case of the metabolite FA-1-1, which is extremely volatile, TLC scraping and LSC was done immediately after two-dimensional co-chromatography with authentic standards, using UV light to locate the residue for scraping.

Radiocarbon residues in chloroform washes, extracts, and plant material from pear leaves are summarized below (Table11). The data show that most of the radiocarbon residues were removed in the chloroform wash through the early stages of sampling and that there was very little translocation of radioactivity from treated to untreated leaves. The data also show a rapid decline in radiocarbon residues, with an initial half-life of approximately 4 days.

Table 11 Distribution of radiocarbon residues in leaves and fruit from pear trees leaf-treated with [^{14}C] triflumizole

Plant Part or Fraction	0 DAT	1 DAT	3 DAT	7 DAT	14 DAT	21 DAT	31 DAT	60 DAT	90 DAT
% TAR →	103.04	96.74	77.00	50.20	15.09	25.50	21.97	19.10	16.73
Radiocarbon Residue (% TRR)									
Treated Leaves									
$CHCl_3$ Wash	100.0	95.0	92.8	82.1	48.2	54.7	43.7	24.0	13.6
MeOH:H_2O									
$CHCl_3$	- [a]	3.5	4.2	9.7	20.7	16.9	17.8	18.3	15.7
Aq. MeOH	-	0.9	1.8	5.5	13.6	15.9	23.0	34.9	40.8
PES	-	0.5	0.9	2.4	10.8	9.2	12.2	20.2	25.6
Subtotal	100.0	99.9	99.8	99.8	93.4	96.7	96.8	97.4	95.7
Untreated Leaves	-	0.1	0.2	0.2	6.5	3.1	3.0	2.3	3.2
Fruit	-	< 0.01	< 0.01	0.0	0.1	0.2	0.3	0.4	1.1
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Triflumizole equivalents (mg eq/kg)									
Treated Leaves	73.27	77.66	53.03	36.74	8.79	14.46	10.11	8.83	8.31
Untreated Leaves	– [*]	0.03	0.06	0.08	0.24	0.13	0.06	0.03	0.17
Fruit	–	0.001	0.002	0.006	0.007	0.006	0.006	0.002	0.002

[a] - Not determined

The TLC analysis shows that the only major (> 10% TRR) identified metabolite that appears consistently in the residue data is FM-6-1. With the overall decline in radiocarbon residues with increasing time after treatment, unidentified residues (Others) and bound residues (PES) become more prominent in the residue profile in terms of %TRR (Table12).

Table 12 Identification and distribution of radiocarbon residues in pear leaves treated with [^{14}C] triflumizole

Plant Part or Fraction	0 DAT	1 DAT	3 DAT	7 DAT	14 DAT	21 DAT	31 DAT	60 DAT	90 DAT
% TAR →	103.0	96.7	77.1	51.3	24.0	31.8	31.2	35.5	38.5
Radiocarbon Residue (% TRR)									
Chloroform Wash									
Triflumizole	92.3	51.5	24.1	12.4	4.5	3.4	2.6	0.5	< 0.01
FM-5-1	0.6	4.8	4.5	1.7	1.1	0.9	1.4	0.6	0.4
FM-6-1	1.3	18.4	32.2	29.7	6.6	8.7	6.3	1.3	0.6
FM-8-1	0.2	1.0	2.2	3.4	0.8	2.7	2.6	1.0	0.4
FD-1-1	0.7	1.4	2.4	1.8	2.9	1.9	1.5	1.1	0.5
FA-1-1	0.2	0.7	0.9	0.4	0.2	0.3	0.4	0.1	0.1
Others	4.7	17.1	26.5	31.0	14.1	25.9	16.0	8.2	4.0
Subtotal	100.0	95.0	92.7	80.4	30.3	43.8	30.8	12.9	5.9
Chloroform Extract									
Triflumizole	– [a]	1.1	0.8	0.8	0.3	< 0.01	< 0.01	< 0.01	< 0.02
FM-5-1	–	0.2	0.2	0.3	0.4	0.4	0.2	0.2	0.1
FM-6-1	–	0.3	0.4	1.8	3.0	3.0	1.0	0.4	0.2
FM-8-1	–	0.2	0.3	0.9	1.2	2.0	0.7	0.6	0.4
FD-1-1	–	0.2	0.2	0.2	0.3	0.2	0.3	0.4	0.3
FD-2-1	–	0.1	0.1	0.2	0.5	0.3	0.5	0.4	0.3
FD-6-1	–	0.1	0.1	0.2	0.3	0.2	0.3	< 0.01	< 0.01
FA-1-1	–	0.1	0.1	0.2	0.4	0.3	0.2	0.2	0.1
Others	–	1.3	2.0	4.9	6.6	7.3	9.4	7.7	5.5
Subtotal	–	3.5	4.2	9.5	13.0	13.6	12.5	9.9	6.8
Aqueous Methanol Extract									
FM-8-1	–	–	–	0.2	0.1	0.2	0.2	0.3	0.2
FD-7-1	–	–	–	0.6	0.9	1.5	1.9	2.1	2.0
M-1 (FD-6-1) [b]	–	–	–	0.1	0.3	0.4	0.5	0.5	0.5
M-2	–	–	–	0.2	0.4	0.4	0.5	0.5	0.6
M-3 (FD-2-1)	–	–	–	0.6	1.2	1.4	1.9	1.7	2.1
M-4 (FA-1-1, FD-6-1)	–	–	–	0.2	0.6	0.7	1.3	1.7	1.4
M-5	–	–	–	0.1	0.2	0.4	0.7	0.9	0.7
M-6	–	–	–	0.1	0.3	0.6	0.9	1.2	0.9
M-7	–	–	–	0.5	0.7	1.3	1.5	1.7	1.6
M-8 (FD-2-1)	–	–	–	0.3	0.6	1.0	1.3	1.6	2.4
M-9 (FD-2-1)	–	–	–	0.4	0.5	0.8	1.2	1.1	1.2
Others	–	–	–	2.0	2.6	4.1	4.3	5.3	4.1
Subtotal	–	0.9	1.8	5.4	8.5	12.8	16.2	18.8	17.7
PES	–	0.5	1.2	4.7	48.1	29.9	40.5	58.4	69.5
Sum of Chloroform Wash, Chloroform Extract, and Aqueous Methanol. [mg eq/kg] if > 0.01									
Triflumizole	92.3 [0.954]	52.6 [0.509]	24.9 [0.192]	13.2 [0.066]	4.8	3.4	2.6	0.5	< 0.01
FM-5-1	0.6	5 [0.048]	4.7 [0.036]	2 [0.01]	1.5	1.3	1.5	0.8	0.5
FM-6-1	1.3 [0.013]	18.7 [0.181]	32.6 [0.251]	31.5 [0.158]	9.6 [0.014]	11.6 [0.03]	7.3 [0.016]	1.7	0.8
FM-8-1	0.2	1.2 [0.012]	2.5 [0.019]	4.5 [0.023]	2.2	4.9 [0.012]	3.4	2	0.9
FD-1-1	0.7	1.6 [0.015]	2.6 [0.02]	2 [0.01]	3.2	2.1	1.8	1.5	0.8
FD-2-1 [c]	–	0.1	0.1	1.4	2.6	3.1	4.4	4.3	5.5
FD-6-1 [c]	–	0.1	0.1	0.4	0.8	0.8	1.2	1	0.9
FD-7-1	–	–	–	0.6	0.9	1.5	1.9	2.1	2
FA-1-1 [c]	0.2	0.8	0.9	0.7	0.9	1	1.3	1.1	0.8

Plant Part or Fraction	0 DAT	1 DAT	3 DAT	7 DAT	14 DAT	21 DAT	31 DAT	60 DAT	90 DAT
% TAR →	103.0	96.7	77.1	51.3	24.0	31.8	31.2	35.5	38.5
Others	4.7 [0.049]	19.3 [0.187]	30.3 [0.233]	39 [0.196]	25.3 [0.038]	40.4 [0.103]	34.1 [0.075]	26.5 [0.051]	18.4 [0.031]
PES	–	0.5	1.2	4.7 [0.024]	48.1 [0.073]	29.9 [0.076]	40.5 [0.089]	58.4 [0.112]	69.5 [0.116]
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

[a] Not determined.

[b] Aglycone formed by enzymatic hydrolysis.

[c] Includes conjugate.

Radiocarbon residues in chloroform washes, extracts, and plant material from pear fruit are summarized below (Table). The data show that through the two-week sampling period, nearly all of the radiocarbon residues were associated with the peel of the fruit and that most of that radioactivity was removed in the chloroform wash from fruit at the first three sampling intervals. The data also show a rapid decline in radiocarbon residues, with an initial half-life of between 1 and 3 days, and a slower rate of decline between 3 and 14 DAT.

Table 13 Distribution of radiocarbon residues in fruit from pear trees treated with [^{14}C] Triflumizole

Plant Part or Fraction	0 DAT	1 DAT	3 DAT	7 DAT	14 DAT
% TAR →	100.4	68.6	57.7	55.0	41.7
Radiocarbon Residue (% of Applied)					
Peel					
$CHCl_3$ Wash	100.0	63.3	47.6	39.0	23.2
MeOH:H_2O					
$CHCl_3$	– [a]	22.1	28.7	27.1	27.8
Aq. MeOH	–	1.5	2.5	4.8	7.6
Acetone	–	4.5	7.0	7.5	12.4
NaOH					
$CHCl_3$	–	6.3	9.6	14.1	19.1
H_2O	–	0.4	0.5	1.0	1.4
PES	–	1.4	2.1	2.9	4.3
Subtotal	100.0	99.4	97.9	96.5	95.8
Flesh	–	0.4	1.9	3.1	3.8
Core	–	0.2	0.2	0.3	0.4
Total	100.0	100.0	100.0	99.9	100.0
Triflumizole equivalents (mg eq/kg)					
Peel	9.61	6.52	4.79	3.05	2.11
Flesh	–	0.005	0.013	0.017	0.013
Core	–	0.013	0.008	0.015	0.010
Total	1.19	0.62	0.53	0.42	0.27

[a] Not determined

The metabolic profile in pear fruits is summarized in Table 214. Major residues in pear fruit were triflumizole (through 3 DAT), FM-6-1, and FD-1-1 (3 DAT only). Residues of FM-6-1 and FA-1-1 were greater in fruit than in leaves. Individual unknown metabolites (Others) were < 5% TRR.

Table 2 Identification and distribution of radiocarbon residues in pear fruits treated with [^{14}C] triflumizole

Plant Part and Compound	0 DAT		1 DAT		3 DAT		7 DAT		14 DAT	
% TAR →	100.35		68.59		57.68		54.97		41.69	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Peel										
Triflumizole	93.56	1.11	25.67	0.16	13.89	0.07	7.86	0.03	1.80	< 0.01
FM-5-1	1.16	0.01	7.77	0.05	4.51	0.02	2.95	0-01	2.30	0.01

Plant Part and Compound	0 DAT		1 DAT		3 DAT		7 DAT		14 DAT	
% TAR →	100.35		68.59		57.68		54.97		41.69	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
FM-6-1	< 0.01	< 0.01	25.92	0.16	30.03	0.16	32.36	0.14	32.05	0.09
FM-8-1	< 0.01	< 0.01	0.63	< 0.01	1.23	0.01	1.64	0.01	1.94	0.01
FD-1-1	1.33	0.02	9.30	0.06	10.51	0.06	7.53	0.03	7.32	0.02
FA-1-1	0.39	0.01	4.51	0.03	4.26	0.02	4.98	0.02	5.13	0.01
Others	3.56	0.04	24.22	0.15	31.47	0.17	36.40	0.15	40.99	0.11
PES	– [a]	–	1.36	0.01	2.08	0.01	2.86	0.01	4.32	0.01
Flesh	–	–	0.42	< 0.01	1.87	0.01	3.13	0.02	3.79	0.01
Core	–	–	0.2	< 0.01	0.15	< 0.01	0.29	< 0.01	0.36	< 0.01

[a] Not determined

Grapes

The metabolism of triflumizole in grape vines grown in a greenhouse was investigated (Soeda and Hashimoto, 1987c). Treatments were with triflumizole, uniformly labelled with ^{14}C in the phenyl ring, applied either to a branch of approximately 10 leaves and blossoms at the berry crossing time (67 days prior to harvesting mature fruit) or to a bunch of young fruits 35 days before harvesting mature fruit. For both treatments, the rate was equivalent to 280 g ai/ha. Branches were harvested 0, 3, 7, 14, 31, and 67 DAT and were divided into treated leaves and fruit. In the second experiment, treated fruits were harvested 0, 3, 7, 14, and 35 DAT. Samples were surface washed with dichloromethane (DCM) and then extracted with methanol:water (8:2, v/v). The methanol extract was partitioned against DCM. Radioactivity in the washes and extracts was determined by LSC directly and in PES by combustion/LSC. The concentrated DCM wash, DCM partition phase, and the water partition phase were subjected to 1D-TLC analysis using co-chromatography of known standards. The activity in ^{14}C-spots was determined by LSC of the scrapings derived from the TLC plates.

Most of the radioactivity in leaves was recovered from the surface wash. Residues decreased from 99% of the applied dose (AD) to 15% TAR at 67 DAT (Table). Radioactivity was translocated to fruits at less than 2% TAR. The parent compound was found to be the major residue (decreasing from 98% TRR at 0 DAT to 7.5% TRR at 67 DAT). The only other major residue was FM-6-1 (11.1% TRR at 67 DAT). Post-extraction residues in leaves gradually increased from 3.1% TRR (1.73 mg eq/kg) to 26.02% TRR (1.79 mg eq/kg) over the course of the study. Minor metabolites and those remaining at the origin increased to 39% TRR at 67 DAT (

Table).

Table 15 Residues in grape leaf and fruit after application of [^{14}C] triflumizole to leaves, expressed as % TAR and mg eq/kg (triflumizole-equivalents)

Plant Part or Fraction	0 DAT		3 DAT		7 DAT		14 DAT		31 DAT		67 DAT	
	% TAR	mg eq/kg	% TAR	mg eq/kg	% TAR	mg eq/kg	% TAR	mg eq/kg	% TAR	mg eq/kg	% TAR	mg eq/kg
Fruit	0.96	0.64	1.72	1.00	1.97	0.82	1.28	0.47	0.43	0.26	0.24	0.11
Leaves												
Surface wash	99.0	65.6	85.9	50.17	75.6	31.21	34.7	12.73	17.3	10.43	4.8	2.21
MeOH extract												
$CHCl_3$ phase	n.a.	n.a.	5.8	3.39	7.6	3.13	3.4	1.248	3.7	2.23	2.9	1.33
Water phase	n.a	n.a.	0.9	0.53	2.7	1.11	2.3	0.844	3.4	2.05	3.8	1.75
PES			3.0	1.75	4.3	1.77	4.1	1.505	4.1	2.47	3.9	1.79
Total	99.0	65.6	95.6	55.9	90.1	37.2	44.4	16.3	28.5	17.2	15.0	6.9

Table 16 Residues in grape leaf after application of [^{14}C] triflumizole to leaves, expressed as % TRR [a] and mg eq/kg (triflumizole-equivalents)

	0 DAT		3 DAT		7 DAT		14 DAT		31 DAT		67 DAT	
Compound or fraction [b]	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triflumizole	98	64.3	87.4	48.8	80.0	29.7	70.8	11.5	50.9	8.8	7.5	0.51
FM-5-1	0.16	0.11	0.65	0.36	0.68	0.25	0.68	0.11	0.91	0.16	< 0.01	< 0.01
FM-6-1	0.12	0.08	2.13	1.19	3.08	1.15	3.98	0.65	7.09	1.22	11.07	0.76
FM-8-1	0.14	0.09	0.44	0.25	0.81	0.30	1.19	0.19	2.35	0.40	4.87	0.33
FD-1-1	0.18	0.11	0.97	0.54	1.67	0.62	1.28	0.21	0.98	0.17	2.47	0.17
FD-2-1	–	–	0.29	0.16	0.49	0.18	0.99	0.16	1.83	0.31	5.14	0.35
FD-6-1	–	–	0.07	0.04	0.07	0.03	0.11	0.02	0.32	0.05	1.73	0.12
FD-7-1	–	–	0.06	0.03	0.13	0.05	0.29	0.05	0.53	0.09	1.20	0.08
FA-1-1	0.15	0.10	0.28	0.16	0.36	0.13	0.34	0.06	0.21	0.04	0.73	0.05
Other	1.25	0.82	4.58	2.56	7.92	2.95	11.21	1.82	20.61	3.54	39.3	2.70
PES	–	–	3.10	1.73	4.79	1.78	9.12	1.48	14.29	2.46	26.02	1.79
Total	100	–	100	–	100	–	100	–	100	–	100	–

[a] Expressed as percentage of total radioactivity at sampling time

[b] From surface wash and methanol extracts; Other includes minor components and TLC-origin activity.

After fruit application, most of the radioactivity was recovered from the surface wash, and decreased to approximately 7.5% TAR at 35 DAT (Table 17 317). Triflumizole decreased from 95.45% TRR (7.44 mg eq/kg) to 31% TRR (0.31 mg eq/kg) at 35 DAT, and the metabolite FM-6-1 increased to 7.6% TRR (0.07 mg eq/kg) at 35 DAT. Post-extraction solids increased to 17.1% TRR at 35 DAT. The results are summarized in Table 18.

Table 17 3Residues in grape fruits after application of [^{14}C] triflumizole to fruits, expressed as % TAR [a] and mg eq/kg

	0 DAT		3 DAT		7 DAT		14 DAT		35 DAT	
%TAR →	100		49.7		24.6		22.7		24.8	
Fraction	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Surface wash	100	7.79	88.24	5.44	68.1	1.46	44.4	0.68	30.3	0.298
MeOH extract										
$CHCl_3$ phase	–	–	7.8	0.488	16.1	0.348	22.2	0.341	22.5	3.73
Water phase	–	–	1.7	0.106	6.9	0.148	15.6	2.379	30.0	4.99
PES	–	–	2.2	0.002	8.9	0.152	17.8	0.295	17.1	2.83

[a] Expressed as percentage of total radioactivity as applied

Table 18 Residues in grape fruits after application of [^{14}C] triflumizole to fruits, expressed as % TRR [a] and mg eq/kg

	0 DAT		3 DAT		7 DAT		14 DAT		35 DAT	
Compound or fraction [b]	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triflumizole	95.45	7.44	86.89	5.37	69.77	1.49	49.38	0.76	31.17	0.31
FM-5-1	0.25	0.02	0.66	0.04	1.10	0.02	1.01	0.02	< 0.01	< 0.01
FM-6-1	1.43	0.11	1.29	0.08	1.75	0.04	2.56	0.04	7.59	0.07
FM-8-1	< 0.01	< 0.01	0.71	0.04	1.87	0.04	3.88	0.06	3.64	0.04
FD-1-1	0.31	0.02	2.96	0.18	4.03	0.09	4.89	0.08	3.67	0.04
FD-2-1	–	–	0.77	0.05	3.05	0.07	6.83	0.11	7.63	0.07
FD-6-1	–	–	0.20	0.01	0.57	0.01	0.66	0.01	1.69	0.02
FD-7-1	–	–	0.16	0.01	0.49	0.01	0.75	0.01	2.30	0.02
FA-1-1	0.01	< 0.01	0.62	0.04	1.10	0.02	0.97	0.01	0.65	0.01
Other	2.55	0.20	3.46	0.21	7.4	0.16	11.27	0.17	24.58	0.24
PES	–	–	2.19	0.14	8.87	0.19	17.80	0.27	17.08	0.16

[a] Expressed as percentage of total radioactivity at sampling time

[b] From surface wash and methanol extracts; Other includes minor components and TLC-origin activity.

Cucumbers

The metabolism of triflumizole in cucumbers was studied under greenhouse conditions by Soeda (1983). Three types of treatment were made: Foliar treatment to investigate metabolism, fruit treatment to investigate metabolism, and foliar treatment to investigate translocation.

In the foliar metabolism study, radio-labelled triflumizole was applied to leaves (0.13 mg/plant) of multiple plants at the 2nd-to-3rd leaf stage. At sampling times of 1, 3, 7, 14, 21, and 45 (one plant) days after treatment (DAT), two whole plant was harvested, the roots were washed with water, and the plants were sectioned into root, lower part (below the treated leaf), treated leaf, upper part (above the treated leaf), and fruit. Treated leaves were surface washed with chloroform and then the leaf was homogenized with methanol (80%). The methanol extract was adjusted to 50% with distilled water and then partitioned against chloroform. Chloroform leaf wash and extract were assayed, separately, by 1-D and 2-D TLC for residue identification. The aqueous extract was assayed for radioactivity by LSC, and the extracted treated leaf and the remaining plant parts (post-extraction solids, PES) were assayed for radioactivity by combustion followed by LSC. Levels of total radioactivity in the various washes, extracts, and tissues demonstrate that the majority of the applied material is recovered in the surface wash of treated leaves and that very little is distributed throughout the plant (Table19). Residue in the leaves decreased with increasing DAT. The decrease was most significant for triflumizole, with no concomitant increase seen in any of the identified metabolites (Table). Residue levels (mg eq/kg) were not provided in the study.

Table19 Distribution of radiocarbon residues in treated cucumber plants

	1 DAT		3 DAT		7 DAT		14 DAT		21 DAT		45 DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant parts and fractions	87.0% TAR	0.113	81.6 % TAR	0.106	62.4 % TAR	0.081	44.2 % TAR	0.057	28.4 % TAR	0.037	13.8 % TAR	0.018
Treated Leaf												
Chloroform wash	87.4	0.099	84.2	0.089	81.7	0.066	73.8	0.042	54.9	0.020	29.7	0.005
80% Me0H extract												
Chloroform fraction	7.2	0.008	8.8	0.009	9.3	0.008	13.1	0.008	16.9	0.006	13.8	0.002
Aqueous fraction	5.3	0.006	6.5	0.007	8.5	0.007	9.7	0.006	19.7	0.007	33.3	0.006
PES	0.1	< 0.001	0.4	< 0.001	0.3	< 0.001	2.7	0.002	7	0.003	14.5	0.003
Subtotal	100	0.113	99.9	0.106	100.6	0.082	99.3	0.057	98.6	0.036	91.3	0.016
Upper parts	0.01	< 0.001	0.01	< 0.001	0.08	< 0.001	0.52	< 0.001	0.74	< 0.001	4.6	0.001
Lower parts	0.01	< 0.001	0.07	0.000	0.11	< 0.001	0.16	< 0.001	0.49	< 0.001	0.43	< 0.001
Root	– [a]	< 0.001	–	< 0.001	–	< 0.001	0.02	< 0.001	0.28	< 0.001	1.7	< 0.001
Fruit	– [b]	< 0.001	–	< 0.001	–	< 0.001	–	< 0.001	–	< 0.001	1.9	< 0.001

[a] Less than 0.01.

[b] Fruit were not present.

Table 20 Distribution and identification of radiocarbon residues in cucumber leaves

Fraction	Analyte	1 DAT		3 DAT		7 DAT		14 DAT		21 DAT		45 DAT	
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
		82.3% TAR	0.107	75.9% TAR	0.099	56.8% TAR	0.074	38.4% TAR	0.050	20.4% TAR	0.027	6.0% TAR	0.008
Surface wash	Triflumizole	79.5	0.085	73.4	0.072	52.3	0.039	28.9	0.014	15.7	0.004	5	–
	FM-5-1	1.7	0.002	2.2	0.002	3	0.002	4.2	0.002	2	0.001	3.3	–

Fraction	Analyte	1 DAT		3 DAT		7 DAT		14 DAT		21 DAT		45 DAT	
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
	FM-6-1	5	0.005	7.2	0.007	11.6	0.009	13.8	0.007	13.2	0.004	5	–
	FM-8-1	–[A]	–	–	–	3.3	0.002	3.1	0.002	5.4	0.001	6.7	0.001
	FD-1-1	1.6	0.002	2.9	0.003	4.4	0.003	2.9	0.001	2.9	0.001	3.3	–
	FA-1-1	0.4	–	0.7	0.001	0.5	–	1.3	0.001	0.5	–	–	–
	Others[b]	4.3	0.005	4.1	0.004	14.6	0.011	30.7	0.015	36.8	0.010	45	0.004
	Subtotal	92.3	0.099	90.5	0.089	89.8	0.066	84.9	0.042	76.5	0.020	68.3	0.005
Flesh extract	Triflumizole	1.8	0.002	3.8	0.004	1.4	0.001	2.1	0.001	1.5	0.000	1.7	–
	FM-5-1	0.2	–	0.3	–	0.2	–	0.3	–	–	–	–	–
	FM-6-1	1.8	0.002	1.3	0.001	1.9	0.001	2.9	0.001	2	0.001	1.7	–
	FM-8-1	0.4	–	1.2	0.001	1.1	0.001	1.8	0.001	5.9	0.002	1.7	–
	FD-1-1	0.2	–	0.3	–	0.5	–	0.5	–	0.5	0.000	1.7	–
	FA-1-1	–	–	0.1	–	0.4	–	0.3	–	0.5	–	–	–
	Others	3.2	0.003	2.5	0.002	4.8	0.004	7.3	0.004	13.2	0.004	25	0.002
	Subtotal	7.7	0.008	9.5	0.009	10.2	0.008	15.1	0.008	23.5	0.006	31.7	0.002
Total (Surface & flesh)	Triflumizole	81.3	0.087	77.2	0.076	53.7	0.040	31	0.015	17.2	0.005	6.7	0.001
	FM-5-1	1.9	0.002	2.5	0.002	3.2	0.002	4.4	0.002	2	0.001	3.3	–
	FM-6-1	6.8	0.007	8.6	0.008	13.6	0.010	16.7	0.008	15.2	0.004	6.7	0.001
	FM-8-1	0.4	–	1.2	0.001	4.4	0.003	4.9	0.002	11.3	0.003	8.3	0.001
	FD-1-1	1.8	0.002	3.2	0.003	4.9	0.004	3.4	0.002	3.4	0.001	5	–
	FA-1-1	0.4	–	0.8	0.001	0.9	0.001	1.6	0.001	1	–	–	–
	Others	7.4	0.008	6.6	0.007	19.4	0.014	33.1	0.017	50	0.013	70	0.005
	Total	100	0.107	100	0.099	100	0.074	100	0.050	100	0.027	100	0.008

[a] Less than 0.1

[b] Sum of minors and origin materials.

In the fruit metabolism study, radio-labelled triflumizole (0.041 mg/plant) was applied as small droplets to a small fruit. Two fruits were harvested at each sampling time of 1, 3, 7, and 14 DAT. Total radioactive residues and residue identification and characterization was by the same methods used for treated leaves. The behaviour of total residues (Table) and the distribution of residues (Table 22) is similar to that observed for leaves.

Table21 Distribution of radiocarbon residues in treated cucumber fruit

		1 DAT		3 DAT		7 DAT		14 DAT	
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plant part	Fraction	100.4% TAR	0.041	96.0% TAR	0.039	94.2% TAR	0.038	83.4% TAR	0.034
Surface	Chloroform wash	71	0.029	54.1	0.021	41.8	0.016	20.9	0.007
Peel	80% Me0H								
	Chloroform extract	21.9	0.009	23	0.009	23	0.009	18.7	0.006
	Aqueous residue	1.8	0.001	3.1	0.001	8.8	0.003	21.7	0.007
	PES	3.2	0.001	5.6	0.002	9.1	0.003	16.4	0.006
	Subtotal	26.9	0.011	31.8	0.013	41	0.016	56.8	0.019
Flesh	80% Me0H								
	Chloroform extract	1.7	0.001	10.2	0.004	12.4	0.005	14.5	0.005
	Aqueous residue	0	0.000	2	0.001	3.1	0.001	5.3	0.002
	PES	0.4	0.000	2	0.001	1.7	0.001	2.5	0.001
	Subtotal	2.1	0.001	14.2	0.006	17.2	0.007	22.3	0.008
Peel + Flesh	80% Me0H								
	Chloroform extract	23.6	0.010	33.2	0.013	35.5	0.013	33.2	0.011

		1 DAT		3 DAT		7 DAT		14 DAT	
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
	Aqueous residue	1.8	0.001	5.1	0.002	11.9	0.005	27	0.009
	PES	3.6	0.001	19.8	0.008	26.3	0.010	38.7	0.013
	Subtotal	29	0.012	45.9	0.018	58.2	0.022	67.1	0.023
Weight of fruit, g		39		75		160		280	

Table 22 Distribution and Identification of Radiocarbon Residues in Treated Cucumber Fruit

Fraction	Analyte	Radiocarbon Residue (% TRR) [a]			
		1 DAT 95.0% TAR	3 DAT 83.8% TAR	7 DAT 72.8% TAR	14 DAT 45.1% TAR
Surface Wash	Triflumizole	69.1 (0.027 mg eq/kg)	57.2 (0.020 mg eq/kg)	46.0 (0.014 mg eq/kg)	24.4 (< 0.01 mg eq/kg)
	FM-5-1	0.8	0.5	0.7	1.1
	FM-6-1	1.7	1.0	1.8	2.7
	FM-8-1	0.3	0.4	0.4	0.9
	FD-1-1	0.3	0.5	0.7	1.1
	FA-1-1	– [b]	–	0.1	0.7
	Others [c]	2.8	2.5	4.4	7.5
Fruit [Peel/Flesh]	Triflumizole	15.9 [15.4/0.5]	17.8 [14.0/3.8]	15.8 [13.9/1.9]	17.5 [15.7/1.8]
	FM-5-1	0.5 [0.5/–]	0.8 [0.6/0.2]	1.1 [1.0/0.1]	1.1 [0.7/0.4]
	FM-6-1	2.9 [2.3/0.6]	9.4 [4.1/4.8]	15.4 [5.1/10.3]	24.2 [7.1/17.1]
	FM-8-1	0.2 [0.2/–]	0.8 [0.5/0.4]	1.1 [0.7/0.4]	2.7 [1.1/1.6]
	FD-1-1	1.3 [1.2/0.1]	2.5 [2.0/0.5]	2.5 [2.2/0.3]	1.6 [1.3/0.2]
	FA-1-1	0.6 [0.5/0.1]	0.8 [0.5/0.4]	1.4 [1.2/0.1]	1.6 [0.9/0.7]
	Others	3.5 [3.1/0.4]	5.8 [4.2/1.7]	8.7 [5.8/2.9]	12.9 [7.8/5.1]
Total	Triflumizole	84.9	74.9	61.8	41.9
	FM-5-1	1.4	1.3	1.8	2.2
	FM-6-1	4.6	10.4	17.2	27.1
	FM-8-1	0.5	1.2	1.5	3.5
	FD-1-1	1.6	3.0	3.2	2.7
	FA-1-1	0.6	0.8	1.5	2.2
	Others	6.3	8.4	13.0	23.1
	Total	100.0	100.0	100.0	100.0

[a] Except for triflumizole in surface wash, all other residues are < 0.01 mg eq/kg.

[b] Less than 0.1%.

[c] Sum of minors and origin materials.

To investigate translocation, an application of radio-labelled triflumizole (0.16 mg/plant) was made to the surface of a leaf which had a young fruit growing at the base of its petiole. Fruits were harvested 3, 7, and 14 DAT, weighed, and total radioactive residue determined by combustion/LSC. The results (Table) show that very little radioactivity was translocated from the treated leaf to the adjacent fruit.

Table 23 Translocation of Triflumizole Residues from a Treated Leaf to an Adjacent Fruit

Measurement	3 DAT	7 DAT	14 DAT
Fruit weight (g)	5.2	74.5	234
Radiocarbon residue (% of applied)	0.02	0.05	0.07
Radiocarbon residue (mg eq/kg triflumizole equivalents)	0.01	0.01	0.01

Summary of plant metabolism

The metabolism of triflumizole was studied in apples (leaves only), pears, cucumbers and grapes. The metabolic profile observed in these studies was similar across all four crops (

). The most abundant residue was parent triflumizole. Generally triflumizole decreased rather rapidly (half-life on the order of 3 days), with the exception of grapes where metabolism was considerably

slower. Loss of triflumizole was accompanied by an increase in the FM-6-1 metabolite, which was the only metabolite to consistently occur at ≥ 10% TRR.

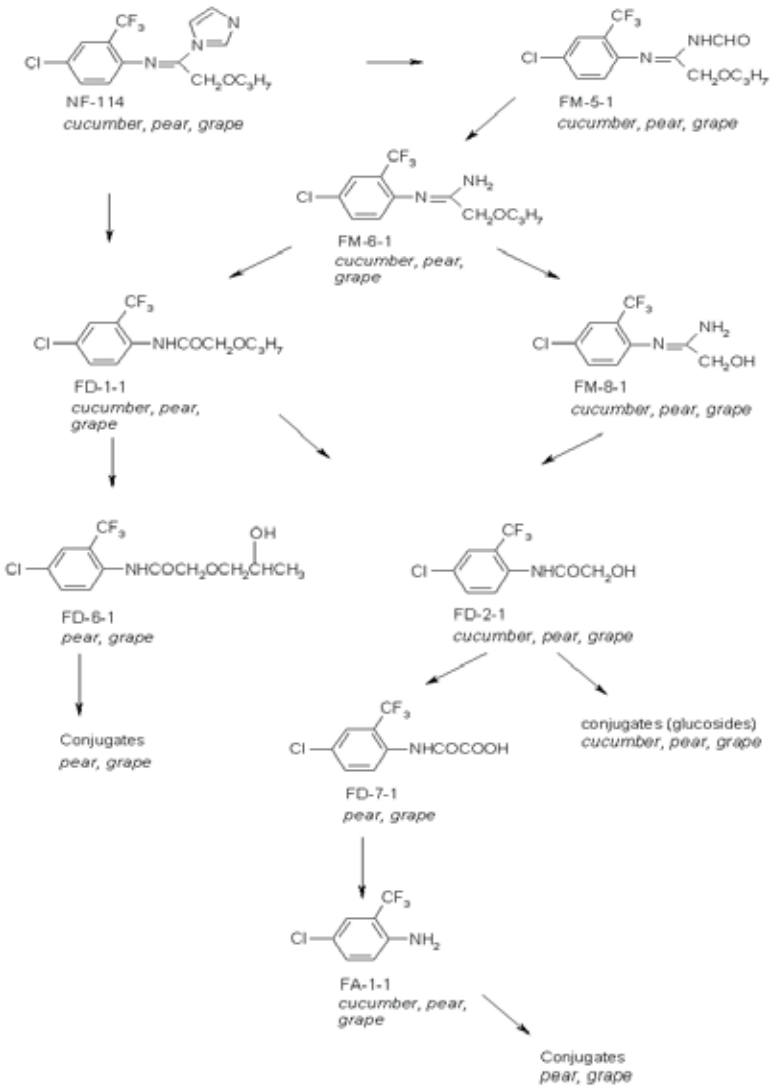


Figure 3 Proposed metabolic pathways of triflumizole in plants

ENVIRONMENTAL FATE

The Meeting received information on aqueous and soil-surface photolysis, soil dissipation, aerobic soil metabolism, and confined and field rotational crop studies.

Environmental fate in soil

The fate and behaviour of triflumizole in soil were investigated using [^{14}C] triflumizole labelled in the phenyl ring.

Aerobic degradation

Study 1

The metabolism and degradation rate (DT_{50} and DT_{90}) of triflumizole was investigated in a sandy loam soil (Harned, 1985; Doc. No. 84253). Soil was added to incubation flasks and adjusted to 75% 1/3-bar moisture. Radio-labelled triflumizole was added to achieve a concentration of 0.60 mg eq/kg (approximately 0.56 kg/ha in the top 7.6 cm of soil). The flasks were capped with trapping towers and incubated, in darkness at 25 °C. Samples for extractable, bound, and volatile ^{14}C were collected at 0, 0.33, 1, 2, 3, 4, 8, 12, 24, and 42 days after treatment.

Table 24 Soil characteristics

Parameters	Soil
pH	5.6
Organic matter (%)	7.34
Cation exchange capacity (meq/100 g soil)	43.5
Soil type (USDA)	Sandy loam
Clay (%)	6.4
Silt (%)	26.0
Sand (%)	67.6

Total extractable residues were determined by acetone extraction and LSC. Extracts were examined by HPLC, using known standards, to identify individual components. Quantification of extracted components was by fraction collection and LSC. Bound residues were determined by combustion/LSC. Total volatile organics were determined by combustion/LSC of the foam plugs in the trapping towers and $^{14}CO_2$ was determined by ascarite traps in the trapping towers. The results are summarized below (Table). The residue profile of the extracted radiocarbon is summarized in Table . The profile shows and increasing proportion of bound residues with increased incubation time and also indicates that the majority of the volatile radioactivity is not in the form of CO_2.

Table 25 Distribution of radiocarbon following aerobic incubation of soil treated with [^{14}C] triflumizole

Fraction	Radiocarbon Residues (% TAR)									
	0 DAT	0.33 DAT	1 DAT	2 DAT	3 DAT	4 DAT	8 DAT	12 DAT	24 DAT	42 DAT
Extractable [a]	101.1	107.9	93.7	94.7	100.0	96.3	90.5	84.8	70.7	52.6
Bound	1.8	2.8	4.3	4.6	5.8	5.8	7.5	9.5	12.5	19.3
Volatile [b]	–	0.3	0.3	0.8	1.1	1.5	3.1	3.5	3.9	7.9
CO_2	–	–	–	0.0	–	–	–	–	0.1	0.8
Total Recovery	102.9	111.0	98.3	100.1	106.9	103.6	101.1	97.8	87.2	80.6

[a] Acetone extract

[b] Trapped in polyurethane plug

Table 26 Profile of extractable radiocarbon residues from soil treated with [^{14}C] triflumizole

Compound	Extractable Radiocarbon Residues (% TAR)									
	0 DAT	0.33 DAT	1 DAT	2 DAT	3 DAT	4 DAT	8 DAT	12 DAT	24 DAT	42 DAT
Triflumizole	88.1	95.0	78.6	82.5	84.1	83.1	73.7	63.1	40.4	26.0
FA-1-1	1.3	1.2	2.4	4.1	6.3	7.4	9.4	13.1	21.4	22.2
FM-6-1	2.8	0.0	0.0	0.0	1.4	0.0	0.0	0.0	0.0	0.0
FM-5-1	0.0	0.7	0.0	0.7	0.7	0.2	0.6	0.4	0.0	0.0
FD-1-1	2.7	2.9	1.9	2.4	1.5	1.8	2.2	2.3	2.1	1.1
Polar	0.8	4.0	2.7	0.2	1.1	1.0	0.9	0.2	1.6	1.7

Study 2

In a second study (Shiotani, 1982), the residue profile from two Japanese soils (Odawara or Oiso) was investigated. In this study, soils treated with [^{14}C] triflumizole were incubated indoors at 15 °C or

25 °C for 1 hr, 7, 14, 21, 28, 42, 56, 70, 84, and 98 days after treatment. Extractable, bound, volatile residues and $^{14}CO_2$ were examined. In the case of extractable residues, methanol:water (8:2, v/v) extracts were partitioned against dichloromethane and cleaned-up on silica gel columns. Residue identification was by HPLC. In a separate part of the study, the Odawara and Oiso soils were treated and incubated outdoors during the day and inside at night and during rainy days. The soils were sampled at intervals of 1, 3, 7, 14, and 28 days after treatment and extracted in the same manner as those incubated indoors (polyurethane plugs and CO_2 traps were not used with the outdoor-incubated soils).

Residue profiles by soil, incubation temperature, and time are summarized in Table –32.

Table 27 Recovery of radioactivity and proportion of radioactive components in the CH_2Cl_2 fraction of the extracts for Odawara soil, incubated at 15 °C (expressed as % TAR)

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Polyurethane foam	Trapped $^{14}CO_2$	Total	Triflumizole	FM5-1	FM6-1	FD1-1	FA-1-1	Others
0	94.7	94.7	0	5.3	–	–	100.0	88.0	0.3	1.5	0.8	n.d.[a]	1.5
7	88.9	87.9	1.0	11.2	2.3	n.d.	102.4	70.8	1.2	3.6	3.2	2.4	4.8
14	82.0	80.8	1.2	14.5	4.1	n.d.	100.6	60.8	1.7	4.2	4.0	4.9	3.5
21	73.1	71.9	1.2	18.1	8.5	0.1	99.8	52.3	1.2	2.5	3.1	8.2	3.2
28	67.0	64.7	2.3	22.3	12.0	0.2	101.5	45.3	1.1	3.2	2.7	7.7	3.5
42	54.1	51.1	3.0	24.4	19.2	0.3	98.0	35.8	1.3	2.6	2.8	4.3	3.1
56	43.2	40.0	3.2	28.1	27.4	0.5	99.2	27.0	1.1	2.4	2.2	3.9	2.6
70	35.5	32.3	3.2	28.1	27.4	0.5	97.9	22.5	0.9	1.7	1.8	2.3	1.9
84	27.5	24.5	3.0	30.3	31.3	0.8	96.4	14.9	0.9	1.4	1.6	2.4	2.2
98	23.3	20.3	3.0	33.0	37.6	1.0	94.9	9.9	0.8	1.3	1.6	2.4	3.1

[a] Not detected

Table 28 Recovery of radioactivity and proportion of radioactive components in the CH_2Cl_2 fraction of the extracts for Odawara soil, incubated at 25 °C (expressed as % TAR)

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Polyurethane foam	Trapped $^{14}CO_2$	Total	Triflumizole	FM5-1	FM6-1	FD1-1	FA-1-1	Others
0	94.7	94.7	0	5.3	–	–	100.0	88.0	0.3	1.5	0.8	n.d.[a]	1.5
7	76.4	75.4	1.0	16.2	9.2	0.1	101.9	60.7	1.0	2.8	2.4	4.3	2.9
14	60.7	59.4	1.3	27.0	14.3	0.4	102.4	38.2	1.2	3.5	2.9	9.1	2.9
21	46.3	44.1	2.2	31.5	21.3	0.8	99.9	28.6	0.9	2.7	2.3	5.9	2.4
28	34.0	31.3	2.7	34.3	26.4	1.2	95.9	19.0	0.9	2.0	1.8	4.6	1.9
42	23.4	21.0	2.4	38.4	32.1	1.6	95.5	12.7	0.4	1.5	1.1	2.9	1.4
56	13.1	11.8	1.3	40.1	39.0	2.3	94.5	6.3	0.3	0.6	0.6	2.1	1.2
70	8.8	7.5	1.3	42.2	40.6	2.7	94.3	n.a.[b]	n.a.	n.a.	n.a.	n.a.	n.a.
84	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
98	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

[a] Not detected

[b] Not analysed

Table 29 Recovery of radioactivity and proportion of radioactive components in the CH_2Cl_2 fraction of the extracts for Oiso soil, incubated at 15 °C (expressed as % TAR)

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Polyurethane foam	Trapped $^{14}CO_2$	Total	Triflumizole	FM5-1	FM6-1	FD 1-1	FA-1-1	Others
0	91.2	91.2	0	8.8	–	–	100.0	80.1	0.2	2.9	1.5	n.d.[a]	2.1
7	87.3	86.9	0.4	13.0	1.2	n.d.	101.5	58.9	1.2	3.0	3.6	14.1	3.1
14	81.6	81.1	0.5	18.2	2.0	0.1	101.9	41.6	2.1	4.8	4.4	20.6	3.7

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Polyurethane foam	Trapped $^{14}CO_2$	Total	Triflumizole	FM5-1	FM6-1	FD 1-1	FA-1-1	Others
21	75.3	74.8	0.5	22.1	3.5	0.2	101.1	38.5	1.7	4.3	4.9	19.8	2.9
28	69.1	68.6	0.5	25.3	7.1	0.4	101.9	34.8	2.3	3.8	5.7	16.7	2.7
42	58.3	56.9	1.4	30.7	10.3	0.7	100.0	28.0	2.1	3.5	5.2	12.6	3.1
56	48.1	46.4	1.7	35.5	12.1	1.3	97.0	22.3	1.7	2.9	4.2	10.5	2.8
70	40.7	38.7	2.0	37.6	13.3	1.8	93.4	18.9	1.5	2.2	3.5	8.0	2.5
84	34.4	32.4	2.0	37.8	14.4	2.3	88.9	14.2	1.4	2.1	3.5	7.0	2.5
98	26.9	24.7	2.2	39.0	17.5	2.5	85.9	9.8	1.2	2.0	3.0	5.3	2.1

[a] Not detected

Table 30 Recovery of radioactivity and proportion of radioactive components in the CH_2Cl_2 fraction of the extracts for Oiso soil, incubated at 25 °C (expressed as % TAR)

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Polyurethane foam	Trapped $^{14}CO_2$	Total	Triflumizole	FM5-1	FM6-1	FD1-1	FA-1-1	Others
0	91.2	91.2	0	8.8	–	–	100.0	80.1	0.2	2.9	1.5	n.d.[a]	2.1
7	77.6	77.0	0.6	20.1	2.1	0.2	100.0	43.2	2.1	6.0	4.8	17.2	2.5
14	69.9	69.2	0.7	26.3	4.0	0.7	100.9	29.2	1.8	5.3	4.1	23.4	2.8
21	57.5	56.8	0.7	30.0	9.2	1.2	97.9	22.1	1.6	3.9	3.8	20.5	2.9
28	48.5	47.8	0.7	34.6	11.9	2.3	97.3	18.1	1.4	2.7	3.2	17.4	2.7
42	34.0	32.4	1.6	45.7	14.2	3.6	97.5	8.3	1.0	1.8	2.6	15.0	2.0
56	24.6	23.2	1.4	46.5	20.3	5.1	96.5	8.0	0.8	1.0	2.1	8.5	1.9
70	16.3	14.9	1.4	48.6	28.0	5.7	98.6	n.a.[b]	n.a.	n.a.	n.a.	n.a.	n.a.
84	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
98	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

[a] Not detected

[b] Not analysed

Table 31 Recovery of radioactivity and proportions of radioactive components in the CH_2Cl_2 fraction of the extracts for Odawara soil, from the outdoor experiment (expressed as % a.r.)

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Total	Triflumizole	FM5-1	FM6-1	FD1-1	FA-1-1	Others
1	66.6	63.3	3.3	17.8	84.4	50.4	2.3	4.3	4.1	0.6	2.4
3	53.8	50.8	3.0	21.9	75.7	34.5	3.2	5.1	2.5	1.8	4.3
7	36.8	35.9	0.9	23.0	59.8	19.9	3.5	5.9	1.3	2.6	2.4
14	29.2	27.8	1.4	23.4	52.6	14.3	3.5	5.9	1.0	2.0	0.5
28	20.1	18.4	1.7	23.6	43.7	12.7	0.5	3.5	0.8	0.8	0.7

Table 32 Recovery of radioactivity and proportions of radioactive components in the CH_2Cl_2 fraction of the extracts for Oiso soil, from the outdoor experiment (expressed as % a.r.)

Time (d)	Me-OH-H_2O extract	CH_2Cl_2 fraction	H_2O fraction	Soil residue	Total	Triflumizole	FM5-1	FM6-1	FD1-1	FA-1-1	Others
1	73.0	71.4	1.6	16.3	89.3	59.6	2.7	1.3	5.3	0.9	3.3
3	64.2	62.2	2.0	21.0	85.2	46.1	2.8	2.0	4.6	2.1	2.7
7	51.3	50.2	1.1	21.0	72.3	36.6	3.3	3.1	3.2	3.7	2.2
14	47.2	44.7	2.5	24.0	71.2	30.4	4.7	4.0	2.6	3.9	0.7
28	41.4	40.5	0.9	24.4	65.8	28.7	2.5	3.9	2.0	2.5	0.8

Table 33 4DT_{50} values and DT_{90} values for [^{14}C] triflumizole and FA-1-1 applied in two soils, incubated indoor at 15 and 25 °C respectively

	Odawara				Oiso			
Soil	15°C		25°C		15°C		25°C	
Substance →	Triflumizole	FA-1-1	Triflumizole	FA-1-1	Triflumizole	FA-1-1	Triflumizole	FA-1-1
DT_{50} (days)	33 (22)[a]	9.3 (6.2)	13 (19)	7.8 (12)	27 (18)	12 (8)	11 (16)	13 (19)
DT_{90} (days)	110 (74)	31 (21)	43 (64)	26 (39)	91 (61)	40 (27)	37 (55)	26 (39)
r^2	0.996		0.996		0.95		0.97	

[a] Recalculation to 20 °C are given in parentheses

Table 34 DT_{50} values and DT_{90} values for [^{14}C] triflumizole applied in two outdoor soils

Soil	Odawara	Oiso
DT_{50} (days)	7.1	22
DT_{90} (days)	23	74
r^2	0.85	0.75

Dissipation times for 50% and 90% loss for parent triflumizole and the FA-1-1 metabolite are summarized in Table 33 4 and Table and the proposed metabolic pathway for triflumizole in soil is shown in

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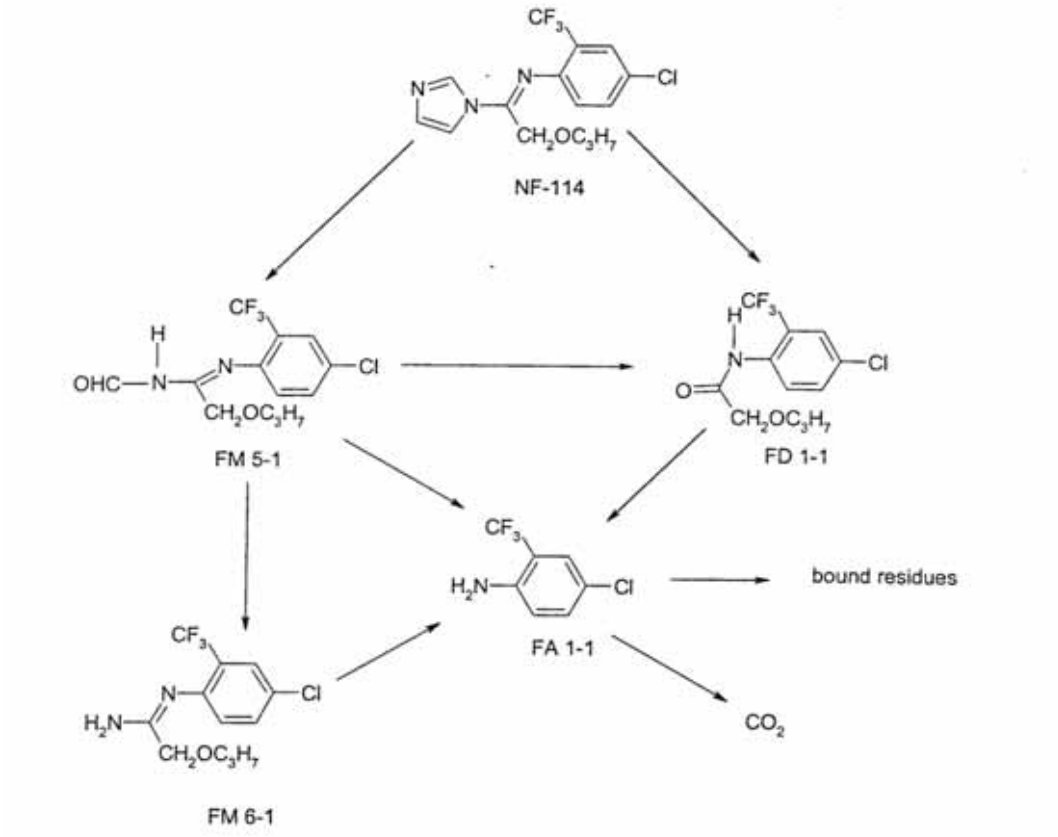


Figure 4 Proposed metabolic pathway of triflumizole in soil

Residues in rotational crops

Confined rotational crop studies

The metabolic profile of triflumizole in rotational lettuce, radish (roots and tops), and wheat (forage, hay, straw, and grain) was investigated (Yu *et al.,* 2001). The study consisted of two untreated control plots, six plots treated at 1× GAP, and two plots treated at 10× GAP with [^{14}C] triflumizole. The rotational crops were planted into the 1× treated plots at plant-back intervals (PBIs) of 30, 120, and 365 days after treatment. The 10× plots had a single PBI of 30 days.

For each PBI, samples were harvested and homogenized in the presence of dry ice. In the case of radish samples, the roots were washed to remove soil particles prior to homogenization. Homogenized samples were extracted with methanol:water and assayed by LSC to determine extractable residues. Post-extraction solids were dried for several days and then assayed by combustion/LSC to determine non-extractable radioactivity. Metabolite characterization and identification was done using the 10× matrices (30-day PBI) except for wheat forage, for which the 1× 30-day PBI samples were used. Metabolite characterization and identification was accomplished via HPLC (multiple systems) with known standards. Structural information for unknown metabolites was evaluated using mass spectrometry techniques including ESI/LC/MS, ESI/MS/CID/MS (CID chemical ionization positive ion/negative ion modes), and TOF/LC/MS. Enzymatic hydrolysis with ß-glucosidase was used to elucidate structures where MS analysis indicated the presence of glucoside conjugates (lettuce and wheat hay samples). Finally, PES samples were subjected to fractionation and derivatization procedures to determine the distribution of non-extractable residues in natural components such as starch, cellulose, and lignin.

Table 35 Total radiocarbon residues in rotational crop matrices

	Total Radiocarbon Residues as Triflumizole-Equivalents (mg eq/kg)		
Crop	30 DAT	120 DAT	365 DAT
Lettuce	0.0824	0.0861	0.0206
Radish Root	0.2336	0.1882	0.0320
Radish Top	0.1944	0.1449	0.0960
Wheat Forage	0.4420	0.3729	0.1263
Wheat Hay	0.9958	0.5834	0.3208
Wheat Straw	1.6495	0.6095	0.4784
Wheat Grain	0.1051	0.0554	0.0672

Forty-nine metabolites were identified, five of which were > 10% TRR and > 0.01 mg eq/kg in at least one matrix (Table). Five of the remaining forty-four metabolites were identified as having a hydroxide (or O-conjugate) appended at the Number 6 position of the aniline ring.

Table 36 Major metabolites identified in the confined rotational crop study at a 30-day plant-back interval. See Figure 5 for chemical structures.

Metabolite Identification	Molecular Weight	Crop Commodity	% TRR	Triflumizole Equivalents (mg eq/kg)
XIV (2Z)-4-hydroxyhex-2-enedioic acid	160	Radish Tops	12.54	0.2464
		Wheat Grain	61.35	0.3999
		Wheat Hay	14.87	0.8757
		Wheat Straw	11.06	1.1877
FD-9-1 [4-chloro-2-(trifluoromethyl) phenyl]urea	238	Lettuce	34.87	0.425
		Radish Tops	30.62	0.6016
		Wheat Straw	11.7	1.2564
XXVI 1-[4-chloro-2-hydroxy-6-	270	Radish Roots	11.36	0.1087

Metabolite Identification	Molecular Weight	Crop Commodity	% TRR	Triflumizole Equivalents (mg eq/kg)
trifluoromethyl)phenyl]-3-hydroxyurea				
XXVIII 3-(2-aminoprop-2-enoyl)-1-[4-chloro-2-(trifluoromethyl)phenyl]urea	307	Wheat Grain	13.27	0.0865
XXI N'-[4-chloro-2-(trifluoromethyl) phenyl]-2-[(3,4,5,6-tetrahydroxyoxan-2-yl)methoxy]ethanimidamide	414	Lettuce	22.89	0.279

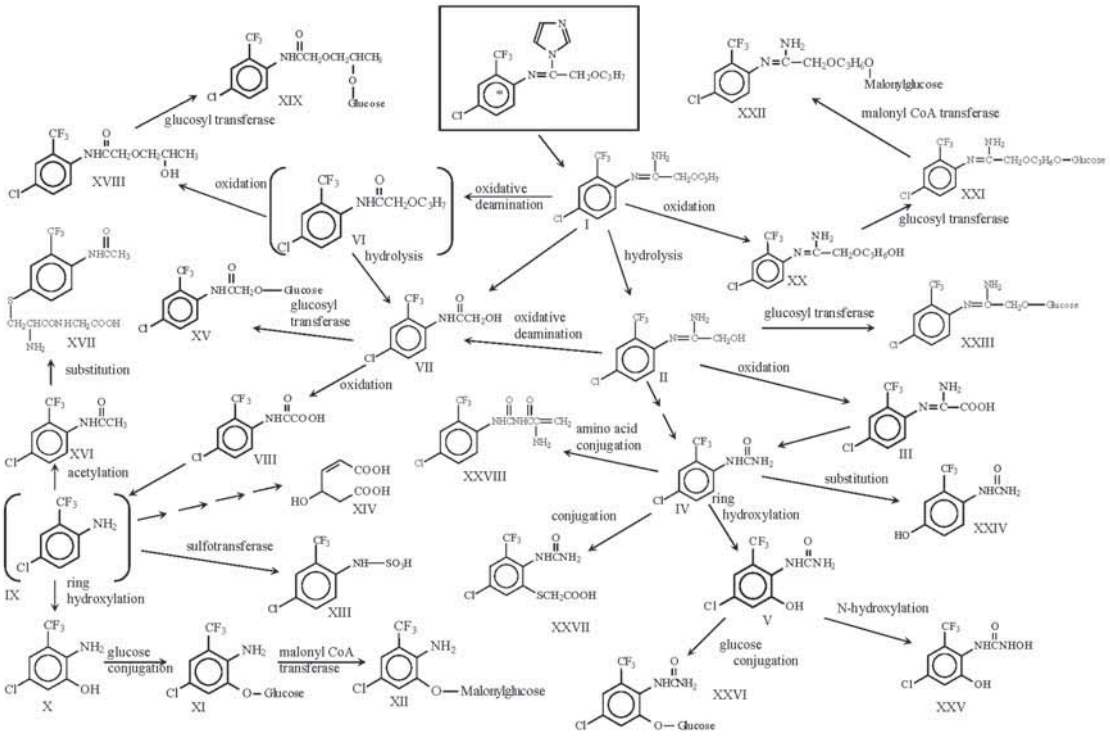


Figure 5 Proposed metabolic pathways in rotational crop—wheat. Pathway is representative of other rotational crops.

Field rotational crop studies

Two studies were submitted depicting residues of triflumizole in field rotational crops. In the first study (Gaydosh, 2000b), triflumizole (as Procure® 50 WS) was applied to cucumbers at the vining stage followed by four subsequent applications at 7 ± 1 day intervals. Trials were in Watsonville, California and Uvalde, Texas. Applications were each 0.28 kg ai/ha for a total rate of 1.4 kg ai/ha. Cucumbers were harvested immediately after the final triflumizole application and rotational crops of lettuce, turnips and wheat were planted approximately 30, 60, 90, and 180 days after harvesting the cucumber crop (except for 90-day wheat in California which was replanted at 103 days due to crop damage). Samples were analysed by a common-moiety method that converts triflumizole and its aniline-containing metabolites to FA-1-1, as described in the residue methods section below. Results are expressed as triflumizole-equivalents. The limit of quantitation (LOQ) was 0.01 mg eq/kg with a corresponding limit of detection (LOD) of 0.003 mg eq/kg.

Across the two site locations, residues of triflumizole in treated cucumbers ranged from 0.12 to 0.20 mg eq/kg. Residues in rotational crops are summarized in Table ; residues in samples from untreated plots were < 0.01 mg eq/kg for all matrices.

Table 37 Triflumizole-equivalent residues in rotational lettuce, turnip, and wheat

Crop Matrix	California			Texas		
	PBI (days)	Sample 1	Sample 2	PBI (days)	Sample 1	Sample 2
Lettuce	30	< 0.01	< 0.01	30	< 0.01	< 0.01
	59	< 0.01	< 0.01	60	< 0.01	< 0.01
	94	0.013	0.012	91	< 0.01	< 0.01
	120	< 0.01	< 0.01	177	< 0.01	< 0.01
Turnip Tops	30	0.02	0.02	30	< 0.01	< 0.01
	59	< 0.01	< 0.01	60	< 0.01	< 0.01
	94	< 0.01	< 0.01	91	< 0.01	< 0.01
	120	< 0.01	< 0.01	177	< 0.01	< 0.01
Turnip Roots	30	< 0.01	< 0.01	30	< 0.01	< 0.01
	59	< 0.01	< 0.01	60	< 0.01	< 0.01
	94	< 0.01	0.01	91	< 0.01	< 0.01
	120	< 0.01	< 0.01	177	< 0.01	< 0.01
Wheat Forage	30	0.02	0.03	30	0.01	0.01
	59	0.05	0.06	60	0.01	< 0.01
	103	0.03	0.03	91	< 0.01	< 0.01
	120	0.04	0.04	177	< 0.01	< 0.01
Wheat Hay	30	0.02	0.02	30	< 0.01	< 0.01
	59	0.02	0.02	60	< 0.01	< 0.01
	103	0.01	0.01	91	< 0.01	< 0.01
	120	0.03	0.03	177	< 0.01	< 0.01
Wheat Grain	30	< 0.01	< 0.01	30	< 0.01	< 0.01
	59	< 0.01	< 0.01	60	< 0.01	< 0.01
	103	< 0.01	< 0.01	91	< 0.01	< 0.01
	120	< 0.01	< 0.01	177	< 0.01	< 0.01
Wheat Straw	30	0.02	0.02	30	< 0.01	0.011
	59	0.02	0.02	60	< 0.01	0.011
	103	0.02	0.02	91	< 0.01	0.011
	120	0.02	0.04	177	< 0.01	< 0.01

In the second study (Puhl, 2005), triflumizole (as Procure® 50 WS) was applied to squash at approximately 30 days prior to harvest followed by four subsequent applications at 7 ± 1 day intervals. Trials were in Yuma, Arizona and Uvalde, Texas. Applications were each 0.28 kg ai/ha for a total rate of 1.4 kg ai/ha. At approximately 30, 60, 90, 180, and 270 days after the last application, cabbage, cotton, onion, tomato, and wheat were planted into treated and untreated plots. Samples were analysed by a common-moiety method that converts triflumizole and its aniline-containing metabolites to FA-1-1, as described in the residue methods section below. Results are expressed as triflumizole-equivalents. The limit of quantitation (LOQ) was 0.01 mg eq/kg. Average procedural recoveries of triflumizole and FA-1-1 ranged from 79 to 95%, with a maximum standard deviation of 13%.

In the rotational crops, quantifiable residues were found only in wheat forage, wheat straw, wheat hay, and onion; residues in wheat grain, cabbage, tomato, and cottonseed were < 0.01 mg eq/kg in all samples (Table).

Table 38 Triflumizole-equivalent residues in rotational cabbage, cotton, onion, tomato, and wheat

Crop Matrix	PBI (days)	Arizona		Texas	
		Sample 1	Sample 2	Sample 1	Sample 2
Cabbage	30	< 0.01	< 0.01	< 0.01	< 0.01
	60	< 0.01	< 0.01	< 0.01	< 0.01
	90	< 0.01	< 0.01	< 0.01	< 0.01
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	< 0.01	< 0.01	< 0.01	< 0.01
Cottonseed	30	< 0.01	< 0.01	< 0.01	< 0.01
	60	< 0.01	< 0.01	< 0.01	< 0.01
	90	< 0.01	< 0.01	< 0.01	< 0.01
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	< 0.01	< 0.01	< 0.01	< 0.01
Onion	30	0.01	0.01	< 0.01	< 0.01
	60	< 0.01	< 0.01	< 0.01	< 0.01
	90	< 0.01	< 0.01	< 0.01	< 0.01
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	< 0.01	< 0.01	< 0.01	< 0.01
Tomato	30	< 0.01	< 0.01	< 0.01	< 0.01
	60	< 0.01	< 0.01	< 0.01	< 0.01
	90	< 0.01	< 0.01	< 0.01	< 0.01
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	< 0.01	< 0.01	< 0.01	< 0.01
Wheat Forage	30	0.02	< 0.01	< 0.01	0.013
	60	0.14	0.20	0.01	< 0.01
	90	0.19	0.12	0.01	0.011
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	0.077	0.089	< 0.01	< 0.01
Wheat Grain	30	< 0.01	< 0.01	< 0.01	< 0.01
	60	< 0.01	< 0.01	< 0.01	< 0.01
	90	< 0.01	< 0.01	< 0.01	< 0.01
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	< 0.01	< 0.01	< 0.01	< 0.01
Wheat Hay	30	0.14	0.08	0.02	0.01
	60	0.11	0.07	0.02	0.01
	90	0.09	0.07	0.02	0.02
	180	< 0.01	< 0.01	< 0.01	< 0.01
	270	0.04	0.04	< 0.01	< 0.01
Wheat Straw	30	0.05	0.09	< 0.01	< 0.01
	60	0.08	0.09	< 0.01	< 0.01
	90	0.14	0.09	< 0.01	0.134
	180	< 0.01	0.01	< 0.01	< 0.01
	270	0.06	0.05	< 0.01	< 0.01

RESIDUE ANALYSIS

Analytical methods

The Meeting received analytical methods and validation data for analysis of triflumizole in plant and animal commodities. These methods fall into two classes. The first are common-moiety methods which analysed for residues of triflumizole and its aniline-containing metabolites, FM-6-1, FM-5-1, FD-1-1, FD-2-1, FM-8-1, and FA-l-1 by converting each of the metabolites to FA-1-1. The second

class are residue specific methods which were developed to analyse, separately, triflumizole and FM-6-1 in crops or FA-1-5 (free and sulphate-conjugated) in animal matrices. For both classes, alterations have been made as necessary to resolve analytical issues and to permit use of different separation/detection strategies. Limits of quantitation and detection are reported as being 0.01 to 0.02 mg eq/kg, with no consistent distinction between the two. Descriptions are provided below.

Common-moiety methods

The analytical method described by Ball (1986a) is the basis for the common moiety methods for crop commodities. In this method, extraction of residues begins by macerating the sample in distilled water for 5 minutes. Concentrated glacial acetic acid and sodium acetate are added to the homogenate and the mixture is then refluxed at boiling for 90 minutes. A sodium hydroxide solution (20%) is added to the flask, which is then fitted with a distillation head and heated until a volume of 100 mL is distilled into hexane. The distillate is shaken against 0.1 N HCl. The organic phase is collected, concentrated, and an aliquot taken for analysis. Separation/quantification strategies that have been used with this extraction method include GC-MSD, GC-FTD, GC-NPD, HPLC-UV (254 nm), and HPLC-Amperometric detector. Residues are analysed as FA-1-1 and expressed in terms of triflumizole.

A common-moiety method was also developed for animal commodities (Perkins and Polakoff, 1986). This method uses 20% NaOH and a Bleidner extractor to digest/distil/extract residues into hexane. The hexane is passed over anhydrous sodium sulphate and concentrated prior to analysis. As with the common-moiety method for crops, the terminal residue is the FA-1-1 metabolite. Parameters are described for GC-NPD analysis.

Residue-specific methods

A method for the analysis of triflumizole and FM-6-1 is described by Gomyo *et al.* (1990). In this method, residues are extracted from crop matrices with methanol by shaking for 30 min. The extract is then filtered and partitioned against dichloromethane. Residues in the organic phase are cleaned up by column chromatography, concentrated to dryness, and then reconstituted in dichloromethane for HPLC determination or acetone for GC determination.

Corley (2012) describes a method for the analysis of triflumizole and FM-6-1 in hops by HPLC-MS/MS. In that study, residues were extracted with acetonitrile, the extract cleaned up by polymeric and NH_2 solid-phase extraction, and the extract analysed by HPLC-MS/MS. Transitions for triflumizole were m/z 346.1 $\Rightarrow$73 (quant.) and 346.1 $\Rightarrow$ 278 (conf.). Transitions for FM-6-1 were m/z 295.1 $\Rightarrow$ 215 (quant.) and 295.1 $\Rightarrow$ 73 (conf.).

Analysis of FA-1-5 (free and sulphate-conjugate) in animal matrices is described in Perkins and Polakoff (1986). In this method, residues are extracted from milk and liver with 0.2 N HCl (boiling, 1 hr). Following extraction, the residues are cleaned up by solid-phase extraction (C-18) and analysed by HPLC with amperometric detection.

Table 39 Summary of method validation recovery results for triflumizole and metabolites in crop and livestock matrices

Matrix	Method Type	Separation/ Quantification	Residue	Fortification (mg./kg)	Recovery (%)	Reference
Apple	Common	HPLC-UV	Triflumizole	0.05	75.4, 75.6	Ball, 1986a
				0.1	85, 74.4, 81.5	
				1	75.8, 78.3	
			FM-5-1	0.1	76	
			FM-6-1	0.1	97	
			FM-8-1	0.1	103	
			FD-1-1	0.1	94	
			FD-2-1	0.1	91	
			FA-1-1	0.1	79	
Apple	Common	HPLC-UV	Triflumizole	0.05	86, 83, 80, 72, 71	Gomyo, Morishima, Ono, 1990

Matrix	Method Type	Separation/ Quantification	Residue	Fortification (mg./kg)	Recovery (%)	Reference
				0.2	77, 70, 69	
	Common	GC-FTD	Triflumizole	0.05	91, 81	
				0.2	94, 84, 80	
			FM-5-1	0.2	81	
			FM-6-1	0.2	78	
			FM-8-1	0.2	91	
			FD-1-1	0.2	84	
			FD-2-1	0.2	87	
			FA-1-1	0.2	77	
			FD-7-1	0.2	91	
			FM-2-1	0.2	73	
	Specific	HPLC-UV	Triflumizole	0.05	93, 90	
				0.2	92, 90	
			FM-6-1	0.05	90,86	
				0.2	86, 85	
	Specific	GC-FTD	Triflumizole	0.2	95, 85	
			FM-6-1	0.2	92, 86	
Cucumber	Specific	GC-FTD	Triflumizole	0.2	90, 84	
			FM-6-1	0.2	90, 88	
Cucumber	Common	HPLC-UV	Triflumizole	0.05	91, 91, 86, 81, 80	
				0.2	97, 97	
			FM-5-1	0.05	74	
			FM-6-1	0.05	76	
			FM-8-1	0.05	85	
			FD-1-1	0.05	79	
			FD-2-1	0.05	85	
			FA-1-1	0.05	75	
	Specific	HPLC-UV	Triflumizole	0.05	80, 78	
				0.2	83, 80	
			FM-6-1	0.05	78,78	
				0.2	95, 85	
Grape	Common	HPLC-UV	Triflumizole	0.05	100, 70	Ball, 1986a
				0.1	74, 74, 108, 69.8	
				0.5	87.4, 77.4	
			FM-5-1	0.05	82, 82	
				0.1	71, 61	
			FM-6-1	0.05	64, 94	
				0.1	82, 91	
			FM-8-1	0.05	104, 142	
				0.1	92, 98	
			FD-1-1	0.05	70, 94	
				0.1	57, 65	
			FD-2-1	0.05	86, 144	
				0.1	87, 78	
			FA-1-1	0.05	94, 126	
				0.1	86, 79	
Grape	Specific	HPLC-UV	Triflumizole	0.2	91, 81	Gomyo, Morishima, Ono, 1990
			FM-6-1	0.5	87, 87	
		GC-FTD	Triflumizole	0.2	87	
			FM-6-1	0.5	80	
	Specific	HPLC-UV	Triflumizole	0.2	91,87	
			FM-6-1	0.2	87,80	
		GC-FTD	Triflumizole	0.2	82, 75	
			FM-6-1	0.2	90, 87	
Green Pepper	Specific	HPLC-UV	Triflumizole	0.2	84, 73	
			FM-6-1	0.2	84, 81	
Hops	Specific	LC-MS/MS	Triflumizole	0.05	98, 98, 97	Corley, 2012
				1	99, 94, 91	

Matrix	Method Type	Separation/ Quantification	Residue	Fortification (mg./kg)	Recovery (%)	Reference
				50	102, 103, 101	
			FM-6-1	0.05	97, 105, 98	
				1	106, 97, 94	
				50	102, 97, 99	
Lettuce	Common	GC-MSD	Triflumizole	0.01	75, 78, 82, 72, 75, 80	Gaydosh, 2000a
				0.5	71, 70, 75, 70, 71, 70	
			FM-5-1	0.01	92, 89, 91	
				0.5	73, 73, 76	
			FM-6-1	0.01	84, 80, 87	
				0.5	87, 82, 88	
			FM-8-1	0.01	94, 93, 97	
				0.5	77, 82, 83	
			FD-1-1	0.01	99, 98, 105	
				0.5	84, 83, 85	
			FD-2-1	0.01	84, 80, 85	
				0.5	86, 86, 94	
			FA-1-1	0.01	92, 86, 91, 82, 89, 88	
				0.5	78, 77, 79, 78, 79, 75	
			FD-9-1	0.01	73, 77, 73	
				0.5	74, 74, 71	
Pear	Common	HPLC-UV	Triflumizole	0.1	60	Ball, 1986a
			FM-5-1	0.1	58	
			FM-6-1	0.1	65	
			FM-8-1	0.1	117	
			FD-1-1	0.1	72	
			FD-2-1	0.1	91	
			FA-1-1	0.1	86	
Pear	Common	HPLC-UV	Triflumizole	0.2	81, 73	Gomyo, Morishima, Ono, 1990
			FM-6-1		80, 76	
	Common	GC-FTD	Triflumizole	0.05	90, 87	
				0.2	89, 86	
			FM-6-1	0.2	82	
			FM-8-1	0.2	89	
Peas	Specific	HPLC-UV	Triflumizole	0.2	87,84	
			FM-6-1	0.2	101,98	
Strawberry	Common	HPLC-UV	Triflumizole	0.2	86, 83	
	Specific	HPLC-UV	Triflumizole	0.2	91,87	
			FM-6-1	0.2	81,77	
		GC-FTD	Triflumizole	0.2	79, 77	
			FM-6-1	0.2	74, 72	
Tomato	Common	HPLC-UV	Triflumizole	0.1	83, 80	
	Specific	HPLC-UV	Triflumizole	0.2	81,81	
			FM-6-1		88, 87	
Turnip Root	Common	GC-MSD	Triflumizole	0.01	73, 73, 76, 84, 85, 90	Gaydosh, 2000a
				0.5	74, 70, 82, 78, 74, 78	
			FM-5-1	0.01	86, 90, 95	
				0.5	85, 81, 82	
			FM-6-1	0.01	89, 79, 80	
				0.5	83, 81, 82	
			FM-8-1	0.01	83, 80, 91	
				0.5	78, 78, 80	
			FD-1-1	0.01	91, 88, 92	
				0.5	90, 86, 83	
			FD-2-1	0.01	102, 95, 99	
				0.5	90, 80, 84	
			FA-1-1	0.01	86, 79, 91, 95, 87, 97	
				0.5	79, 77, 78, 78, 78, 85	

Matrix	Method Type	Separation/ Quantification	Residue	Fortification (mg./kg)	Recovery (%)	Reference
			FD-9-1	0.01	79, 80, 79	
				0.5	75, 74, 74	
Watermelon	Common	HPLC-UV	Triflumizole	0.2	75, 75	Gomyo, Morishima, Ono, 1990
Wheat Forage	Common	GC-MSD	Triflumizole	0.01	100, 96, 98, 78, 63, 78	Gaydosh, 2000a
				0.5	81, 84, 82, 84, 77, 75	
			FM-5-1	0.01	90, 83, 90	
				0.5	73, 71, 72	
			FM-6-1	0.01	78, 85, 88	
				0.5	78, 75, 75	
			FM-8-1	0.01	82, 81, 87	
				0.5	73, 72, 76	
			FD-1-1	0.01	81, 88, 67	
				0.5	77, 77, 79	
			FD-2-1	0.01	77, 76, 93	
				0.5	80, 80, 83	
			FA-1-1	0.01	76, 65, 63, 81, 77, 75	
				0.5	72, 72, 71, 73, 76, 77	
			FD-9-1	0.01	74, 79, 86	
				0.5	74, 67, 70	
Wheat Grain	Common	GC-MSD	Triflumizole	0.01	91, 89, 82, 91, 79	
				0.5	80, 76, 74, 70, 81	
			FM-5-1	0.01	89, 85, 94	
				0.5	71, 74, 80	
			FM-6-1	0.01	84, 81, 91	
				0.5	75, 78, 76	
			FM-8-1	0.01	87, 94, 94	
				0.5	75, 75, 76	
			FD-1-1	0.01	90, 96, 91	
				0.5	76, 74, 78	
			FD-2-1	0.01	104, 80, 106	
				0.5	87, 88, 90	
			FA-1-1	0.01	78, 78, 75, 79, 76, 70	
				0.5	78, 79, 80, 76, 79, 74	
			FD-9-1	0.01	69, 64, 85	
				0.5	68, 72, 70	
Wheat Straw	Common	GC-MSD	Triflumizole	0.01	80, 72, 71, 78, 80, 69	
				0.5	74, 76, 74, 68, 75, 69	
			FM-5-1	0.01	94, 87, 88	
				0.5	78, 77, 78	
			FM-6-1	0.01	80, 68, 72	
				0.5	73, 73, 74	
			FM-8-1	0.01	83, 88, 80	
				0.5	78, 78, 78	
			FD-1-1	0.01	87, 81, 82	
				0.5	80, 77	
			FD-2-1	0.01	82, 77, 77	
				0.5	80, 81, 81	
			FA-1-1	0.01	72, 68, 59, 71, 60, 62, 65, 62, 61	
				0.5	68, 74, 71, 75, 72, 75, 64, 68, 71	
			FD-9-1	0.01	74, 68, 81	
				0.5	69, 70, 67	
Egg	Common	HPLC-Amperometric	Triflumizole	100	84 (avg.)	Parkins & Polakoff, 1986
			FM-6-1	100	84 (avg.)	
			FD-1-1	100	33 (avg.)	

Matrix	Method Type	Separation/ Quantification	Residue	Fortification (mg./kg)	Recovery (%)	Reference
			FA-1-1	25	86 (avg.)	
				50	82 (avg.)	
Fat	Common	HPLC-Amperometric	FA-1-1	25	76 (avg.)	
				50	80 (avg.)	
				20		
				25	104 (avg.)	
				50	90 (avg.)	
Kidney	Common	HPLC-Amperometric	FA-1-1	25	87 (avg.)	
				50	83 (avg.)	
				20	85 (avg.)	
				25	88 (avg.)	
				50	82 (avg.)	
Liver	Common	HPLC-Amperometric	FA-1-1	25	80 (avg.)	
				50	90 (avg.)	
				100	86 (avg.)	
			Triflumizole	100	90 (avg.)	
			FM-8-1	100	88 (avg.)	
			FM-8-1-S	100	90 (avg.)	
			FA-1-1	20	82 (avg.)	
				80	85 (avg.)	
				50	92 (avg.)	
				100	75 (avg.)	
			FA-1-5	18	69 (avg.)	
	Specific	HPLC-Amperometric	FA-1-5-S	0.01	89.1, 76.6, 74.7, 66.2, 85.3, 74.4, 82.5, 116.3, 77.3, 68.1, 75.1, 105.4	Batorewicz & Bakker, 1990
				0.1	108.6, 122.9, 104.7, 103.5, 87.9, 98.1, 95.0, 58.7, 100.2, 69.0, 81.8, 96.3	
				0.05	70.2, 63.3, 52.2	Noon, 1993
				0.2	71.6, 64.7, 40.0	
Milk	Common	GC-NPD	Triflumizole	0.2 (2 hr reflux)	61, 88	Eckert, 1989
				0.2 (4 hr reflux)	50, 80	
				0.2 (6 hr reflux)	40, 80	
	Common	HPLC-Amperometric	FA-1-1	10	89 (avg.)	Parkins & Polakoff, 1986
				20	84 (avg.)	
			FA-1-5	50	109 (avg.)	
			FA-1-1	20	88 (avg.)	
				50	92 (avg.)	
			FA-1-5	49	80 (avg.)	
			Triflumizole	100	92 (avg.)	
			FD-2-1	100	87 (avg.)	
			FM-8-1	100	78 (avg.)	
	Specific	HPLC-Amperometric	FA-1-5-S	0.01	59.1, 110.0, 64.7, 68.8, 68.5, 76.0, 63.7, 77.9, 90.2, 68.7, 77.9, 71.7	Batorewicz & Bakker, 1990
				0.1	127.2, 101.2, 111.6, 109.9, 87.0, 98.6, 109.6, 99.6, 91.6, 67.2, 96.8, 95.1	
				0.025	75.4, 81.9, 79.6	Noon, 1993
				0.1	78.3, 80.2, 81.4	
Muscle	Common	GC-NPD	Triflumizole	0.2 (2 hr reflux)	86, 79	Eckert, 1989

Matrix	Method Type	Separation/ Quantification	Residue	Fortification (mg./kg)	Recovery (%)	Reference
				0.2 (4 hr reflux)	99	
				0.2 (6 hr reflux)	102	
				0.2 (4 hr reflux)	104, 87, 87, 70, 94, 80	
	Common	HPLC-Amperometric	FA-1-1	25	100 (avg.)	Parkins & Polakoff, 1986
				50	72 (avg.)	
				20	94 (avg.)	
				25	96 (avg.)	
				50	94 (avg.)	

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of residues of triflumizole and its metabolites in crops (apples, grapes, strawberries, cucumbers, cherries, muskmelons, squash, lettuce, and wheat forage) and livestock. In many cases, the storage stability data were submitted as an integral part of the field trial study rather than as a separate storage stability study volume. The storage stability data are summarized in Table . Compounds, as listed in Table 40 were added to control samples and placed into frozen storage for the time indicated. For all matrices except cherries and strawberries, the fortification was to homogenized sample matrix. The state of the cherry and strawberry storage stability matrices was not described in the study report. Samples were stored frozen (-20 °C).

Table 40 Summary of storage stability data for triflumizole and metabolites in crop and livestock matrices

Matrix	Method	Compound	Storage Time	Amount Fortified (mg eq/kg)	Concurrent Recovery (%)	Residue Remaining (%)	Reference
Apple	Common—HPLC	Triflumizole	0 Day	2	n.s.	81.5	Ball, 1988
			38 Months		n.s.	59	
		FM-6-1	0 Day		n.s.	76.5	
			38 Months		n.s.	52	
	Specific—GC	Triflumizole	0 Day	0.5	82	85	Ball, 1989
			1 Month		78	78.5	
			2 Months		86	79.5	
			3 Months		91	78	
			6 Months		98	77	
			12 Months		91	60	
		FM-6-1	0 Day		81	82	
			1 Month		–	–	
			2 Months		94	82	
			3 Months		94	81.5	
			6 Months		108	99.5	
			12 Months		112	71	
		FD-2-1	0 Day		77	86	
			1 Month		–	–	
			2 Months		84	85.5	
			3 Months		95	84.5	
			6 Months		82	87	
			12 Months		112	73	
		FA-1-1	0 Day		77	80.5	
			1 Month		–	–	
			2 Months		86	77.5	
			3 Months		89	82.5	
			6 Months		74	64.5	
			12 Months		106	60.5	
Cherries	Common—GC	Triflumizole	0 Day	0.2	75.5	75.5	Gaydosh, 1997
			30 Days		69.5	75.7	

Matrix	Method	Compound	Storage Time	Amount Fortified (mg eq/kg)	Concurrent Recovery (%)	Residue Remaining (%)	Reference
			60 Days		73	62	
			120 Days		84.5	85.7	
			354 Days		86	88	
Cucumber	Common—GC	Triflumizole	832 Days	0.2	74	51	Homa, 2012
		FM-6-1			94	72	
		FA-1-1			79	30.7 [a]	
		Triflumizole	0 Day	0.5	84.9	85.4	Maselli, 1999
			1 Month		106.7	89.8	
			3 Months		107.3	96.4	
			6 Months		74	72.9	
			8 Months [b]		61.6	61	
	Common—HPLC	Triflumizole	0 Day	2	n.s.	76	Ball, 1988
			38 Months		n.s.	52	
		FM-6-1	0 Day		n.s.	70.5	
			38 Months		n.s.	48	
Grape	Specific—GC	Triflumizole	0 Day	2	75	80.5	Ball, 1989
			1 Month		77	78	
			2 Months		88	88	
			3 Months		93	86	
			6 Months		86	78	
			12 Months		78	76.5	
		FM-6-1	0 Day	2	70	71	
			1 Month		–	–	
			2 Months		83	79	
			3 Months		82	81	
			6 Months		108	86.5	
			12 Months		78	70	
		FD-2-1	0 Day	2	85	81	
			1 Month		–	–	
			2 Months		88	86	
			3 Months		92	80	
			6 Months		90	79	
			12 Months		91	81	
		FA-1-1	0 Days	2	78	86	
			1 Month		–	–	
			2 Months		86	88	
			3 Months		90	84	
			6 Months		82	70.5	
			12 Months		80	83	
Lettuce	Common—GC	FD-1-1	0 Month	5	79	79	Marin, 2011
			2 Months		111	90	
			4 Months		70	68	
		FM-6-1	0 Month		83	83	
			2 Months		98	93	
			4 Months		84	63	
Cabbage	Common—GC	Triflumizole	0 Day	5	–	–	Barney, 2007d
			238 Days		90	40	
		FM-6-1	0	5	–	–	
			238		92	26	
		FA-1-1	0	5	–	–	
			240		92	58	
Mustard Greens	Common—GC	Triflumizole	0 Day	5	–	–	Barney, 2006
			287 Days		77	54	
		FM-6-1	0 Day	5	–	–	
			287 Days		85	46	
		FA-1-1	0 Day	5	–	–	
			288 Days		83	37	
Swiss Chard	Common—GC	Triflumizole	0 Day	5	–	–	Barney, 2007g

Matrix	Method	Compound	Storage Time	Amount Fortified (mg eq/kg)	Concurrent Recovery (%)	Residue Remaining (%)	Reference
			218 Days		73	71	
		FM-6-1	0 Day	5	–	–	
			225 Days		71	54	
Muskmelon	Common—GC	Triflumizole	0 Day	0.5	82.9	80.6	Gaydosh, Maselli, Puhl, 1999
			29 Days		117.2	115.1	
			97 Days		75.5	82.1	
			181 Days		53.2	67.2	
			299 Days		53.7	56.2	
Tomato	Common—GC	Triflumizole	0 Days	0.5–10	–	–	Barney, 2012
			552 Days		85	68	
		FM-6-1	0 Days	0.5–10	–	–	
			552 Days		85	80	
		FA-1-1	0 Days	0.5–10	–	–	
			552 Days		91	75	
Summer Squash	Common—GC	Triflumizole	0 Day	0.5	91.8	112.1	Gaydosh, 1999
			32 Days		98.3	100.1	
			89 Days		93.9	100.1	
			179 Days		49.7	61.1	
			280 Days		45.5	50.9	
Strawberry	Common—GC	Triflumizole	0 Day	0.5	78	86	Gaydosh, 2001
			42 Days		92.5	83	
			184 Days		78.5	70	
			310 Days		85	63	
	Common—HPLC	Triflumizole	0 Day	2	n.s.	75	Ball, 1988
			38 Months		n.s.	56	
		FM-6-1	0 Day		n.s.	71.5	
			38 Months		n.s.	53	
Papaya	Common—GC	Triflumizole	0 Day	5	–	–	Barney, 2007f
			18 Months		78	74	
Hazelnut	Common—GC	Triflumizole	0 Day	0.5	–	–	
			97 Days		68	70	Thompson, 2002
		FA-1-1	0	0.5	–	–	
			97 Days		67	66	
Wheat Forage	Common—GC	FD-1-1	0 Month	5	93	93	Marin, 2011
			2 Months		117	80	
			4 Months		82	78	
		FM-6-1	0 Month		94	94	
			2 Months		93	92	
			4 Months		60	59	
Fat	Common—GC	FA-1-1	0 Days	0.1	88	88	Eckert, 1990
			14 Days		73	96	
			30 Days		96	98	
			90 Days		87	82	
			6 Months		86	88	
		Triflumizole	0 Days	0.1	80	80	
			14 Days		80	86	
			30 Days		88	101	
			90 Days		85	94	
			6 Months		90		
Kidney	Common—GC	FA-1-1	0 Days	0.1	81	81	
			14 Days		82	82	
			30 Days		87	103	
			90 Days		89	78	
			6 Months		88		

Matrix	Method	Compound	Storage Time	Amount Fortified (mg eq/kg)	Concurrent Recovery (%)	Residue Remaining (%)	Reference
		Triflumizole	0 Days	0.1	97	97	
			14 Days		94	104	
			30 Days		84	88	
			90 Days		94	76	
			6 Months		90	92	
Liver	Common—GC	FA-1-1	0 Days	0.1	97	97	
			14 Days		85	80	
			30 Days		86	78	
			90 Days		92	62	
			6 Months		92	90	
		Triflumizole	0 Days	0.1	102	102	
			14 Days		88	73	
			30 Days		86	74	
			90 Days		110	65	
			6 Months		88	97	
	Specific—HPLC	FA-1-5-S [b]	0 Day	0.1	68.6	86.6	Batorewicz, 1990, 1991
			102 Days		n.s.	89.2	
			15 Months		n.s.	113	
Milk	Common—GC	FA-1-1	0 Days	0.1	84	84	Eckert, 1990
			14 Days		99	101	
			30 Days		92	88	
			90 Days		85		
			6 Months		84	87	
		Triflumizole	0 Days	0.1	80	80	
			14 Days		83	84	
			30 Days		85	88	
			90 Days		84	89	
			6 Months		96	88	
	Specific—HPLC	FA-1-5-S [b]	0 Day	0.1	73.7	73.7	Batorewicz, 1990, 1991
			87 Days		n.s.	100	
			15 Months		n.s.	96.1	
Muscle	Common—GC	FA-1-1	0 Days	0.1	94	94	Eckert, 1990
			14 Days		102	102	
			30 Days		97	107	
			90 Days		98	88	
			6 Months		77	83	
		Triflumizole	0 Days	0.1	100	100	
			14 Days		89	97	
			30 Days		90	94	
			90 Days		96	80	
			6 Months		86	83	

n.s.=Not Specified.

[a] FA-1-1 was subject to evaporative loss in the hen metabolism study. That phenomenon may account for the low recovery from cucumber considering the long storage time involved.

[b] The 0 Day samples were quantitated using external standards. FA-1-5-S was shown to be not stable in the acid used to digest the samples, leading to inaccurate quantitation. Recovery at other time points in this study were quantitated based on procedural standards only (i.e., no correction for 0-day recovery).

USE PATTERN

Labels for triflumizole were provided for registrations in the USA and the Netherlands. Triflumizole is a broad-spectrum foliar fungicide that controls a variety of fungal diseases in fruits and vegetables. It acts as a protectant and as an eradicant by preventing disease symptoms after infection has occurred. Triflumizole's anti-sporulant activity reduces spores after lesions become visible. Triflumizole belongs to the demethylation inhibitor (DMI) group of fungicides classified as Group 3

by the Fungicide Resistance Action Committee (FRAC). Triflumizole displays protective and curative action, and acts as an inhibitor of chitin biosynthesis. The product is mixed with water and applied as a foliar spray using ground equipment equipped for conventional spraying on crops as listed in Table .

Table 41 Use pattern summary for triflumizole

Crop	Country	Application							Max/ season	PHI days	Remarks
		use	form [a]	no. [b]	max. kg ai/ hL	min water L/ha	kg ai/ ha	RTI days	kg ai/ ha		
Apple Pear	USA	F	480SC	(4–8)	0.6	94	0.28–0.56	7–14	2.24	14	–
			50WS		n.s.	n.s.					
Brassica: Head/Stem & Leafy Greens	USA	F	480SC	(2–3)	0.3 (ground) 1.5 (aerial)	94 (ground) 19 (aerial)	0.21–0.28	14	0.63	1	–
Leafy Greens										0	
Cherry	USA	F	480SC	(6–12)	n.s. (ground) 0.6 (aerial)	n.s. (ground) 94 (aerial)	0.28–0.56	7–14	3.36	1	Apply early popcorn, full bloom, and petal fall
			50WS	(6–9)	n.s.	n.s.	0.35–0.56				
Cucurbits	USA	G	SC	(5–10)	0.03	935	0.14–0.28	7–14	1.4	0	–
		F, G	480SC 50WS		n.s.	n.s.					
Tomato	USA	G	SC	(5–10)	0.03	935	0.14–0.28	7–14	1.4	0	–
Grape	USA	F	480SC 50WS	(4–8)	0.06 (50WS, grape; others: n.s.)	468 (50WS, grape; others: n.s.)	0.14–0.28	7–14	1.12	7	Apply pre-bloom
Strawberry										1	
Hazelnut	USA	F	480SC	(4–6)	0.02 (ground) 0.15 (aerial)	935 (ground) 140 (aerial)	0.14–0.21	10–14	0.84	18	Apply at bud-break
			50WS	(5–8)					1.12		
Hops	USA	F	480SC	(3)	0.09 (ground) 0.45 (aerial)	468 (ground) 94 (aerial)	0.42	14	1.4	7	Do not graze livestock in treated areas or harvest for silage or hay
Papaya (and other tropicals)	USA	F	480SC	(2–3)	0.05 (ground) 1.87 (aerial)	701 (ground) 19 (aerial)	0.28–0.35	14	0.84	0	–
Cucumber Courgette (Summer Squash)	Nether-lands	G	EC	1–6	0.015	500	0.075–0.225	7	1.35	1	Treat during harvesting period or 4 wks after planting
Tomato	Nether-lands	G	EC	1–5	0.015	500	0.075–0.225	7	1.125	1	Treat during harvesting period or 4 wks after planting

[a] SC=Terraguard® SC, 480 SC=Procure® 480 SC, 50WS=Procure® 50WS, EC=rocket®

[b] Numbers in parentheses are implied based on the maximum seasonal rate divided by the per-application rate.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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

Crop Group	Commodity	Table
Pome fruits	Apple	Table 42
	Pear	Table 43
Stone fruits	Cherry	Table 44
Berries and other small fruits	Grape	Table 45
	Strawberry	Table 46
Assorted tropical and sub-tropical fruits - inedible peel	Papaya	Table 47
Brassica (cole or cabbage) vegetables, Head cabbages,	Broccoli	Table 48
Flowerhead brassicas	Cabbage	Table 49
Fruiting vegetables, Cucurbits	Cucumber	Table 50
	Muskmelon	Table 51
	Squash	Table 52
Fruiting Vegetables, other than Cucurbits	Tomato	Table 53
Leafy Vegetables	Lettuce (Head and Leaf)	Table 54
	Mustard Green	Table 55
	Swiss Chard	Table 56
	Turnip Green	Table 57
Tree Nuts	Filberts	Table 58
Dried Herbs	Hops	Table 59

For all crops, triflumizole was applied as a foliar spray. Each field trial site consisted of an untreated control plot and at least one treated plot; at some locations, a second plot was treated at an exaggerated rate.

Parameters of the supervised residue trials were generally well reported in the detailed field reports and study volumes, including application equipment, environmental conditions, dates of application and harvest. The study volumes also include post-harvest treatment of the samples, shipping dates and conditions, method validation data from the analytical laboratories, and sufficient details regarding the analytical method. Information on varieties was not always available.

The supervised residue trials and results are summarized below. Residue data are corrected for concurrent recovery. Residues in samples from control plots were < LOQ and are not shown. Formulations in the tables below are as reported in the study volume and may not correspond to the submitted labels (e.g., “Procure W” or “Procure WP” reported vs. “Procure WS” label submitted. Residues in bold typeface are the highest residues considered appropriate for assessment where replicate residues have been identified. Labels from the USA do not specify the number of applications that may be made per growing season.

In the tables that follow, the GAP number of applications from USA labels are expressed in parenthesis to denote that they are implied based on the application rates and the maximum allowed seasonal rate. Also, residues in bold are the highest residue in a given data set appropriate for estimating short-term dietary intake and underlined residues are those for consideration in MRL and STMR estimations.

Pome fruits

Apples

A total of 18 supervised trials on apples were conducted in the USA from 1984 to 1987. Trials were conducted in Washington (four), Michigan (two), Ohio (three), Vermont (four), California (three), Pennsylvania (one), and New York (one) and are reported in Ball 1986c and Ball 1988b.

Trials reported by Ball 1986c consisted of four foliar applications of the triflumizole 50W formulation containing 500 g ai/kg (nominal), except for one trial each in Yakima, Washington; Wapato, Washington; and Lodi, California, which each consisted of two sets of four foliar applications at two different application rates. Trials reported by Ball 1988b consisted of four to six foliar applications of the triflumizole 50W formulation containing 498 to 523 g ai/kg, except for the trial located in Smith Flat, California, which consisted of one foliar application. All applications were made at a rate of approximately 0.56 kg ai/ha per application or 1.12 kg ai/ha per application. Applications were made on a 4-day to 109-day schedule until apples were ready to be harvested. The apples were harvested 7 and 14 days after the last treatment (PHI=7, 14 days), except for the trials located in Bennington, Vermont, and Smith Flat, California, which were residue decline trials with PHIs of 0, 7–9, 13–14, 20–21, and 28–29 days. One to three samples of apples were collected from the treated plots and untreated plots. Samples were stored for less than 2 years (Ball 1986c) or less than 10 months (Ball 1988b) before analysis.

The method for analysis was a common moiety method with gas chromatography. The LOQ was not determined, and the LOD was 0.02 mg eq/kg. Average recovery of all fortifications was 96.4 ± 19.5% for Ball 1986c (n=8) and 81.4 ± 12.9% for Ball 1988b (n=10). All control samples had residues below the limit of quantitation of the method, with two exceptions (in Wooster, Ohio, one control sample had residues of 0.04 mg eq/kg, and in Zillah, Washington, one control sample had residues of 0.03 mg eq/kg), and these are not considered significant. Apples treated with the triflumizole 50W formulation had detectable residues.

A total of two supervised trials on apples were conducted in Japan from 1982 to 1983. Trials were conducted in Aomori (one) and Nagano (one). Trials were reported by Gomyo 1992. None of the trials were conducted at the critical GAP.

The trial located in Aomori received eight foliar applications of the triflumizole WP formulation containing 300 g ai/kg, and the trial located in Nagano had three treated plots that received eight foliar applications of the triflumizole WP formulation containing 300 g ai/kg. Application volume was 6000 L/ha. Applications were made on a 9-day to 34-day schedule until apples were ready to be harvested. The apples were harvested 1, 7, and 21 days after the last treatment.

Apple samples were collected from treated plots and untreated plots. Specimens were stored frozen until analysis (approximately 1–3 months after harvest) by The Institute of Environmental Toxicology, Ibaraki, Japan. Samples were analysed for triflumizole and FM-6-1, and reported as the sum of both.

The method for analysis was high performance liquid chromatography. The LOQ was 0.008 mg eq/kg. Average recovery at 0.4 mg eq/kg was 86.4 ± 1.00% (n=4). All control samples had residues at or below the limit of quantitation of the method, and these are not considered significant. Apples treated with the triflumizole WP formulation had detectable residues.

Table 42 Summary of triflumizole residues in apple trials

			Application						
Trial ID/Country/Year	Storage Time (months)	Form	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP			0.56	n.s.	(4–8)	14	–	–	Label
UR1302/ USA, Yakima, WA/1985	13	W	0.56	n.a.	4	7	0.2	0.2	UR1302 Ball, 1986c

			Application						
Trial ID/Country/Year	Storage Time (months)	Form	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
									[a]
			1.12	n.a.	4	7	0.1	0.1	[a]
UR1302/ USA, East Lansing, MI/1986	1	W	0.56	n.a.	4	14	0.26	0.26	
UR1302/ USA, Wooster, OH/1986 (Red Delicious)	1	W	0.56	n.a.	4	14	0.1	0.1	
UR1302/ USA, Wooster, OH/1986 (Rome)	1	W	0.56	n.a.	4	14	0.1	0.1	
UR1302/ USA, Wooster, OH/1986 (Golden Delicious)	1	W	0.56	n.a.	4	14	0.3	0.3	
UR1302/ USA, Bennington, VT/1986 (McIntosh)	1	W	0.56	n.a.	4	14	< 0.02	< 0.02	
UR1302/ USA, Bennington, VT/1986 (McIntosh)	1	W	0.56	n.a.	4	14	< 0.02	< 0.02	
UR1302/ USA, Wapato, WA/1984 (Jonathan)	24	W	0.56	n.a.	4	14	< 0.02	< 0.02	[a]
			1.12	n.a.	4	14	0.16	0.16	[a]
UR1302/ USA, Wapato, WA/1984 (Golden Delicious)	24	W	0.56	n.a.	4	14	0.24	0.24	[a]
UR1302/ USA, Lodi, CA/1984	24	W	0.56	n.a.	4	14	0.18	0.18	[a]
			1.12	n.a.	4	14	0.34	0.34	[a]
UR1306/ USA, Zillah, WA/1987	8	WP	0.56	0.12	4	14	0.20, 0.38	0.29	UR1306 Ball, 1988b [a]
			0.56	0.015	4	14	0.29, 0.27	0.28	[a]
UR1306/ USA, Linden, CA/1987	8	WP	0.56	0.023	4	14	< 0.02	< 0.02	[a]
			0.56	0.12	4	14	0.06	0.06	[a]
			0.56	0.12	6	14	0.15	0.15	[a]
UR1306/ USA, Bennington, VT/1987	8	WP	0.56	0.05	4	14	0.09	0.09	[a]
			0.56	0.12	4	14	0.06	0.06	[a]
			0.56	0.05	6	14	0.09	0.09	[a]
			0.56	0.12	6	14	0.14	0.14	[a]
UR1306/ USA, Bennington, VT/1987	8	WP	0.56	0.05	4	0	0.29	0.29	[a]
						7	0.16	0.16	[a]
						13	0.17	0.17	[a]
						20	0.06	0.06	[a]
						29	0.12	0.12	[a]
			0.56	0.05	4	0	0.27	0.27	[a]
						9	0.12	0.12	[a]
						14	0.17	0.17	[a]
						21	0.04	0.04	[a]
						28	0.02	0.02	[a]
UR1306/ USA, Upper Black Eddy, PA/1987	9	WP	0.56	0.12	4	14	0.19	0.19	[a]
			0.56	0.015	4	14	0.07	0.07	[a]
			0.56	0.12	6	14	0.15	0.15	[a]

			Application						
Trial ID/Country/Year	Storage Time (months)	Form	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
			0.56	0.015	6	14	0.11	0.11	[a]
UR1306/ USA, East Lansing, MI/1987	9	WP	0.56	0.12	4	14	0.12	0.12	[a]
			0.56	0.015	4	14	0.15	0.15	[a]
			0.56	0.12	6	14	< 0.02	< 0.02	[a]
			0.56	0.015	6	14	0.14	0.14	[a]
UR1306/ USA, Clifton Springs, NY/1987	9	WP	0.56	0.015	4	14	0.14	0.14	[a]
			0.56	0.12	4	14	< 0.02	< 0.02	[a]
			0.56	0.015	6	14	0.12	0.12	[a]
			0.56	0.12	6	14	< 0.02	< 0.02	[a]
UR1306/ USA, Smith Flat, CA/1987	9	WP	0.56	0.015	1	0	0.08	0.08	[a]
						7	0.05	0.05	[a]
						14	< 0.02	< 0.02	[a]
						21	< 0.02	< 0.02	[a]
						28	< 0.02	< 0.02	[a]
RD-9235 Japan, Aomori/ 1982	3	WP	0.9	0.015	8	1	0.09	0.09	RD-9235 Gomyo, 1992
						7	0.03	0.03	
						21	0.02	0.026	
RD-9235 Japan, Nagano/ 1983	1	WP	0.9	0.015	8	1	0.24	0.24	
						7	0.03	0.03	
						21	0.01	0.01	

[a] The residue value(s) are not supported by the available storage stability data.

Pears

A total of seven supervised trials on pears were conducted in the USA from 1985 to 1987. Trials were conducted in Washington (two), California (two), Vermont (one), Oregon (one), and New York (one). Trials were reported in Ball 1986d and Ball 1988d.

Trials reported by Ball 1986d consisted of four foliar applications of the triflumizole 50W formulation containing 500 g ai/kg, except for one trial located in Ryde, California, which consisted of three sets of four foliar applications. Trials reported by Ball 1988d consisted of four to six foliar applications of the triflumizole 50W formulation containing 500 to 514 g ai/kg. All applications were made at a rate of approximately 0.56 kg ai/ha per application or 1.12 kg ai/ha per application. Applications were made on a 7-day to 100-day schedule until pears were ready to be harvested. The pears were harvested 14 days after the last treatment (PHI=14), except for the trial located in Ryde, California, which had a PHI of 15 days.

One to two samples of pears were collected from each of the treated and untreated plots and shipped, frozen, to the analytical laboratories. At each laboratory, samples were processed in a Hobart food processor and stored frozen until analysis. Samples were stored for less than 14 months (Ball 1986b) or less than 7 months (Ball 1988d) before analysis.

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/ECD. The LOQ was not determined, and the LOD was 0.02 mg eq/kg. Average recovery of all fortifications was 78.4 ± 21.1% for Ball 1986d (n=7) and 79.2 ± 13.6% for Ball 1988d (n=10). All control samples had residues below the limit of quantitation of the method. Pears treated with the triflumizole 50W formulation had detectable residues.

A total of two supervised trials on pears were conducted in Japan in 1985. Trials were conducted in Niigata (one) and Hiroshima (one). Trials were reported in Gomyo 1992.

The trials each had two plots that received eight foliar applications of the triflumizole WP formulation containing 300 g ai/kg. Application volume was 5000 L/ha, resulting in a rate of 0.75 kg ai/ha. Applications were made on a 4-day to 10-day schedule until pears were ready to be harvested. The pears were harvested 1, 7, and 21 days after the last treatment (PHI=1, 7, and 21). None of the trials were conducted at the critical GAP.

Pear samples were collected from treated plots and untreated plots. Specimens were stored frozen (approximately 1–2 months) until analysis by The Institute of Environmental Toxicology, Ibaraki, Japan. Samples were analysed for triflumizole and FM-6-1, and reported as the sum of both.

The method for analysis was high performance liquid chromatography. The LOQ was 0.02 mg eq/kg. Average recovery at 0.5 mg eq/kg was 87.3% (n=2). All control samples had residues at or below the limit of quantitation of the method, and these are not considered significant. Pears treated with the triflumizole WP formulation had detectable residues.

Table 43 Summary of triflumizole residue data in pears

			Use Pattern						
Trial ID/Country/Year	Storage Time (months)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)[a]	Average Residue (mg eq/kg)	Reference, remarks
USA GAP			0.56	n.s.	(4–8)	14	–	–	Label
UR1303/ USA, Wapato, WA/1985	14	W	0.56	0.015	4	14	0.29	0.29	UR1303 Ball, 1986d [a]
UR1303/ USA, Ryde, CA/1985	14	W	0.56	0.015	4	15	0.19	0.23	[a]
			0.56	0.015	4	15	0.27		[a]
			1.12	0.03	4	15	0.72	0.72	[a]
UR1303/ USA, Bennington, VT/1986	1	W	0.56	0.015	4	14	0.26, 0.30	0.28	
UR1307/ USA, Hood River, OR/1987	9	WP	0.56	0.12	4	14	0.13, 0.14	0.14	UR1307 Ball, 1988d [a]
				0.015	4	14	0.17, 0.11	0.14	[a]
				0.12	6	14	0.21, 0.15	0.18	[a]
UR1307/ USA, Clifton Springs, NY/1987	8	WP	0.56	0.015	4	14	0.34	0. 34	[a]
			0.56	0.12	4	14	0.43	0.43	[a]
UR1307/ USA, Zillah,, WA/1987	13	WP	0.56	0.015	4	14	0.36	0.36	[a]
UR1307/ USA, Linden, CA/1987	9	WP	0.56	0.023	4	14	0.12	0.12	[a]
					6	14	0.15	0.15	[a]
RD-9235 Japan, Niigata/ 1985	32	WP	0.75	0.075	8	1	0.31	0.31	RD-9235 Gomyo, 1992 [a]
						7	0.22	0.22	[a]
						21	0.11	0.11	[a]
RD-9235 Japan, Hiroshima/ 1985	32	WP	0.75	0.075	8	1	0.32	0.32	[a]
						7	0.23	0.23	[a]
						21	0.14	0.14	[a]

[a] The residue value(s) are not supported by the available storage stability data.

Cherries

A total of eight supervised trials on cherries were conducted in the USA in 1995. Trials were conducted in California (one), Michigan (three), New York (two), Washington (one), and Utah (one). Trials were reported in Gaydosh 1997 and Maselli 1997.

Each trial site, with the exception of California, contained two treated plots. One plot received six airblast applications of the triflumizole 50WS formulation containing 522.5 g ai/kg in 750 litres of water, and the second plot received six airblast applications of the triflumizole 50WS formulation containing 500 g ai/kg in 2340 litres of water. The treated plot in California received six airblast applications of the triflumizole 50WS formulation containing 522.5 g ai/kg in 2340 litres of water. All applications were made at a rate of approximately 0.52 to 0.57 kg ai/ha per application on a 1-day to 34-day schedule until cherries were ready to be harvested. The cherries were harvested 1, 3, and 7 days after the last treatment (PHI=1, 3, and 7).

Two samples of cherries each were collected from two treated plots (one treated plot in California) and one untreated plot. Specimens were stored frozen and shipped frozen to Morse Laboratories, Sacramento, California. Samples were de-stemmed, pitted, and ground in the presence of dry ice in a Hobart food grinder. Samples were stored frozen until analysis (< 12 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/ECD. The LOQ was 0.05 mg eq/kg, and the LOD was not reported. Average recovery of all fortifications was 76.5 ± 5.59% (n=11) for Gaydosh 1997 and 76.9 ± 7.77% (n=24) for Maselli 1997. All control samples had residues below the limit of quantitation of the method. Cherries treated with the triflumizole 50WS formulation had detectable residues.

Table 44 Summary of triflumizole residue data in cherries

		Use Pattern						
Trial ID/Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.56	n.s.	(6–12)	1	–	–	Label
RP-95012/ USA, Ripon, CA/1995	WS	0.52–0.57	0.024	6	1	1.0, 1.3	1.14	RP-95012 Gaydosh, 1997
					3	0.98	0.98	
					7	0.74, 0.54	0.64	
RP-95012/ USA, Conklin, MI/1995	WS	0.56	0.076–0.079	6	1	0.70, 0.68	0.69	
					3	0.65, 0.79	0.72	
					7	0.38, 0.57	0.47	
			0.023–0.024	6	1	0.88, 1.0	0.96	
					3	1.0, 0.64	0.84	
					7	0.45, 0.78	0.61	
RP-95012/ USA, Lyons, NY/1995	WS	0.56	0.072–0.074	6	1	0.91, 0.77	0.84	
					3	0.37, 0.56	0.47	
					7	0.35, 0.43	0.39	
			0.023–0.024	6	1	**1.5**, 0.91	1.19	
					3	0.61, 0.68	0.64	
					7	0.48, 0.45	0.47	
RP-95012/ USA, Zillah, WA/1995	WS	0.56	0.075	6	1	0.50, 0.62	0.56	
					3	0.28, 0.44	0.36	
					7	0.4, 0.32	0.36	
			0.024	6	1	0.50, 0.68	0.59	
					3	0.40, 0.51	0.46	
					7	0.49, 0.21	0.35	
RP-95013/ USA, Conklin, MI/1995	WS	0.56	0.075–0.079	6	1	1.1, 1.1	1.08	RP-95013 Maselli, 1997
					3	0.67, 0.99	0.83	

		Use Pattern						
Trial ID/Country/Year	For m.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	D AT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
					7	0.79, 0.67	0.73	
			0.023–0.024	6	1	1.0, 1.1	1.03	
					3	0.73, 0.81	0.77	
					7	0.99, 0.59	0.79	
RP-95013/ USA, Hart, MI/1995	WS	0.56	0.075–0.078	6	1	1.2, 1.2	1.19	
					3	0.52, 0.58	0.55	
					7	0.47, 0.7	0.58	
			0.023–0.024	6	1	1.5, 1.5	1.50	
					3	0.77, 0.74	0.75	
					7	0.52, 0.53	0.53	
RP-95013/ USA, Lyons, NY/1995	WS	0.55–0.57	0.075	6	1	0.32, 0.76	0.54	
					3	0.84, 0.83	0.84	
					7	0.3, 0.34	0.32	
		0.56–0.57	0.024	6	1	0.78, 0.66	0.72	
					3	1.0, 1.4	1.21	
					7	0.44, 0.41	0.42	
RP-95013/ USA, Perry, UT/1995	WS	0.56	0.075	6	1	0.82, 1.1	0.96	
					3	0.54, 0.63	0.59	
					7	0.33, 0.92	0.63	
			0.024	6	1	1.4, 1.1	1.26	
					3	0.90, 0.86	0.88	
					7	0.57, 0.78	0.68	

Berries and other small fruits

Grapes

A total of 18 supervised trials on grapes were conducted in the USA from 1985 to 1987. Trials were conducted in Washington (three), California (six), New York (eight), and Ohio (one). Trials were reported in Ball 1986b and Ball 1988c.

Trials reported by Ball 1986b received four foliar applications of the triflumizole 50W formulation containing 500 g ai/kg, except for the trials located in Portland, New York, which received three foliar applications. Trials reported by Ball 1988c received four foliar applications of the triflumizole 50W formulation containing 500 g ai/kg (nominal), with the exception of one site in Selma, California, which received one foliar application. All applications were made at a rate of approximately 0.28 kg ai/ha per application or 0.56 kg ai/ha per application. Applications were made on a 14-day to 78-day schedule until grapes were ready to be harvested. The grapes were harvested 7 days after the last treatment (PHI=7), except for the 44852 trial, which has PHIs of 0, 3, 7, 14, and 21 days.

Samples of grapes were collected from treated plots and untreated plots. Specimens were stored frozen and shipped frozen to the analytical laboratories. Samples were processed in a Hobart grinder and stored frozen until analysis (< 9 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by HPLC/UV or GC/NPD. The LOD was 0.02 mg eq/kg. Average recovery of all fortifications was 80.0 ± 14.1% (n=11) for Ball 1986b and 95.8 ± 16.0% (n=13) for Ball 1988c. Three control samples had residues at or below the limit of detection (in Madera, California, one control sample had residues of 0.03 mg eq/kg, and in Selma, California, two control samples had residues of 0.03 mg eq/kg each), and these are not considered significant. Grapes treated with the triflumizole 50W formulation had detectable residues.

Table 45 Summary of triflumizole residue data in grapes

		Use Pattern						
Trial ID/ Country/Year	Form	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.28	n.s.	4–8	7	–	–	Label
UR1304/ USA, Grandview, WA/1985	W	0.28	0.060	4	7	0.09	0.09	UR1304 Ball, 1986b
UR1304/ USA, Manteca, CA/1985	W	0.28	n.a.	4	7	0.10	0.10	
		0.56	n.a.	4	7	0.20	0.20	
UR1304/ USA, Madera, CA/1985	W	0.28	n.a.	4	7	0.18	0.18	
UR1304/ USA, Selma, CA/1985 Thompson Seedless	W	0.28	n.a.	4	7	0.15	0.15	
UR1304/ USA, Selma, CA/1985 Thompson Seedless	W	0.28	n.a.	4	7	0.15	0.15	
UR1304/ USA, Naples, NY/1985	W	0.28	n.a.	4	7	0.94	0.94	
UR1304/ USA, Portland, NY/1985	W	0.28	n.a.	3	7	0.09	0.09	
UR1304/ USA, Portland, NY/1985	W	0.28	n.a.	3	7	0.09	0.09	
UR1304/ USA, Geneva, NY/1986 Rosette	W	0.28	n.a.	4	7	1.2	1.2	
UR1304/ USA, Geneva, NY/1986 Rosette	W	0.28	n.a.	4	7	1.4	1.4	
UR1308/ USA, Phelps, NY/1987 Aurora	W	0.28	0.060	4	7	0.89	0.89	UR1308 Ball, 1988c
UR1308/ USA, Phelps, NY/1987 Catawba	W	0.28	0.060	4	7	2.02	2.02	
UR1308/ USA, Dundee, NY/1987	W	0.28	0.015	4	7	0.50	0.50	

		Use Pattern						
Trial ID/ Country/Year	Form	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
UR1308/ USA, Grandview, WA/1987 White Reisling	W	0.28	0.015	4	7	0.15, 0.16	0.16	
UR1308/ USA, Grandview, WA/1987 Chardonnay	W	0.28	0.015	4	7	0.21	0.21	
UR1308/ USA, Wooster, OH/1987	W	0.56	0.200	4	7	0.84	0.84	
UR1308/ USA, Selma, CA/1987 Thompson Seedless	W	0.28	0.030	1	0	0.38	0.38	
					3	0.16	0.16	
					7	0.05	0.05	
					14	0.06	0.06	
					21	< 0.02	< 0.02	
UR1308/ USA, Selma, CA/1987 Thompson Seedless	W	0.28	0.060	4	7	0.21	0.21	
			0.015	4	7	0.31	0.31	

Strawberries

A total of eight supervised trials on strawberries were conducted in the USA in 1999. Trials were conducted in Pennsylvania (one), Georgia (one), Florida (one), Indiana (one), California (three), and Oregon (one). Trials were reported in Gaydosh 2001.

At each trial, plots received three foliar applications of the triflumizole 50WS formulation containing 500 g ai/kg. All applications were made at a rate of approximately 0.41 to 0.43 kg ai/ha per application. Applications were made on a 10-day to 14-day schedule until strawberries were ready to be harvested. The strawberries were harvested 1 and 3 days after the last treatment (PHI=1 and 3 days).

Two samples of strawberries were collected from one treated plot and one sample was collected from one untreated plot. Specimens were stored frozen and shipped frozen to Morse Laboratories, Inc., Sacramento, California. Samples were processed with dry ice in a Hobart grinder and stored frozen until analysis (< 9 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.02 mg eq/kg, and the LOD was not reported. Average recovery of all fortifications was 80.5 ± 5.0% (n=12) for strawberries. All control samples had residues at or below the limit of quantitation of the method. Strawberries treated with the triflumizole 50WS formulation had detectable residues.

Table 46 Summary of triflumizole residue data in strawberries

			Use Pattern						
Trial ID/Country/Year	Storage Time (days)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP			0.28	n.s.	(4–8)	1	–	–	Label
RP-99005/ USA, New Tripoli, PA/1999	232	WS	0.42	0.09	3	1	0.68, 0.71	0.70	RP-99005 Gaydosh, 2001 [a]
						3	0.40, 0.22	0.31	[a]
RP-99005/ USA, Cochran, GA/1999	232	WS	0.41–0.43	0.088–0.091	3	1	0.95, 0.82	0.89	[a]
						3	0.83, 0.48	0.66	[a]
RP-99005/ USA, Eustis, FL/1999	253	WS	0.42	0.09	3	1	0.97, 0.75	0.86	[a]
						3	0.44, 0.30	0.37	[a]
RP-99005/ USA, Noblesville, IN/1999	195	WS	0.42–0.43	0.090–0.091	3	1	0.48, 0.45	0.47	[a]
						3	0.24, 0.22	0.23	[a]
RP-99005/ USA, Salinas, CA/1999	235	WS	0.42	0.089–0.094	3	1	1.2, 0.88	1.0	[a]
						3	0.65, 0.22	0.44	[a]
RP-99005/ USA, Watsonville, CA/1999	246	WS	0.42	0.088–0.091	3	1	1.6, 2.0	1.8	[a]
						3	0.86, 1.2	1.0	[a]
RP-99005/ USA, Oceanside, CA/1999	253	WS	0.41–0.43	0.089–0.091	3	1	0.53, 0.89	0.71	[a]
						3	0.57, 0.22	0.41	[a]
RP-99005/ USA, Mt. Angel, OR/1999	230	WS	0.42	0.09	3	1	0.40, 0.41	0.41	[a]
						3	0.30, 0.17	0.24	[a]

[a] The residue value(s) are not supported by the available storage stability data.

Assorted Tropical and Sub-Tropical Fruits-Inedible Peel

Papaya

A total of four supervised trials on papaya were conducted in the USA from 2005 to 2006. Trials were conducted in Hawaii (three), and Florida (one). Trials were reported by Barney 2007f.

At each trial, plots received five foliar applications of the triflumizole 480SC formulation containing 453 g ai/L. All applications were made at a rate of approximately 0.41 to 0.44 kg ai/ha per application. Applications were made on a 12-day to 14-day schedule until papaya was ready to be harvested. The papaya was harvested 0 days after the last treatment (PHI=0).

Two samples of papaya were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to IR-4 Western Region Satellite Laboratory, Honolulu, Hawaii. Samples of whole fruit were processed with dry ice in a Hobart food cutter and stored frozen until analysis (< 19 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.033 mg eq/kg, and the LOD was 0.011 mg eq/kg. Method validation results for triflumizole averaged 82 ± 7% at 0.050 mg eq/kg, 76 ± 6% at 1.0 mg eq/kg and 81 ± 4% at 10 mg eq/kg. Method validation results for FM-6-1 averaged 81 ±4% at 0.050 mg eq/kg, 72 ± 5% at 1.0 mg eq/kg and 73 ± 4% at 10 mg eq/kg. Method validation results for FA-1-1 averaged 85 ± 4% at 0.050 mg eq/kg, 79 ± 3% at 1.0 mg eq/kg and 75 ± 1% at 10 mg eq/kg. All control

samples had residues at or below the limit of quantitation of the method. Papaya treated with the triflumizole 480SC formulation had detectable residues.

Table 47 Summary of triflumizole residues in papaya

			Use Pattern						
Trial ID/ Country/Year	Storage Time (days)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP			0.35	n.s.	2–3	0	–	–	Label
09332/ USA, Pahoa, HI/2005	461	SC	0.41–0.43	0.050	5	0	0.85, 0.92	0.89	09332 Barney, 2007f
09332/ USA, Keaau, HI/2005	423	SC	0.42–0.43	0.041	5	0	0.53, 0.53	0.53	
09332/ USA, Waimanalo, HI/2005–2006	351	SC	0.41–0.44	0.034	5	0	0.91, 0.85	0.88	
09332/ USA, Homestead, FL/2005	561	SC	0.43	0.024	5	0	0.20, 0.24	0.22	

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

Broccoli

A total of 10 supervised trials on broccoli were conducted in the USA from 2003 to 2007. Trials were conducted in Texas (two), Maryland (one), California (five), New Mexico (one), and Oregon (one). Trials were reported in Gaydosh 2004a and Barney 2007c.

The trial reported in Gaydosh 2004a received five foliar applications of the triflumizole 50WS formulation containing 501 g ai/kg. Trials reported in Barney 2007c received four to six foliar applications of the triflumizole 480SC formulation containing 430 g ai/l. All applications were made at a rate of approximately 0.27 to 0.57 kg ai/ha per application. Applications were made on a 6-day to 8-day schedule until broccoli was ready to be harvested. The broccoli was harvested 0 days after the last treatment (PHI=0), except for the AWD-03907 trial, which had PHIs of 1, 3, and 7 days, and the 06-CA11 trial, which had PHIs of 0, 1, 4, 7, and 11 days.

Samples of broccoli were collected from treated plots and untreated plots. Specimens were stored frozen and shipped frozen to Morse Laboratories, Sacramento, California, where they were processed with dry ice in a Hobart food grinder and stored frozen until analysis (approximately 9 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.05 mg eq/kg (Gaydosh 2004a) or 0.032 mg eq/kg (Barney 2007c), and the LOD was not determined (Gaydosh 2004a) or 0.011 mg eq/kg (Barney 2007c). Average recovery of all triflumizole fortifications was 74 ± 4% (Gaydosh 2004a) and 81 ± 10%, 82 ± 3%, and 84 ± 4%, for the three validation levels. All control samples had residues at or below the limit of quantitation of the method. Broccoli treated with the triflumizole 50WS formulation and the triflumizole 480SC formulation had detectable residues.

Table 48 Summary of triflumizole residue data in broccoli

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.28	n.s.	2–3	1	–	–	Label
RP-03006/ USA, Uvalde, TX/2003	WS	0.53–0.57	0.204–0.245	5	1	0.32, 0.86, 0.93	0.70	RP-03006 Gaydosh, 2004a
					3	0.50, 0.81, 0.28	0.53	
					7	0.17, 0.20, 0.20	0.19	
09319/ USA, Salisbury, MD/2006	SC	0.27–0.28	0.214–0.218	5	0	4.0, 3.8	3.9	09319 Barney, 2007c [a]
09319/ USA, Salinas, CA/2006 Everest	SC	0.29–0.30	0.077–0.094	4	0	0.89, 1.8	1.4	[a]
09319/ USA, Salinas, CA/2006 Greenbelt	SC	0.28–0.29	0.053–0.073	4	0	0.35, 1.2	0.80	[a]
09319/ USA, Parlier, CA/2006	SC	0.28–0.29	0.099–0.101	4	0	0.82, 0.41	0.62	[a]
09319/ USA, Las Cruces, NM/2006	SC	0.28–0.30	0.099	4	0	0.28, 0.61	0.45	[a]
09319/ USA, Aurora, OR/2006	SC	0.28–0.30	0.199–0.200	5	0	0.43, 0.54	0.49	[a]
09319/ USA, Holtville, CA/2007	SC	0.28	0.115–0.119	4	0	0.78, 0.89	0.83	[a]
					1	0.74, 0.43	0.59	[a]
					4	0.10, 0.29	0.19	[a]
					7	0.46, 0.37	0.41	[a]
					11	0.11, 0.26	0.18	[a]
09319/ USA, Davis, CA/2006	SC	0.28–0.31	0.100–0.120	4	0	0.65, 0.53	0.59	[a]
09319/ USA, Weslaco, TX/2006–2007	SC	0.28–0.29	0.159–0.160	6	0	1.1, 0.85	0.97	[a]

[a] The residue value(s) are not supported by the available storage stability data.

Cabbages, Head

A total of nine supervised trials on cabbage were conducted in the USA from 2006 to 2007. Trials were conducted in Maryland (one), New Jersey (one), New York (one), Wisconsin (one), Florida (one), Texas (one), Tennessee (one), Colorado (one) and California (one). Trials were reported in Barney 2007d.

At each trial, plots received four foliar applications of the triflumizole 480SC formulation containing 430 g ai/kg, except for the Tennessee trial, which received five foliar applications. All applications were made at a rate of approximately 0.27 to 0.30 kg ai/ha per application. Applications

were made on a 6-day to 8-day schedule until cabbage was ready to be harvested. The cabbage was harvested 0 days after the last treatment (PHI=0).

Two samples of cabbage (with wrapper leaves) each were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to Morse Laboratories, Sacramento, California, where they were processed with dry ice in a Hobart food grinder and stored frozen until analysis (< 9 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.030 mg eq/kg, and the LOD was 0.010 mg eq/kg. Average recovery of all fortifications was 83 ± 7% (n=16) for cabbage. All control samples had residues at or below the limit of quantitation of the method. Cabbage treated with the triflumizole 480SC formulation had detectable residues.

Table 49 Summary of triflumizole residue data in cabbage

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.28	n.s.	2–3	1	–	–	Label
09143/ USA, Salisbury, MD/2006	SC	0.28–0.29	0.084	4	0	0.61, 0.26	0.43	09143 Barney, 2007d [a]
09143/ USA, Bridgeton, NJ/2006	SC	0.27–0.29	0.069–0.074	4	0	0.57, 1.0	0.79	[a]
09143/ USA, Freeville, NY/2006	SC	0.28	0.099	4	0	0.41, 0.55	0.48	[a]
09143/ USA, Arlington, WI/2006	SC	0.28–0.30	0.086–0.090	4	0	0.47, 0.18	0.33	[a]
09143/ USA, Citra, FL/2006	SC	0.29	0.086	4	0	1.2, 1.0	1.10	[a]
09143/ USA, Weslaco, TX/2007	SC	0.28	0.077–0.079	4	0	0.42, 0.19	0.31	[a]
09143/ USA, Crossville, TN/2006	SC	0.29–0.30	0.085–0.088	5	0	0.17, 0.19	0.18	[a]
09143/ USA, Brighton, CO/2006	SC	0.28–0.30	0.149–0.150	4	0	0.75, 0.36	0.55	[a]
09143/ USA, Salinas, CA/2006	SC	0.27–0.28	0.051–0.083	4	0	0.63, 0.60	0.62	[a]

[a] The residue value(s) are not supported by the available storage stability data.

Fruiting vegetables, Cucurbits

The Meeting received supervised residue trials on cucurbit vegetables. Trials with cucumbers were conducted in both indoor and outdoor environments.

Cucumbers (Outdoor)

A total of six supervised trials on cucumbers were conducted in the USA in 1997. Trials were conducted in North Carolina (one), South Carolina (one), Florida (one), Indiana (two), and Texas (one). Trials were reported in Maselli 1999.

At each trial, plots received five foliar applications of the triflumizole 50WS formulation containing 500 g ai/kg. All applications were made at a rate of approximately 0.26 to 0.30 kg ai/ha per application. Applications were made on a 7-day to 11-day schedule until cucumbers were ready to be harvested. The cucumbers were harvested 0 day after the last treatment (PHI=0).

Two samples of cucumbers were collected from one treated plot and one sample was collected from one untreated plot. Specimens were stored frozen and shipped frozen to PTRL West, Inc., Richmond, California. Samples were processed with dry ice in either a Hobart or WestBend food processor and were stored frozen until analysis (< 30 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/NPD. The LOQ was 0.05 mg eq/kg, and the LOD was not determined. Average recovery of all fortifications was 82.0 ± 9.8% (n=18) for cucumbers. All control samples had residues below the limit of quantitation of the method. Cucumbers treated with the triflumizole 50WS formulation had detectable residues.

Cucumbers (Indoor)

A total of four supervised trials on cucumbers were conducted in USA from 2009 to 2010. Trials were conducted in Texas (one), Florida (one), Colorado (one), and Maryland (one). Trials were reported in Homa 2012.

At each trial, plots received five foliar applications of the triflumizole 480SC formulation containing 431 g ai/L. All applications were made at a rate of approximately 0.26 to 0.30 kg ai/ha per application. Applications were made on a 6- to 8-day schedule until cucumbers were ready to be harvested. The cucumbers were harvested 0 day after the last treatment (PHI=0), except for the trial in Colorado which had PHIs of 0, 3, 7, 12, and 15 days.

Two samples of cucumbers were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to the Western Region Laboratory at the University of California, Davis (UCD), Davis, California. Samples were processed with dry ice in a Hobart food chopper and were stored frozen until analysis (< 15 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD in selective ion monitoring (SIM) mode. Average recovery of all fortifications was 76 ± 8% (n=9) for cucumbers. All control samples had residues below the limit of quantitation of the method. Cucumbers treated with the triflumizole 480SC formulation had detectable residues.

A total of four supervised trials on cucumbers were conducted in Japan from 1982 to 1990. Trials were conducted in Ibaragi (one), Osaka (two), and Chiba (one). Trials were reported in Gomyo 1993.

Two plots received five foliar applications of the triflumizole 15EC formulation containing 150 g ai/L, and two plots received five foliar applications of the triflumizole 30WP formulation containing 300 g ai/kg. All applications were made at a rate of approximately 0.06 to 0.30 kg ai/ha per application. Applications were made on a 6-day to 9-day schedule until cucumbers were ready to be harvested. The cucumbers were harvested 1, 3, and 7 days after the last treatment (PHI=1, 3, and 7 days).

One sample of cucumber was collected from one treated plot and one untreated plot. Specimens were stored frozen until analysis by Environmental Toxicology Laboratory of Nippon Soda Co., Odawara, Japan. Storage time was less than 30 months.

The method for analysis was high performance liquid chromatography with UV detection and results are reported as combined residues of triflumizole and FM-6-1. The LOQ was 0.02 mg eq/kg. All control samples had residues at or below the limit of quantitation of the method. Cucumbers treated with the triflumizole 15EC formulation and the triflumizole 30WP formulation had detectable residues.

A total of eight supervised trials on cucumbers were conducted in the Netherlands in 1993. Trials were conducted in Harmelen (one), Vleuten (one), Houten (one), Maarssen (one), Zevenhuizen (one), Moerkapelle (one), Bleiswijk (one), and Bergschenhoek (one). Trials were reported in Geuijen 1994.

For each trial, plots received three to four foliar applications of the triflumizole 15EC formulation containing 150 g ai/L. All applications were made at a rate of approximately 0.228 to 0.533 kg ai/ha per application. Applications were made on a 7-day schedule until cucumbers were ready to be harvested. The cucumbers were harvested 3 days after the last treatment (PHI=3), except for the trials located in Vleuten and Zevenhuizen, which had PHIs of 0, 1, 3, and 7 days.

One sample each of cucumber was collected from two treated plots and one untreated plot. Specimens were received by the analysis department, TNO Nutrition Research Institute, AJ Zeist, Netherlands, on the day they were harvested. Samples were homogenized using a Stephan cutter. Processed samples were stored refrigerated until analysis.

The method for analysis was high performance liquid chromatography with UV detection. The LOQ was approximately 0.1 mg eq/kg, and the LOD was 0.02 mg eq/kg. A total of 7 control samples had residues at or above the limit of quantitation of the method, and these are not considered significant. Cucumbers treated with the triflumizole 15EC formulation had detectable residues.

Table 50 Summary of triflumizole residue data in cucumbers

			Use Pattern						
Trial ID/Country/Year	Storage Time (months)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA (Outdoor)									
USA GAP			0.28	n.s.	(5-10)	0	–	–	Label
RP-97006/ USA, Knightdale, NC/1997	6	WS	0.28–0.29	0.098–0.103	5	0	0.18, 0.17	0.18	RP-97006 Maselli, 1999
RP-97006/ USA, Elko, SC/1997	7	WS	0.28–0.29	0.069–0.149	5	0	0.12, 0.21	0.17	[a]
RP-97006/ USA, Duette, FL/1997	8	WS	0.28	0.097–0.099	5	0	0.13, 0.11	0.12	[a]
RP-97006/ USA, Noblesville, IN/1997 Marketmore 76	4	WS	0.28	0.099–0.145	5	0	0.18, 0.15	0.17	
RP-97006/ USA, Noblesville, IN/1997 Wisconsin SMR 58	5	WS	0.28–0.29	0.101–0.147	5	0	0.13, 0.12	0.13	
RP-97006/ USA, Donna, TX/1997	6	WS	0.26–0.30	0.101–0.126	5	0	0.11, 0.14	0.13	
USA (Indoor)									
09300/ USA,	1	SC	0.28	0.027	5	0	0.11, < 0.10 [b]	0.11	09300

			Use Pattern						
Trial ID/Country/Year	Storage Time (months)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
Weslaco, TX/2009									Homa, 2012
09300/ USA, Citra, FL/2010	14	SC	0.28–0.29	0.03	5	0	0.27, 0.25	0.26	[a]
09300/ USA, Fort Collins, CO/2009	2	SC	0.27–0.30	0.030–0.033	5	0	0.10, < 0.10 [b]	0.1	
						3	< 0.10 [b], 0.11	0.11	
						7	< 0.10 [b], < 0.10 [b]	< 0.10	
						12	< 0.10 [b]< 0.10 [b]	< 0.10	
						15	< 0.10 [b], < 0.10 [b]	< 0.10	
09300/ USA, Salisbury, MD/2009	4	SC	0.26	0.022	5	0	0.17, 0.25	0.21	
Japan									
RD-9331/ Japan, Ibaragi/1990	3	EC	0.0600 – 0.1875	0.0075	5	1	< 0.02	< 0.02	RD-9331 Gomyo, 1993
						3	< 0.02	< 0.02	
						7	< 0.02	< 0.02	
RD-9331/ Japan, Osaka/1990	2	EC	0.25	0.01	5	1	0.04	0.04	
						3	< 0.02	< 0.02	
						7	< 0.02	< 0.02	
RD-9331/ Japan, Chiba/1982	2	WP	0.3	0.01	5	1	0.1	0.1	
						3	0.09	0.09	
						7	0.04	0.04	
RD-9331/ Japan, Osaka/1983	1	WP	0.2	0.01	5	1	0.28	0.28	
						3	0.25	0.25	
						7	0.1	0.1	
Netherlands									
Netherlands GAP			0.225	0.015	1–6	1	–	–	Label
F-93-30-NL-00/02/ Netherlands, Harmelen/ 1993	> 9 months [c]	EC	0.228–0.244	0.015	3	3	< 0.04	< 0.04	F-93-30 Geuijen, 1994
			0.231–0.250	0.015	3	3	< 0.04	< 0.04	
F-93-30-NL-00/02/ Netherlands, Vleuten/1993	> 9 months	EC	0.234–0.264	0.015	3	0	< 0.05	< 0.05	
						1	< 0.05	< 0.05	
						3	< 0.04	< 0.04	
						7	< 0.02	< 0.02	
			0.246–0.267	0.015	3	0	< 0.06	< 0.06	
						1	< 0.05	< 0.05	
						3	< 0.04	< 0.04	
						7	< 0.02	< 0.02	
F-93-30-NL-00/02/ Netherlands,	> 9 months	EC	0.243–0.268	0.015	3	3	< 0.04	< 0.04	

			Use Pattern						
Trial ID/Country/Year	Storage Time (months)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
Houten/1993									
			0.242–0.269	0.015	3	3	< 0.04	< 0.04	
F-93-30-NL-00/02/ Netherlands, Maarssen/ 1993	> 9 months	EC	0.244–0.268	0.022	3	3	< 0.04	< 0.04	
			0.241–0.270	0.021–0.022	3	3	< 0.04	< 0.04	
F-93-30-NL-00/02/ Netherlands, Zevenhuizen/ 1993	> 9 months	EC	0.498–0.509	0.025	4	0	0.17	0.17	
						1	0.17	0.17	
						3	0.11	0.11	
						7	0.07	0.07	
			0.497–0.505	0.025–0.026	4	0	0.16	0.16	
						1	0.18	0.18	
						3	0.10	0.10	
						7	0.06	0.06	
F-93-30-NL-00/02/ Netherlands, Moerkapelle/ 1993	> 9 months	EC	0.483–0.491	0.025	4	3	0.11	0.11	
			0.500–0.509	0.025–0.026	4	3	0.09	0.09	
F-93-30-NL-00/02/ Netherlands, Bleiswijk/ 1993	> 9 months	EC	0.484–0.500	0.025	4	3	0.14	0.14	
			0.495–0.505	0.025	4	3	0.13	0.13	
F-93-30-NL-00/02/ Netherlands, Bergschenhoek/ 1993	> 9 months	EC	0.492–0.497	0.025	4	3	0.23	0.23	
			0.503–0.533	0.025	4	3	0.18	0.18	

[a] The residue value(s) are not supported by the available storage stability data.

[b] 0.10 mg eq/kg was used to calculate the average residue.

[c] Based on harvest and communication dates specified in the study report.

Muskmelons

A total of six supervised trials on muskmelons were conducted in the USA in 1997. Trials were conducted in North Carolina (one), Indiana (one), Texas (one) and California (three). Trials were reported by Gaydosh, Maselli, and Puhl 1999.

At each trial, plots received five foliar applications of the triflumizole 50WS formulation containing 500 g ai/kg (nominal). All applications were made at a rate of approximately 0.26 to 0.32 kg ai/ha per application. Applications were made on a 7-day to 10-day schedule until muskmelons were ready to be harvested. The muskmelons were harvested 0 day after the last treatment (PHI=0).

Two samples of muskmelons were collected from each treated plot and one sample was collected from each untreated plot. Specimens were stored frozen and shipped frozen to PTRL West, Inc., Richmond, California. Whole melon samples were sectioned and composited to obtain a representative sample for the field. Samples (pulp + peel) were processed with dry ice in a Hobart cutter/mixer and were stored frozen until analysis (< approximately 7 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/NPD. The LOQ was 0.05 mg eq/kg. Average recovery of all fortifications was 93.5 ± 19.0% (n=6) for muskmelons. All control samples had residues below the limit of quantitation of the method. Muskmelons treated with the triflumizole 50WS formulation had detectable residues.

Table 51 Summary of triflumizole residue data in muskmelons

			Use Pattern						
Trial ID/Country/Year	Storage Time (days)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue mg eq/kg)	Average Residue mg eq/kg)	Reference, remarks
USA GAP			0.28	n.s.	(5–10)	0	–	–	Label
RP-97005/ USA, Knightdale, NC/1997	174	WS	0.27–0.28	0.094–0.101	5	0	0.15, 0.13	0.14	RP-97005 Gaydosh, Maselli, Puhl, 1999 [a]
RP-97005/ USA, Noblesville, IN/1997	160	WS	0.28–0.29	0.102–0.146	5	0	0.40, 0.35	0.38	[a]
RP-97005/ USA, Donna, TX/1997	203	WS	0.26–0.32	0.107–0.122	5	0	0.15, 0.15	0.15	[a]
RP-97005/ USA, Winters, CA/1997	214	WS	0.28–0.30	0.054–0.104	5	0	0.07, 0.08	0.07	[a]
RP-97005/ USA, Manteca, CA/1997	158	WS	0.28	0.098–0.100	5	0	0.27, 0.24	0.25	[a]
RP-97005/ USA, Fresno, CA/1997	153	WS	0.28	0.040–0.060	5	0	0.17, 0.25	0.21	[a]

[a] The residue value(s) are not supported by the available storage stability data.

Squash, Summer

A total of five supervised trials on summer squash were conducted in the USA in 1997. Trials were conducted in Pennsylvania (one), South Carolina (one), Florida (one), Indiana (one), and California (one). Trials were reported in Gaydosh 1999.

At each trial, plots received five foliar applications of the triflumizole 50WS formulation containing 522 g ai/kg. All applications were made at a rate of approximately 0.28 to 0.29 kg ai/ha per application. Applications were made on a 7-day to 10-day schedule until squash was ready to be harvested. The squash was harvested 0 days after the last treatment (PHI=0).

Two samples of squash were collected from one treated plot and one sample was collected from one untreated plot. Specimens were stored frozen and shipped frozen to PTRL West, Inc., Richmond, California. Samples were processed with dry ice in a Hobart or WestBend food processor and stored frozen until analysis (< 9 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/NPD. The LOQ was 0.05 mg eq/kg. Average verification recoveries for triflumizole in two verification sets were 79.0 ± 8.0% (n=6) and 77.2 ± 3.7% (n=6), while verification recoveries for FM-6-1 averaged 74.3 ± 11.6% (n=6) and 81.1 ± 8.2% (n=6). All control samples had residues at or below the limit of quantitation of the method. Squash treated with the triflumizole 50WS formulation had detectable residues.

Table 52 Summary of triflumizole residue data in summer squash

			Use Pattern						
Trial ID/Country/Year	Storage Time (days)	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP			0.28	n.s.	(5–10)	0	–	–	Label
RP-97007/ USA, Hamburg, PA/1997	186	WS	0.28–0.29	0.065–0.143	5	0	0.12, 0.15	0.14	RP-97007 Gaydosh, 1999 [a]
RP-97007/ USA, Elko, SC/1997	234	WS	0.28–0.29	0.070–0.151	5	0	0.14, 0.14	0.14	[a]
RP-97007/ USA, Duette, FL/1997	254	WS	0.28	0.097–0.099	5	0	0.13, 0.11	0.12	[a]
RP-97007/ USA, Noblesville, IN/1997	196	WS	0.28–0.29	0.102–0.145	5	0	0.19, 0.19	0.19	[a]
RP-97007/ USA, Lathrop, CA/1997	188	WS	0.28	0.099–0.100	5	0	0.31, 0.39	0.35	[a]

Fruiting vegetables, Other than Cucurbits

Tomatoes

A total of four supervised trials on tomato were conducted in USA in 2010. Trials were conducted in greenhouses in California (1), Maryland (1), Texas (1), and Wisconsin (1). Trials were reported by Barney 2012.

At each trial, plots received four or five foliar applications of the triflumizole 480SC formulation containing 431 g ai/L. All applications were made at a rate of approximately 0.26 to 0.29 kg ai/ha per application. Applications were made on a 6-day to 14-day schedule until tomatoes were ready to be harvested. The tomatoes were harvested 0 day after the last treatment (PHI=0), except for the trial located in California, which had PHIs of 0, 1, 4, 8, 10, and 15 days.

Two samples of tomatoes were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to USDA-ARS laboratory, Beltsville, MD. After reassignment of the analytical laboratory, samples were shipped frozen to IR-4 Western Region Laboratory, Davis, CA. Samples were processed with dry ice in a Hobart or Robot Coupe food chopper and stored frozen until analysis (< 19 months).

The analytical method assayed triflumizole and its aniline-containing metabolites. Total triflumizole residues are quantified using a gas chromatograph equipped with a mass selective detector (MSD) in selective ion monitoring (SIM) mode. The LOQ was 0.50 mg eq/kg. Average recovery of all fortifications was 85.3 ± 9.6% for tomatoes (n=13). All control samples had residues below the limit of quantitation of the method. Tomatoes treated with the triflumizole 480SC formulation had detectable residues.

A total of four supervised trials on tomatoes were conducted in the Netherlands in 1995 and 1996. Trials were conducted in greenhouses in Haps (one), Siebengewald (one), and Wellerlooi (two). Trials were reported by van de Ruit 1997.

At each trial, plots received three foliar applications of the triflumizole EC formulation containing 158 g ai/L. All applications were made at a rate of approximately 0.310 to 0.322 kg ai/ha per application. Applications were made on a 7-day schedule until tomatoes were ready to be harvested. The tomatoes were harvested 3 days after the last treatment (PHI=3), except for the trial located in Haps which was a decline trial with PHIs of 0, 1, 3, and 7 days.

One sample of tomato was collected from one treated plot and one untreated plot, except for the trial located in Haps (PHI=0) and the trial located in Siebengewald, in which duplicate samples were collected. Specimens were stored frozen and shipped frozen to BCO Analytical Services B.V.,

Bergschot, Breda, Netherlands. Samples were homogenized in a blender and stored frozen until analysis (< 19 months).

The analytical method assayed residues of triflumizole and FM-6-1, with quantitation by HPLC/UV. The results are reported as combined residues. The LOQ was 0.01 mg eq/kg. Average recovery of all fortifications was 93.3 ± 5.6% (n=6) for tomatoes. All control samples had residues below the limit of quantitation of the method. Tomatoes treated with the triflumizole EC formulation had detectable residues.

A total of four supervised trials on tomatoes were conducted in Belgium in 1995 and 1996. Trials were conducted in greenhouses in Onze-Lieve-Vrouw-Waver (one), St. Katelijne-Waver (two), and Duffel (one). Trials were reported in van de Ruit 1997.

At each trial, plots received three foliar applications of the triflumizole EC formulation containing 158 g ai/L. All applications were made at a rate of approximately 0.308 to 0.319 kg ai/ha per application. Applications were made on a 7-day schedule until tomatoes were ready to be harvested. The tomatoes were harvested 3 days after the last treatment (PHI=3), except for the trial located in Onze-Lieve-Vrouw-Waver which was a decline trial with PHIs of 0, 1, 3, and 7 days.

One sample of tomato was collected from one treated plot and one untreated plot, except for the trial located in Duffel in which three samples were collected from the treated plot and two samples were collected from the untreated plot. Specimens were stored frozen and shipped frozen to BCO Analytical Services B.V., Bergschot, Breda, Netherlands. Samples were homogenized in a blender and stored frozen until analysis (< 19 months).

The analytical method assayed residues of triflumizole and FM-6-1, with quantitation by HPLC/UV. Results are reported as combined residues. The LOQ was 0.01 mg eq/kg. Average recovery of all fortifications was 93.3 ± 5.6% (n=6) for tomatoes. All control samples had residues below the limit of quantitation of the method. Tomatoes treated with the triflumizole EC formulation had detectable residues.

Table 53 Summary of triflumizole residue data in tomatoes

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA (Greenhouse; Residue by common-moiety)								
USA GAP		0.28	n.s.	5–10	0	–	–	Label
09299/ USA, Parlier, CA/2010	SC	0.28–0.29	0.059–0.060	4	0	< 0.50 [a], < 0.50 [a]	< 0.50	09299 Barney, 2012
					1	< 0.50 [a], < 0.50 [a]	< 0.50	
					4	< 0.50 [a], < 0.50 [a]	< 0.50	
					8	< 0.50 [a], < 0.50 [a]	< 0.50	
					10	< 0.50 [a], < 0.50 [a]	< 0.50	
					15	< 0.50 [a], < 0.50 [a]	< 0.50	
09299/ USA, Salisbury, MD/2010	SC	0.26	0.023	4	0	< 0.50 [a], < 0.50 [a]	< 0.50	
09299/ USA, Weslaco, TX/2010	SC	0.29	0.034–0.035	5	0	< 0.50 [a], 0.67	0.59	

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
09299/ USA, Arlington, WI/2010	SC	0.28–0.29	0.039–0.044	4	0	0.63 , 0.88	0.76	
Netherlands (Greenhouse; Residue=triflumizole + FM-6-1)								
Netherlands GAP		0.225	0.015	1-5	1	–	–	Label
3096040731/ Netherlands, Haps, Brabant/1996	EC	0.311–0.322	0.016	3	0	0.63	0.63	3096040731 van de Ruit, 1997
					1	0.94	0.94	
					3	0.78	0.78	
					7	0.51	0.51	
3096040731/ Netherlands, Siebengewald, Limburg /1996	EC	0.313–0.322	0.016	3	3	0.20	0.20	
3096040731/ Netherlands, Wellerlooi, Limburg /1996	EC	0.314–0.316	0.016	3	3	0.48	0.48	
3096040731/ Netherlands, Wellerlooi, Limburg /1996	EC	0.310–0.316	0.016	3	3	0.48	0.48	
Belgium (Greenhouse; Residue=triflumizole + FM-6-1)								
3096040731/ Belgium, Onze-Lieve-Vrouw-Waver, Antwerpen /1996	EC	0.310–0.314	0.016	3	0	0.26	0.26	3096040731 van de Ruit, 1997
					1	0.20	0.20	
					3	0.18	0.18	
					7	0.27	0.27	
3096040731/ Belgium, St. Katelijne-Waver, Antwerpen /1996	EC	0.308–0.316	0.016	3	3	0.21	0.21	
3096040731/ Belgium, Duffel, Antwerpen /1996	EC	0.314–0.318	0.016	3	3	0.36, 0.38, 0.30	0.35	
3096040731/ Belgium, St. Katelijne-Waver, Antwerpen /1996	EC	0.314–0.319	0.016	3	3	0.14	0.14	

[a] 0.50 mg eq/kg was used to calculate the average residue.

Leafy vegetables (Including brassica leafy vegetables)

The critical GAP in the USA is 2–3 applications at 0.28 kg ai/ha at a 14-day interval. The maximum seasonal use rate is 0.63 kg ai/ha. The PHIs are 0 days for leafy vegetables and 1 day for Brassica leafy vegetables.

Lettuce

A total of 17 supervised trials on head and leaf lettuce were conducted in USA from 2003 to 2006. Trials were conducted in Texas (one), Maryland (one), New Jersey (two), California (eight), New Mexico (two), Georgia (two), and Colorado (one). Once New Jersey trial and the Colorado trial were conducted in a greenhouse. Trials were reported by Gaydosh 2004b and Barney 2007e.

The trial reported by Gaydosh 2004b received five foliar applications of the triflumizole 50WS formulation containing 501 g ai/kg. Trials reported by Barney 2007e received four foliar applications of triflumizole 480SC formulation containing 453 g ai/kg. All applications were made at a rate of approximately 0.27 to 0.59 kg ai/ha per application. Applications were made on a 6-day to 9-day schedule until lettuce was ready to be harvested. The lettuce was harvested 0 days after the last treatment (PHI=0), except for the AWD-03906 trial, which was a decline trial with PHIs of 1, 3, and 7 days, and the 05-NM05 and 05-CA27 trials, which was a decline trial with PHIs of 0, 2–3, 7–8, and 14 days.

Two to three samples of lettuce each were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to Morse Laboratories, Inc., Sacramento, California, or to Cornell Analytical Laboratories, Geneva, New York. Samples were processed with dry ice in a Hobart grinder or Hobart FP 30011 stored frozen until analysis (< approximately 2 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.05 mg eq/kg. Average recovery of all fortifications was 74 ± 4% (n=12) for Gaydosh 2004b and 90 ± 19% (n=8) for Barney 2007e. All control samples had residues below the limit of quantitation of the method. Lettuce treated with the triflumizole 50WS formulation and the triflumizole 480SC formulation had detectable residues.

Table 54 Summary of triflumizole residue data in lettuce

			Use Pattern						
Trial ID/ Country/Year	Lettuce Type	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP	–		0.28	n.s.	2–3	0	–	–	Label
RP-03005/ USA, Uvalde, TX/2003	n.s.	WS	0.55–0.59	0.201–0.250	5	1	4.8, 5.8, 6.3	5.6	RP-03005 Gaydosh, 2004b
						3	3.6, 2.4, 2.6	2.9	
						7	0.34, 0.32, 0.39	0.35	
08993/ USA, Salisbury, MD/2005	Head	SC	0.28–0.29	0.086	4	0	3.0, 2.0	2.5	08993 Barney, 2007e
08993/ USA, Bridgeton, NJ/2005	Head	SC	0.28–0.31	0.087	4	0	2.1, 3.7	2.9	
08993/ USA, Salinas, CA/2005	Head	SC	0.28	0.050–0.086	4	0	1.2, 1.4	1.3	
08993/ USA, Salinas, CA/2005	Head	SC	0.27–0.29	0.050–0.079	4	0	1.2, 0.68	0.94	

			Use Pattern						
Trial ID/ Country/Year	Lettuce Type	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
08993/ USA, Holtville, CA/2006	Head	SC	0.28–0.29	0.111–0.117	4	0	6.9, 8.3	7.6	
08993/ USA, Five Points, CA/2006	Head	SC	0.28–0.29	0.100	4	0	1.0, 0.99	1.0	
08993/ USA, near Mesilla, NM/2005	Head	SC	0.28	0.056	4	0	4.6, 4.5	4.6	
						3	0.40, 1.1	0.75	
						7	0.78, 0.42	0.60	
						14	0.08, 0.11	0.09	
08993/ USA, Tifton, GA/2005	Leaf	SC	0.28	0.094	4	0	11, 10	11	
08993/ USA, Tifton, GA/2005	Leaf	SC	0.28	0.094	4	0	17, 16	17	
08993/ USA, Salinas, CA/2005	Leaf	SC	0.28–0.29	0.051–0.073	4	0	1.2, 1.4	1.3	
08993/ USA, Salinas, CA/2005	Leaf	SC	0.27–0.29	0.069–0.085	4	0	18, 24	21	
08993/ USA, Holtville, CA/2005–2006	Leaf	SC	0.28–0.29	0.109–0.117	4	0	18, 17	18	
						2	13, 16	15	
						8	7.0, 9.1	8.1	
						14	4.4, 2.8	3.6	
08993/ USA, Parlier, CA/2005	Leaf	SC	0.28	0.099–0.100	4	0	3.2, 4.8	4.0	
08993/ USA, near Mesilla, NM/2005	Leaf	SC	0.28–0.29	0.201–0.202	4	0	6.3, 4.6	5.5	
08993/ USA, Bridgeton, NJ/2005	Leaf (Green-house)	SC	0.27–0.30	0.058	4	0	9.6, 9.3	9.5	
08993/ USA, Fort Collins, CO/2005	Leaf (Green-house)	SC	0.28–0.29	0.149–0.150	4	0	17, 12	15	

Mustard greens

A total of ten supervised trials on mustard greens were conducted in the USA from 2004 to 2005. Trials were conducted in New Jersey (one), Maryland (one), Wisconsin (one), Georgia (one), Texas (one), Florida (one), North Carolina (one), Tennessee (one), and California (two). Trials were reported by Barney 2006.

At each trial, plots received four foliar applications of the triflumizole 50WS formulation containing 500 g ai/kg. All applications were made at a rate of approximately 0.27 to 0.29 kg ai/ha per application. Applications were made on a 6- to 10-day schedule until mustard greens were ready to be harvested. The mustard greens were harvested 1 day after the last treatment (PHI=1), except for the New Jersey trial, which was a decline trial with PHIs of 0, 1, 4, and 6 days.

Two samples of mustard greens were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped to Morse Laboratories, Inc., Sacramento, California. Samples were processed with dry ice in a Hobart food grinder and stored frozen until analysis (< 7 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.05 mg eq/kg. Average recovery of all fortifications was 84.6 ± 6.4% (n=9) for mustard greens. All control samples had residues below the limit of quantitation of the method. Mustard greens treated with the triflumizole 50WS formulation had detectable residues.

Table 55 Summary of triflumizole residue data in mustard greens

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.28	n.s.	2–3	0	–	–	Label
08865/ USA, Bridgeton, NJ/2004	WS	0.27–0.28	0.144–0.145	4	0	6.9, 4.5	5.7	08865 Barney, 2006 [a]
					1	0.99, 1.4	1.2	[a]
					4	1.2, 0.64	0.91	[a]
					6	0.59, 0.56	0.57	[a]
08865/ USA, Salisbury, MD/2004	WS	0.27–0.28	0.134–0.136	4	1	8.0, 13	11	[a]
08865/ USA, Arlington, WI/2004	WS	0.28–0.29	0.108–0.111	4	1	3.7, 3.8	3.8	[a]
08865/ USA, Tifton, GA/2004	WS	0.28–0.29	0.062–0.093	4	1	12, 6.3	8.9	[a]
08865/ USA, Weslaco, TX/2004	WS	0.28	0.066–0.099	4	1	2.7, 3.5	3.1	[a]
08865/ USA, Citra, FL/2004	WS	0.28–0.29	0.086	4	1	13, 18	15	[a]
08865/ USA, Clinton, NC/2004	WS	0.28–0.29	0.096–0.097	4	1	2.3, 4.1	3.2	[a]
08865/ USA, Jackson, TN/2004	WS	0.28	0.107–0.109	4	1	13, 11	12	[a]
08865/ USA, Salinas, CA/2004	WS	0.28–0.29	0.064–0.070	4	1	4.9, 4.3	4.6	[a]

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
08865/ USA, Holtville, CA/2004–2005	WS	0.27–0.28	0.109–0.114	4	1	4.1, 6.7	5.4	[a]

Swiss chard

A total of three supervised trials on Swiss chard were conducted in the USA in 2005. Trials were conducted in Maryland (one), Texas (one), and Oregon (one). Trials were reported by Barney 2007g.

At each trial, plots received four foliar applications of the triflumizole 480SC formulation containing 453 g ai/L. All applications were made at a rate of approximately 0.28 to 0.29 kg ai/ha per application. Applications were made on a 6-day to 8-day schedule until Swiss chard was ready to be harvested. The Swiss chard was harvested 0 day after the last treatment (PHI=0).

Two samples of Swiss chard each were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to Cornell Analytical Laboratories, Geneva, New York. Samples were processed with dry ice in a Hobart chopper and stored frozen until analysis (< 7 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.066 mg eq/kg, and the LOD was 0.022 mg eq/kg. Average recovery of all fortifications was 93 ± 16% (n=19) for Swiss chard. All control samples had residues at or below the limit of quantitation of the method. Swiss chard treated with the triflumizole 480SC formulation had detectable residues.

Table 56 Summary of triflumizole residue data in Swiss chard

		Use Pattern						
Trial ID/ Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	MRL GAP Residue (mg eq/kg)	Reference, remarks
USA GAP		0.28	n.s.	2–3	0	–	–	Label
08867/USA, Salisbury, MD/2005	SC	0.28	0.126	4	0	7.7, 9.2	8.5	08867 Barney, 2007g [a]
08867/USA, Weslaco, TX/2005	SC	0.28	0.093	4	0	2.2, 3.1	2.7	[a]
08867/USA, Aurora, OR/2005	SC	0.28–0.29	0.165–0.166	4	0	5.4, 5.3	5.4	[a]

Turnip greens

One supervised trial on turnip greens was conducted in the USA in 2003. The trial was conducted in Texas and was reported by Gaydosh 2004c.

The treated plot received five foliar applications of the triflumizole 50WS formulation containing 501 g ai/kg. All applications were made at a rate of approximately 0.27 to 0.30 kg ai/ha per application. Applications were made on a 7-day schedule until turnip greens were ready to be harvested. The turnip greens were harvested 1, 3, and 7 days after the last treatment.

Specimens were stored frozen and shipped frozen to Morse Laboratories, Inc., Sacramento, California. Samples were processed with dry ice in a Hobart food grinder and stored frozen until analysis (< 4 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.05 mg eq/kg, and the LOD was not determined. Average recovery of all fortifications was 91 ± 12% (n=5). All control samples had residues at or below the limit of quantitation of the method. Turnip greens treated with the triflumizole 50WS formulation had detectable residues.

Table 57 Summary of triflumizole residue data in turnip greens

		Use Pattern						
Trial ID/Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue mg eq/kg)	Reference, remarks
USA GAP		0.28	n.s.	(5–10)	0	–	–	Label
RP-03004/USA, Uvalde, TX/2003	WS	0.27–0.30	0.100–0.127	5	Top			RP-03004
					1	4.3, 8.9, 8.1	7.1	Gaydosh, 2004c
					3	3.6, 2.9, 2.4	3.0	
					7	0.88, 0.78, 0.50	0.72	
					Root			
					1	0.15, 0.10, 0.15	0.13	
					3	0.13, 0.12, 0.11	0.12	
					7	0.098, 0.17, 0.08	0.12	

Tree nuts

Hazelnuts

A total of three supervised trials on hazelnuts (filberts) were conducted in the USA in 2001. Trials were conducted in Oregon. Trials were reported by Thompson 2002.

At each trial, plots received six foliar applications of the triflumizole 50WS formulation containing 500 g ai/kg. All applications were made at a rate of approximately 0.80 to 0.87 kg ai/ha per application. Applications were made on a 13-day to 111-day schedule until hazelnuts were ready to be harvested. The hazelnuts were harvested approximately 18 days after the last treatment.

Two samples of hazelnuts each were collected from one treated plot and one untreated plot. Specimens were stored frozen and shipped frozen to Morse Laboratories, Inc., Sacramento, California. Nutmeat samples were processed with dry ice in a Hobart food grinder and stored frozen until analysis (< 4 months).

The analytical method assayed triflumizole and its aniline-containing metabolites, with quantitation by GC/MSD. The LOQ was 0.05 mg eq/kg for triflumizole and for FA-1-1, and the estimated LOD was 0.01 mg eq/kg for triflumizole and 0.02 mg eq/kg for FA-1-1. Average recovery of all fortifications was 91 ± 8% (n=9) for triflumizole and 88 ± 9% (n=9) for FA-1-1. All control samples had residues below the limit of quantitation of the method. Hazelnuts treated with the triflumizole 50WS formulation had no detectable residues above the LOQ.

Table 58 Summary of triflumizole residue data in hazelnuts

		Use Pattern						
Trial ID/ Country/Year [a]	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.21	n.s.	(4–6)	18	–	–	Label
07996/USA, Aurora, OR/2001	WS	0.86–0.87	0.050–0.073	6	18	< 0.05, < 0.05	< 0.05	07996 Thompson, 2002
07996/USA, Aurora, OR/2001	WS	0.80–0.87	0.051–0.072	6	18	< 0.05, < 0.05	< 0.05	

		Use Pattern						
Trial ID/ Country/Year [a]	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg)	Average Residue (mg eq/kg)	Reference, remarks
07996/USA, Aurora, OR/2001	WS	0.84–0.86	0.051–0.073	6	19	< 0.05, < 0.05	< 0.05	

[a] Trials were conducted in the same orchard complex. One trial was on young, vigorous trees with large nuts, the second was on older, less vigorous trees with smaller nuts, and the third was in a different orchard block with different soil type and trees of an intermediate age.

Dried Herbs

Hops

A total of eight supervised trials on hops were conducted in the USA from 2002 to 2011, from three separate residue studies (#10798/RP-02011, 2006-008, and #10798). Trials were conducted in Idaho, Oregon, and Washington. Trials were reported by Corley 2012, Barney 2007b, and Barney 2007a.

Trials reported in Corley 2012 received three foliar applications of the triflumizole 480SC formulation containing 424.7 g ai/L. Trials reported by Barney 2007b received four foliar applications of the triflumizole 50WS formulation containing 503 g ai/kg. One of two trials reported by Barney 2007a received four foliar applications of the triflumizole 50WS formulation containing 509 g ai/kg, and the second trial received four foliar applications of the triflumizole 480SC formulation containing 430 g ai/L. All applications were made at a rate of approximately 0.42 to 0.58 kg ai/ha per application. Applications were made on a 10-day to 14-day schedule until hops were ready to be harvested. The hops were harvested 7 and 13–14 days after the last treatment (PHI=7, 13–14), except for the OR40 and WA 36 trials, which only had PHIs of 7, and the ID18 and WA35 trials, which was a decline trial with PHIs of 0, 3, 7–8, 14, and 21 days.

Samples of hops were collected from one treated plot and one untreated plot, except for the trial reported in Barney 2007a, which had two treated plots. Specimens were dried in a hop kiln (time not specified; ≤ 3–6 days based on transport dates) stored frozen and shipped frozen to IR-4 Western Region Laboratory, Davis, California, or Morse Laboratories, Inc., Sacramento, California. Samples were processed with a Geno/Grinder or with dry ice in a Rietz disintegrator. Samples were stored frozen from harvest until analysis (< 5 months Corley or < 27 months Barney).

For sample analysis, residue data from Corley 2012 were obtained using two separate analytical procedures, with one measuring triflumizole and FM-6-1 separately by LC-MS/MS, while the second method determined the total triflumizole equivalents by GC-MS (i.e., triflumizole and aniline-containing metabolites that were converted to FA-1-1, and expressed as triflumizole equivalents). The method used (Barney 2007a and Barney 2007b) also determined the total triflumizole by GC-MS. The LOQs were 0.050 mg eq/kg (Corley 2012), 0.01 mg eq/kg (Barney 2007b), or 0.02 mg eq/kg (Barney 2007a). Average recovery of all fortifications was 90.0 ± 18.1% (n=37) for Corley 2012 or 97.6 ± 5.1% (n=18) for Corley 2012, 81.5 ± 13.0% (n=6) for Barney 2007b and 79.2 ± 6.4% (n=6) for Barney 2007a. All control samples had residues at or below the limit of quantitation of the method. Hops treated with the 480SC formulation and the 50WS formulation had detectable residues.

Table 59 Summary of triflumizole residue data in dry hops

		Use Pattern						
Trial ID/Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg) A	Average Residue (mg eq/kg)	Reference, remarks
USA GAP		0.42	n.s.	3	7	–		Label
10798/USA, Parma, ID/2011 (New Port)	SC	0.42–0.43	0.075	3	0	10, 13	12	10798 Corley, 2012 Common/moiety

		Use Pattern						
Trial ID/Country/Year	Form.	Rate (kg ai/ha)	Rate (kg ai/hL)	No. of Appl.	DAT	Residue (mg eq/kg) A	Average Residue (mg eq/kg)	Reference, remarks
					3	10, 11	11	Common/moiety
					8	7, 6	6.5	Common/moiety
					14	8, 6	7	Common/moiety
					21	6.4, 5.3	5.9	Common/moiety
					0	6.5	6.5	Triflumizole + FM-6-1
					3	3.8	3.8	Triflumizole + FM-6-1
					8	1.7	1.7	Triflumizole + FM-6-1
					14	1.2	1.2	Triflumizole + FM-6-1
					21	0.9	0.9	Triflumizole + FM-6-1
10798/USA, Hubbard, OR/2011 (Nugget)	SC	0.43–0.44	0.082	3	7	12, 8.1	10.1	Common/moiety
					7	3.5	3.5	Triflumizole + FM-6-1
10798/USA, Prosser, WA/2011 (Nugget)	SC	0.43–0.45	0.027–0.028	3	0	18, 20	19	Common/moiety
					3	12, 13	13	Common/moiety
					7	12, 9.9	11	Common/moiety
					14	8.9, 8.3	8.6	Common/moiety
					21	6.6, 7	6.8	Common/moiety
					0	9.3	9.3	Triflumizole + FM-6-1
					3	4.4	4.4	Triflumizole + FM-6-1
					7	2.9	2.9	Triflumizole + FM-6-1
					14	1.5	1.5	Triflumizole + FM-6-1
					21	1.3	1.3	Triflumizole + FM-6-1
10798/USA, Prosser, WA/2011 (Willamette)	SC	0.42–0.44	0.024	3	7	7.6, 7.9	7.8	Common/moiety
					7	1.4	1.4	Triflumizole + FM-6-1
08967/USA, Zillah, WA/2002	WS	0.54–0.58	0.12	4	7	8.8, 6.4, 8.5	7.9	08967 Barney, 2007b
					14	7.1, 3.6, 7.4	6.0	
08967/USA, Woodburn, OR/2002	WS	0.56–0.57	0.12	4	6	16, 4.6	11	
					13	7.5, 6.4	7.0	
2006–2008/USA, Greenleaf, ID/2006 (Zeus)	WS	0.56–0.57	0.06	4	7	17, 26, 24	22	2006–2008 Barney, 2007a
					14	22, 21, 23	21	
2006–2008/USA, Greenleaf, ID/2006 (Zeus)	SC	0.56–0.57	0.06	4	7	17, 15, 14	15	
					14	14, 13, 12	13	

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received data depicting the effects of processing on residue levels in apple and grape processed commodities. For apples, processing factors have been calculated for juice, sauce, and wet and dry pomace. For grapes, processing factors have been calculated for juice, raisins (and related commodities), stems, and wet and dry pomace.

Apples

A processing study on apples was conducted from samples taken in three field trials located in commercial apple growing areas of the United States (Ball 1986c). In those trials, the treated plots each received four applications of triflumizole 50W at a rate of 0.56 kg ai/ha (0.5 lb ai/acre), which represents 1–2× the recommended label rate, or 1.12 kg ai/ha (1.0 lb ai/acre), which represents 2–4× the recommended label rate. Samples were collected 14 days after the last treatment. One sample was taken at each treated plot for processing into juice, sauce, wet pomace, and dry pomace. Processed commodities were produced by grinding and pressing, using a procedure that simulated commercial practice. Processed samples and a retained whole apple sample were shipped frozen to the laboratory, where the samples remained frozen until being assayed for total triflumizole via GC analysis.

The method was validated prior to analysis with a limit of quantitation of 0.02 mg eq/kg in all matrices. The performance of the method was also demonstrated during analysis of the samples by analysing fortified control matrices concurrently with experimental samples. The recoveries were 62% for whole unwashed apples, 125% for apple juice, 85% for apple sauce, 106% for wet pomace, and 96% for dry pomace. The overall mean concurrent recovery for all matrices was 95 ± 25% (n=5).

The results of the processing study are summarized in Table . Results indicate that residues did not concentrate in apple juice, sauce, or wet pomace, but was concentrated in dry apple pomace during processing.

Table 60 Processing factors for residues of triflumizole in apple processed fractions

Processed Fraction	Residues (mg eq/kg)		Processing Factors	Reference
	Apple (RAC)	Fraction		
Juice	< 0.02	0.04	Not determined	Ball 1986c (UR1302)
	0.16	0.04	0.25	
	0.24	0.05	0.21	
	0.18	0.04	0.22	
	0.34	0.08	0.24	
	Average		0.23	
Sauce	< 0.02	0.02	Not determined	
	0.16	0.02	0.13	
	0.24	0.02	0.08	
	0.18	0.05	0.28	
	0.34	0.31	0.91	
	Average		0.35	
Pomace, wet	< 0.02	0.18	Not determined	
	0.16	0.16	1.0	
	0.24	0.20	0.83	
	0.18	0.17	0.94	
	0.34	0.28	0.82	
	Average		0.90	
Pomace, dry	< 0.02	0.45	Not determined	
	0.16	0.50	3.13	
	0.24	0.62	2.58	
	0.18	0.10	0.56	
	0.34	0.13	0.38	
	Average		1.67	

Grapes

A processing study on grapes was conducted from samples taken in nine field trials located in commercial grape growing areas of the United States (Ball 1986b and Ball 1988c). Each field trial site consisted of control and treated plots. The treated plots each received four applications of triflumizole 50W at a rate of 0.28 kg ai/ha (0.25 lb ai/acre), which represents 1–2× the recommended label rate, except for the trials located in Portland, New York which received three applications.

Samples were collected 7 days after the last treatment. One sample was taken at each treated plot for processing into wet pomace, dry pomace, raisins (washed and unwashed), stems, and juice. Two samples were taken at the plot treated with triflumizole 50W at a rate of 0.56 kg ai/ha for processing into raisin waste. Samples were processed by grinding and pressing, using a procedure that simulated commercial practice. Processed samples and a retained whole grape sample were shipped frozen to the laboratory, where the samples remained frozen until analysis.

Combined residues of triflumizole and its aniline-containing metabolites were analysed in the different grape matrices using a validated analytical method with residue determinations made by HPLC (Ball 1986b) or GC (Ball 1988c). The method was validated prior to sample analysis. The performance of the method was also demonstrated during analysis of the samples by analysing fortified control matrices concurrently with experimental samples. The overall mean recoveries were 80–84% for whole grapes, 92–101% for wet pomace, 78–98.2% for dry pomace, 94% for raisins, 64% for stems, and 116% for juice. The overall mean concurrent recovery for all matrices was 88 ± 14% (n=11).

The results of the processing study are summarized in Table . Results indicate that residues in raisins (washed and unwashed), grape juice and raisin waste are reduced, but those in wet and dry grape pomace and grape stems were concentrated during processing.

Table 61 Processing factors for residues of triflumizole in grape processed fractions

Processed Fraction	Residues (mg eq/kg)		Processing Factors	Reference
	Grape (RAC)	Fraction		
Pomace, wet	0.09	0.07	0.78	Ball 1986b (UR1304)
	0.10	0.13	1.30	
	0.15	0.12	0.80	
	0.94	6.20	6.60	
	0.09	0.60	6.67	
	0.09	0.88	9.78	
	Average		4.32	
Pomace, dry	0.09	0.20	2.22	
	0.10	0.63	6.30	
	0.15	0.12	0.80	
	0.94	7.83	8.33	
	0.09	0.69	7.67	
	0.09	1.12	12.44	
	Average		6.29	
Raisins	0.18	0.03	0.17	
	0.15	0.04	0.27	
	Average		0.22	
Stems	0.18	0.68	3.78	
	0.15	0.38	2.53	
	Average		3.16	
Juice	0.94	0.24	0.26	
	0.09	0.03	0.33	
	0.09	0.06	0.67	
	Average		0.42	
Unwashed raisins	0.21	0.11	0.52	Ball 1988c (UR1308)
	0.31	0.07	0.23	
	Average		0.37	
Washed raisins	0.21	0.05	0.24	
	0.31	< 0.02	>0.06	
	Average		0.15	
Raisin Waste	0.21	< 0.02	>0.10	
	0.31	0.17, 0.22 (Avg.=0.20)	0.65	
	Average		0.37	

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received a lactating dairy cow feeding study (Parkins, 1988). The only crop with potential impact to the poultry dietary burden is cabbage; this crop represents 5% of the poultry diet in the EU only, which is of minimal impact. Therefore, a poultry feeding study has not been submitted.

Lactating dairy cows

Six dairy cows were dosed, for 28 consecutive days, orally via gelatin capsules containing technical triflumizole. The cows were randomly divided into two groups of three, each group receiving one of the following feeding levels: 10 ppm (low dose) and 50 ppm (high dose) of triflumizole in the diet. An additional two cows served as control animals. Samples of milk were taken each morning and evening. At the end of the experiment, the animals were sacrificed and samples of muscle, fat, entire liver and both kidneys were taken for analysis.

Samples from the low-dose group and the high-dose group were analysed for residues containing the 2-amino-3-trifluoro-methyl-4-chloroaniline (FA-1-1) moiety using gas chromatography with a nitrogen-phosphorus detector (GC-NPD). The limit of determination was 0.01 mg eq/kg. The total residues in milk and cattle tissues were reported as triflumizole (MW: 346) equivalents by converting the analysed FA-1-1 (MW: 195) level using a molecular weight conversion factor of 1.77. The reported LOQ in terms of triflumizole is 0.02 mg eq/kg.

For the high-dose group, the metabolite 2-amino-3-trifluoro-methyl-5-chlorophenol (FA-1-5) was also analysed using HPLC with electrochemical detection. Recoveries from milk fortified with either 0.01 mg eq/kg or 0.05 mg eq/kg FA-1-1 averaged 86.3% and 86.8%, respectively; for FA-1-5, concurrent recoveries in milk were 98%, 95% and 97% for spiked levels of 0.1, 0.2, and 0.3 mg eq/kg, respectively. For tissues, the average recoveries of FA-1-1 were 86% for fat, 93% for kidney, 73% for liver, and 85% for muscle. In the fortified samples with FA-1-5, the concurrent recovery in kidney and liver averaged 70%.

During the 28-day feeding period, residues analysed as FA-1-1 in milk samples from the low-dose group were < 0.01 mg eq/L. Residues analysed as FA-1-1 in milk from the high-dose group ranged from 0.01 mg eq/L to 0.06 mg eq/L. On average, residues were fairly consistent in milk after Day 2 of dosing (Table). Residues of FA-1-5 in milk (high-dose group) were < 0.01 mg/L.

Table 62 Total residues in milk (high-dose group)

Time (days)	Triflumizole-Equivalent Residues (mg/L) [a]	FA-1-5
0	< 0.02, < 0.02, < 0.02 [< 0.02]	< 0.01
2	0.04, 0.04, 0.04 [0.04]	< 0.01
4	0.04, 0.04, 0.02 [0.03]	< 0.01
6	0.05, 0.05, 0.04 [0.05]	< 0.01
8	0.04, 0.04, 0.05 [0.04]	< 0.01
10	0.04, 0.04, 0.05 [0.04]	< 0.01
14	0.04, 0.04, 0.02 [0.03]	< 0.01
16	0.04, 0.04, 0.02 [0.03]	< 0.01
18	0.05, 0.04, 0.05 [0.05]	< 0.01
20	0.05, 0.05, 0.07 [0.06]	< 0.01
22	0.04, 0.04, 0.02 [0.03]	< 0.01
26	0.04, 0.05, 0.04 [0.04]	< 0.01
28	0.04, 0.04, 0.11 [0.06]	< 0.01

[a] Bracketed values are mean

In tissues, average residues (as triflumizole-equivalents) in the low-dose group were < 0.02 mg eq/kg in muscle, 0.06 mg eq/kg in fat, 0.31 mg eq/kg in kidney, and 0.48 mg eq/kg in liver. In the high-dose group, average tissue residues were 0.09 mg eq/kg in muscle, 0.33 mg eq/kg in fat,

1.55 mg eq/kg in kidney, and 4.25 mg eq/kg in liver (Table 63). No residues of FA-1-5 were found in kidney or liver (LOD=0.03 mg eq/kg).

Table 63 Summary of feeding study results with triflumizole in dairy cattle

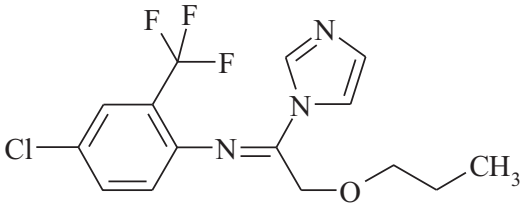
Matrix	Feeding Level (mg eq/kg feed)		
	0	10	50
	Triflumizole-Equivalent Residue (mg eq/kg) [a]		
Fat	< 0.02, < 0.02, < 0.02 [< 0.02 ± 0.00] {–}	0.02, 0.04, 0.12 [0.06 ± 0.05] {0.00560}	0.27, 0.25, 0.48 [0.33 ± 0.13] {0.00661}
Kidney	< 0.02, < 0.02, < 0.02 [< 0.02 ± 0.00] {–}	0.25, 0.21, 0.46 [0.31 ± 0.13] {0.03068}	1.7, 1.3, 1.6 [1.6 ± 0.20] {0.03103}
Liver	< 0.02, < 0.02, < 0.02 [< 0.02 ± 0.00] {–}	0.50, 0.46, 0.50 [0.48 ± 0.02] {0.04838}	3.9, 4.6, 4.2 [4.2 ± 0.35] {0.08496}
Muscle	< 0.02, < 0.02, < 0.02 [< 0.02 ± 0.00] {–}	< 0.02, < 0.02, < 0.02 [< 0.02 ± 0.00] {< 0.002}	0.09, 0.09, 0.11 [0.09 ± 0.01] {0.00189}
Milk	< 0.02–< 0.02 [< 0.02 ± 0.00] {–}	< 0.02–< 0.02 [< 0.02 ± 0.00] {< 0.002}	0.02–0.11 [0.04 ± 0.02] {0.00082}

[a] Values in brackets are mean ± std. dev.; values in braces are average matrix:feed ratios. For milk, the range of residues from Day 2 to Day 28 is shown.

APPRAISAL

Residue and analytical aspects of triflumizole were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2013 JMPR by the Forty-fourth Session of the CCPR.

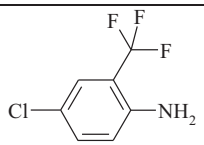
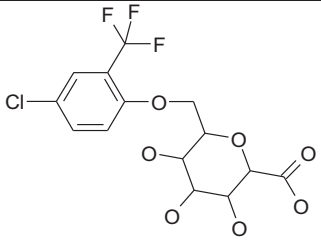
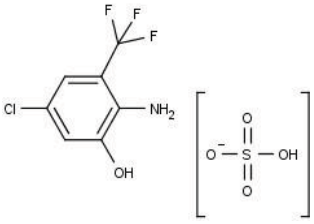
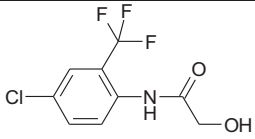
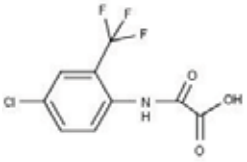
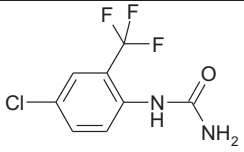
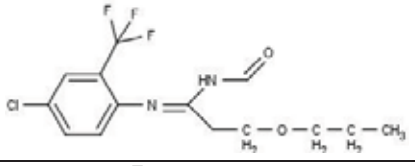
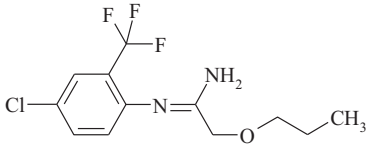
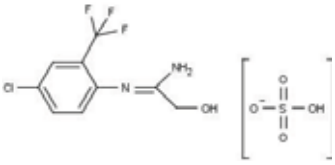
Triflumizole is a broad-spectrum foliar fungicide that controls a variety of fungal diseases in fruits and vegetables. It acts as a protectant and as an eradicant by preventing disease symptoms after infection has occurred. Triflumizole has anti-sporulant activity which reduces spores after lesions become visible. Triflumizole belongs to the demethylation inhibitor (DMI) group of fungicides classified as Group 3 by the Fungicide Resistance Action Committee (FRAC). Triflumizole has protective and curative action, and acts as an inhibitor of chitin biosynthesis. The product is mixed with water and applied as a foliar spray using ground equipment equipped for conventional spraying on crops. The Meeting received information on identity, animal and plant metabolism, environmental fates in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, dairy cattle feeding studies, and fates of residues in processing.

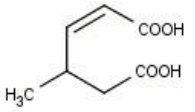
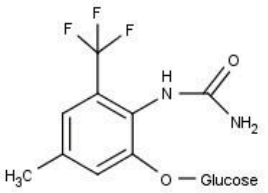


(E)-4-chloro-α,α,α-trifluoro-N-(1-imidazol-1-yl-2-propoxyethylidene)-o-toluidine

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

Identifier	Chemical Structure

Identifier	Chemical Structure
FA-1-1	
FA-1-glucuronide	
FA-1-5 (Free and sulphate conjugate)	
FD-2-1	
FD-7-1	
FD-9-1	
FM-5-1	
FM-6-1	
FM-8-1 (Free and sulphate conjugate)	

Identifier	Chemical Structure
XIV	
XXVI	

Animal metabolism

The Meeting received animal metabolism studies with triflumizole in rats, lactating goat and laying hens. The metabolism and distribution of triflumizole in animals were investigated using triflumizole universally labelled in the phenyl ring.

In the metabolism studies, rats were dosed at 10 mg/kg bw per day (single and multiple dose studies) or at 300 mg/kg bw per day (single dose study). Metabolite identification in tissues was not provided. The metabolite profile indicates that triflumizole is extensively metabolized in rats: less than 2% of the radiolabel recovered from urine or faeces was identified as parent compound. A few differences in metabolite pattern were observed between males and females after repeated low and single high doses, but not after a single low dose. The major urinary metabolites are the sulphate conjugates of N-(4-chloro-2-trifluoromethylphenyl)-2-hydroxy-acetamidine (FM-8-1) and 2-amino-5-chloro-3-trifluoromethylphenol (FA-1-5). In faeces, N-(4-chloro-2-trifluoromethylphenyl)-2-hydroxy-acetamide (FD-2-1) is a major metabolite. Differences between dose regimens exist with respect to other major metabolites. 2-(4-Chloro-2- trifluoromethylphenylimino)- 2-imidazol- 1-yl-ethanol (FM-2-1) is the major metabolite after a single oral low dose, 4-Chloro-2-trifluoromethylphenylamine (FA-1-1) is a major metabolite after single and repeated low dosing, whereas N-(4-chloro-2-trifluoromethylphenyl)-2-propoxy-acetamide (FD-1-1) is the major metabolite after a single high dose. The presence of the metabolite N-(4-chloro-2-trifluoromethylphenyl)- 2-propoxy-acetamidine (FM-6-1) was confirmed in rat faeces and urine.

Lactating goat was dosed with [^{14}C]triflumizole at a dose equivalent to approximately 280 ppm in the diet twice daily for 5 consecutive days. Gelatine capsules containing [^{14}C]triflumizole were administered orally via a balling gun. The majority of the dose was rapidly excreted in urine (56% of the TAR) and faeces (19.9% TAR).

Total radioactive residues (TRR) in the milk accounted for 0.18% TAR. Tissues contained approximately 0.65% TAR. Total material balance was 76.7% TAR, with an additional 5–12% TAR estimated to be in the bladder, digestive tract and carcass; $^{14}CO_2$ was not monitored.

Levels of radiolabel material reached a plateau in milk within approximately 24 hours and account for approximately 0.16% TAR. TRR was highest in liver and kidney (11.3 and 3.4 mg eq/kg, respectively) and approximately an order of magnitude lower in muscle (0.3 mg eq/kg) and fat (0.66 mg eq/kg). The residue in liver accounted for 0.4% TAR.

Triflumizole was not detected in any matrix. Major residues in liver were FA-1-1 (79.2% TRR, of which 54.8% TRR was bound) and FA-1-5-sulphate (12.4% TRR). In milk, major residues were FA-1-5-sulphate (29.1% TRR), FA-1-5-glucuronide (11.8% TRR), FD-2-1 (10.4% TRR), and FM-8-1-sulphate (12.6% TRR). No other individual compounds accounted for more than 10% of the TRR. The study noted that residues in other matrices were not identified due to low radioactivity; however, it is unclear why further characterization/identification was not done in muscle and liver given the level of residues in those tissues (0.3 and 0.66 mg eq/kg, respectively).

Laying hens were orally dosed with [^{14}C]triflumizole at a dose equivalent to 39 (3 hens) or 53 (2 hens) ppm in the diet twice daily for 5 consecutive days. The majority of the dose was rapidly eliminated in the excreta. Average radioactive residues in the excreta and cage wash accounted for 85.3% of the total administered dose.

Total ^{14}C residues in eggs did not plateau during the dosing period. Egg yolk and white were assayed separately for the eggs from hens treated at 53 ppm. Initially, TRR levels were greater in egg white than in the yolk; however, by Day 3 of dosing residues in yolk were much greater than in the egg white. On Day 5, residues in whole eggs from hens treated at 39 ppm ranged from 0.215 to 0.431 mg eq/kg. Residues ranged from 0.899 to 2.312 mg eq/kg in egg yolk and from 0.074 to 0.408 mg eq/kg in egg white. Residues in tissues from hens treated at the lower rate were consistently lower than those from hens treated at the higher rate. At the lower rate, residue ranges (mg eq/kg) were 0.989–1.232 in liver, 0.537–0.677 in kidney, 0.050–0.067 in fat, 0.017–0.047 in leg muscle, and 0.026–0.049 in breast muscle. At the higher rate, residues (mg eq/kg) were 0.781 and 1.115 in kidney, 0.312 and 0.358 in fat, 0.066 and 0.073 in leg muscle, and 0.054 and 0.087 in breast muscle; liver samples were used for metabolite identification and were not assayed for total radioactivity.

Major residues identified in egg white/yolk consisted of triflumizole (13.4/3.7% TRR), FD-6-1 or FD-4-1 (12.6/3.0% TRR), and FA-1-1 (35.3/22.1% TRR). Volatility of FA-1-1 resulted in some evaporative losses of that analyte. Unextracted residues accounted for 17.2 and 39.8% TRR in egg white and yolk, respectively. All other identified or characterized residues were less than approximately 4% TRR. Analysis of the liver samples indicated that the only metabolite was FA-1-1 or a compound convertible to FA-1-1 during proteolytic digestion.

In animal metabolism studies, triflumizole was metabolized to several compounds. The majority of the dosed radioactivity was excreted and radioactive residues in tissues were generally low. Triflumizole was identified as a residue in egg whites only. Metabolite FA-1-1 was the most prominent component in liver and egg. Sulphur- and glucuronide-conjugated FA-1-5 was the most prominent residue in milk.

Plant metabolism

The Meeting received plant metabolism studies performed on apple, pear, grape, and cucumber with triflumizole universally labelled in the phenyl ring.

In the apple metabolism study, trees were grown in a greenhouse and treated by spotting clusters of leaves with the test substance (approximately 0.127 mg/4-leaf cluster). Branches were harvested at 0, 1, 3, 7, 14, 21, 31, 60, and 90 DAT; fruits were not harvested.

Over the 90-day time span, the overall residue decreased from 51.65 mg eq/kg to 3.03 mg eq/kg, with an initial half-life of approximately 4 days, and was associated primarily with the treated leaves. Radioactive residue in the surface wash of treated leaves accounted for the majority of the residue until 14-DAT sampling. By the 14-DAT sampling, over half of the radioactivity in treated leaves was accounted for as extracted residues or associated with the post-extraction solids (PES). Radioactivity in untreated leaves above the treatment location remained relatively constant throughout the study, ranging from 0.04–0.07 mg eq/kg. Residues in untreated leaves below the treatment location showed a small increase in levels with time, with a maximum of 0.24 mg eq/kg at the 21-DAT sampling. In treated leaves, the only major (> 10% TRR) identified compounds that appeared consistently in the residue profile were triflumizole (0.02–0.12 mg eq/kg) and FM-6-1 (1.2–31% TRR, 0.01–0.02 mg eq/kg); FD-2-1 accounted for 12.6% TRR (< 0.01 mg eq/kg) at the 90-DAT sampling. As overall residues of radiocarbon declined with increasing time after treatment FD-2-1, unidentified metabolites and the contribution from PES became more prominent in the residue profile in terms of %TRR.

In the pear metabolism study, pear trees grown in a greenhouse were treated with test substance by spotting leaves (approximately 1 mg/4-leaf cluster) or the surface of pear fruits (approximately 0.166 mg/fruit) with radiolabelled test substance. Branches from leaf-treated trees

were harvested at 0, 1, 3, 7, 14, 21, 31, 60, and 90 DAT and were divided into treated leaves, untreated leaves, and fruit. Treated fruits were harvested 0, 1, 3, 7, and 14 DAT.

Data from the leaf-treatment samples showed very little movement of radioactivity away from the treated leaves. Radioactivity reached maxima of 0.98% TAR in untreated leaves and 0.18% TAR in untreated fruit. In treated leaves, the majority of the radioactivity was associated with the surface wash through the 31-DAT sampling, at which point extractable residues and PES-associated residues, combined, constitute more than half of the TRR (56% TRR, 5.6 mg eq/kg). As with the apple study, the data show a rapid decline in radiocarbon residues, with an initial half-life of approximately 4 days.

For treated fruit, the vast majority (> 95%) of the radioactive residue was confined to the peel, with very little movement into the flesh or core of the pear. For peel, residues in surface wash were the majority at the 0- and 1-DAT sampling times, after which extractable residues became the most prominent. The data also show a rapid decline in radiocarbon residues, with an initial half-life of between 1 and 3 days, and a slower rate of decline between 3 and 14 DAT.

In leaves, triflumizole and FM-6-1 were the only residues > 10% TRR. After 3 days and for the remainder of the study, FM-6-1 occurred at levels greater than triflumizole, peaking at 0.25 mg eq/kg at 3 DAT. In fruit, major residues were triflumizole (maximum = 93.56% TRR, 1.11 mg eq/kg, 0 DAT), FM-6-1 (maximum = 30.0% TRR, 0.14 mg eq/kg, 7 DAT) and FD-1-1 (10.51% TRR, 0.06 mg eq/kg, 3 DAT).

In the grape metabolism study, grape vines were grown in a greenhouse. Treatments were with triflumizole, uniformly labelled with ^{14}C in the phenyl ring, applied either to a branch of approximately 10 leaves and blossoms approximately 67 days prior to harvesting mature fruit, or to a bunch of young fruits 35 days before harvesting mature fruit. For both treatments, the rate was equivalent to 0.280 kg ai/ha. Treated branches were harvested 0, 3, 7, 14, 31, and 67 DAT and were divided into treated leaves and fruit; treated fruits were harvested 0, 3, 7, 14, and 35 DAT.

Following treatment of leaves, the % TAR decreased from 99% TAR to 15% TAR at 67 DAT. Most of the radioactivity in leaves was recovered from the surface wash, with < 2% TAR being translocated to fruits. Triflumizole was the major residue out to 31 DAT (51% TRR, 8.8 mg eq/kg). The only other major residue was FM-6-1 (11.1% TRR, 0.76 mg eq/kg at 67 DAT). Post-extraction residues in leaves gradually increased from 3.1% TRR (1.73 mg eq/kg) to 26.02% TRR (1.79 mg eq/kg) over the course of the study. Minor metabolites and those remaining at the origin increased to 39% TRR at 67 DAT, with no individual component exceeding 8% TRR.

In treated fruit over the 35-day period of the study, overall radioactivity decreased from 100% TAR (7.79 mg eq/kg) to 24.8% TAR (1.93 mg eq/kg). The majority of the radioactivity in treated fruit was recovered from the surface wash for the 0-, 3-, and 7-DAT samples; at the 14- and 35-DAT sampling points. More of the total residue was associated with extracted and bound residues. At all of the sampling times, the only major residue was triflumizole. FM-6-1 reached a maximum of 7.59% TRR (0.07 mg eq/kg) at 35 DAT, at which point triflumizole constituted 31% TRR (0.31 mg eq/kg).

The cucumber study was conducted under greenhouse conditions. Three types of treatment were made: Foliar treatment to investigate metabolism (0.13 mg/plant), fruit treatment to investigate metabolism (0.041 mg/plant), and foliar treatment to investigate translocation (0.16 mg/plant). In the case of the foliar treatment for metabolism, whole plants were harvested 1, 3, 7, 14, 21, and 45 days after treatment (DAT). For the fruit metabolism and translocation investigations, fruits were harvested 1 (metabolism only), 3, 7, and 14 DAT.

Results from all three lines of investigation demonstrated that the majority of the radioactivity (65–76% of applied) remained on the surface of the harvested material and that there was very little movement of radioactivity within the cucumber plants. Triflumizole accounted for most of the radioactivity through 14 DAT; at 21 DAT and 45 DAT, FM-6-1 residues were approximately equal to those of triflumizole and at 45 DAT, residues of F-8-1 were greater than either triflumizole or FM-6-1 on a % TRR basis. No metabolite exceeded 10% of the TRR except for FM-6-1approximately 12% TRR, < 0.01 mg eq/kg) at the 7- and 14-DAT sampling times in the cucumber fruit study. In those samples, FM-6-1 was associated with the peel and flesh of the fruit and not the surface wash.

The metabolic profile observed in these studies was similar across all four crops. Over the time course of the studies, triflumizole and FM-6-1 account for the majority of the radioactivity. Generally triflumizole decreased rather rapidly (half-life on the order of 3 days), with the exception of grapes where metabolism was slower (half-life approximately 14 days). Loss of triflumizole was accompanied by an increase in the FM-6-1 metabolite, which was the only metabolite to consistently occur at ≥ 10% TRR.

Environmental fate in soil

The Meeting received information on soil-surface photolysis, soil dissipation, aerobic soil metabolism, and confined and field rotational crop studies.

Under aerobic conditions in soil, the imidazole ring of triflumizole is opened to yield FM-5-1, FD-1-1, and FM-6-1. The resulting amide or amine moiety is then hydrolysed to form the FA-1-1 metabolite, which then becomes associated with bound residues or is oxidized to CO_2. The DT_{50} was 16–22 days at 20 °C with indoor studies and 7.1–22 days with outdoor studies.

In the soil photolysis study, triflumizole degraded significantly. On average after 11 days of exposure, triflumizole had declined to 30.0% of the AD. This was accompanied by formation of FM-6-1, which reached a maximum of 21.8% TAR after 7 days of exposure to simulated sunlight and then dissipated to 15.6% TAR at Day 11. The metabolites FD-1-1 (3.6% TAR at Day 11), FM-8-1 (1.8% TAR, Day 11), FD-7-1 (8.6% TAR, Day 11), and CO_2 (3.5% TAR, Day 11) were also observed. Bound residues increased over the time course of the study and averaged 18.7% TAR by the end of the 11-day period.

The results of both the aerobic soil and soil photolysis study indicate that triflumizole is not likely to be persistent in the environment.

In the confined rotational crop study, radiolabelled triflumizole was applied to bare soil at approximately 1.4 kg ai/ha (1× GAP; 6 plots) or approximately 12.7 kg ai/ha (10× GAP; 2 plots) and rotational crops of lettuce, radish, and wheat were planted into each. Plant-back intervals (PBIs) were 30, 120, and 365 days for the 1× plots and 30 days for the 10× plots. Radish was sampled for roots and tops, and wheat was sampled for forage, grain, hay, and straw. The 1X plots were used to determine % TRR only and the 10× plots were used to make metabolite identifications (except for 1× samples for wheat forage).

Quantifiable levels of TRR occurred for all crop matrices at all PBIs. Highest values were for wheat straw, starting at 1.65 mg eq/kg at the 30-day PBI and declining to 0.478 mg eq/kg at the 365-day PBI. The lowest values were for lettuce (0.086 to 0.021 mg eq/kg at the 120- and 365-day PBI, respectively). For each crop matrix, the TRR declined when going from shorter to longer PBIs with the exception of lettuce which had a maximum at the 120-day PBI (0.086 mg eq/kg) and wheat grain for which the 365-day PBI TRR (0.067 mg eq/kg) was greater than the 120-day PBI TRR (0.055 mg eq/kg).

Triflumizole underwent extensive metabolism in the confined rotational crop study. Forty-nine metabolites were identified, four of which were > 10% TRR and > 0.01 mg eq/kg in at least one matrix. No triflumizole was identified in any crop and no single metabolite was consistently the predominant residue. Two of the major metabolites were identified as glucose conjugates of FM-6-1 and FM-8-1, which constituted 22.9% TRR in lettuce and 16.1% TRR in wheat forage, respectively. The other major metabolites in the confined rotational crop study (XIV and XXVI) were not significant residues in either the crop or livestock metabolism profiles.

Field rotational crop studies were conducted with lettuce, turnip, cabbage, cotton, onion, tomato and wheat. Triflumizole was applied to squash at approximately 30 days prior to harvest or to cucumbers at the vining stage. In both studies, the initial application was followed by four subsequent applications at 7 ± 1 day intervals. All applications were 0.28 kg ai/ha for a total rate of 1.4 kg ai/ha. For all commodities except wheat fodder, forage, and hay, residues of triflumizole, analysed by the common-moiety method, were < 0.01 mg/kg, or in a few cases slightly above 0.01 mg/kg, at plant-back intervals (PBIs) of 30, 60, 90, 120, 180, and 270 days (not all crops were sampled at all

intervals). In wheat animal forage, hay, and straw, quantifiable residues persisted throughout all of the plant-back intervals with no particular trend related to PBI. Residue ranges were < 0.01–0.20 mg/kg in wheat forage, < 0.01–0.144 mg/kg in wheat hay, and < 0.01–0.134 mg/kg in wheat straw.

The field rotational crop study indicates that residues are generally not expected (< 0.01 mg/kg) in food crops planted in rotation following treatment to the previous crop. When residues do occur, they are not expected to be much above 0.01 mg/kg. In contrast, readily quantifiable residues in forage/fodder, hay, and straw commodities from cereal grains are expected when such crops are planted in rotation following a treated.

Methods of analysis

The Meeting received description and validation data for analytical methods for residues of total triflumizole (all residues convertible to the FA-1-1 metabolite, expressed as triflumizole) as well as methods for specific residues (e.g., triflumizole, FM-6-1, FA-1-5). Recovery data were provided for raw and processed agricultural commodities as well as animal commodities.

Methods that analyse total triflumizole in crops involve an initial extraction with water. Hydrolysis is accomplished by refluxing in concentrated acetic acid and sodium acetate followed by distillation in the presence of sodium hydroxide into hexane. Residues are cleaned up by column chromatography, and analysed by GC-NPD, GC-MSD, or HPLC-UV.

Methods for the analysis of specific residues in crops use solvent extraction (methanol or acetonitrile), clean-up by solid-phase partitioning and liquid/liquid partitioning, and analysis by GC-NPD, GC-MSD, HPLC-UV, or LC-MS/MS.

Recoveries for the common-moiety method averaged 80.8% and recoveries for the specific-residue method averaged 86.5%. Recoveries for both methods were generally within the range of 70–120%. Limits of quantitation are reported as being of 0.01 mg/kg for most matrices, 0.02 mg/kg for hops, and 0.05 mg/kg for nutmeats.

Data describing multi-residue method testing for residues of triflumizole were not provided.

In animals, the common-moiety method uses 20% NaOH and a Bleidner extractor to digest/distil/extract residues into hexane. The terminal residue is the FA-1-1 metabolite and analysis is by GC-NPD. Recoveries for this method ranged from 78 to 92%.

In the procedure for determining residues containing FA-1-5, the sample was extracted using an acidic digestion followed by clean up and concentration using solid phase extraction (SPE) on reversed phase C18 columns. Final separation and quantification was conducted by HPLC on a C18 column and electrochemical detection. Recoveries for this method averaged 85%.

All of the submitted methods are adequate for the analysis of triflumizole residues.

Stability of residues in stored analytical samples

The Meeting received data on the stability of residues of triflumizole and its metabolites in crops (apple, grape, strawberry, cucumber, cherry, muskmelon, squash, lettuce, and wheat forage) and livestock. The test compound was added to homogenized test matrix. Samples were placed into frozen storage and analysed by the common-moiety or analyte-specific method(s) used in the supervised residue trial.

For the following commodities, stability of residues during storage was demonstrated for at least 3 months in hazelnut, 4 months in wheat forage, 6 months in animal commodities, 12 months in cherries and grapes, and 18 months in papaya.

For the following commodities, stability of residues during storage was demonstrated for no longer than 2 months in lettuce, 3 months in muskmelon and summer squash, and 6 months in apple, cucumber, and strawberry.

For the following commodities, stability of residues during storage was not demonstrated for any time period: cabbage, mustard greens, Swiss chard, and tomato.

Definition of the residue

In the lactating goat metabolism study, TRR were significantly higher in liver than in any other matrix, including milk. Triflumizole was not detected in liver or milk. In liver, free and conjugated FA-1-1 constituted nearly 80% of the residue, with FA-1-5-sulphate making up an additional 12%. In milk, sulphate and glucuronide conjugates of FA-1-5 account for 40% of the residue; the only other major residues were FD-2-1 (10.4% TRR) and FM-8-1-sulphate (12.6% TRR).

In the laying hen metabolism study, TRR were highest in liver, kidney, and egg. Liver contained only FA-1-1 (or a metabolite converted to FA-1-1 during proteolytic digestion). In egg, major residues were triflumizole (13.4% TRR in whites), FD-6-1 or FD-4-1 (12.6% TRR in white), and FA-1-1 (up to 35% TRR in white and 22% TRR in yolk). Hen kidney was not subjected to analysis for residue identification.

In the lactating cattle feeding study, samples were analysed using a common-moiety method, which would have captured all of the major residues observed in the lactating goat and laying hen metabolism studies except for FA-1-5-sulphate, which was the major metabolite in milk observed in the metabolism study. However, in the feeding study, residues of FA-1-5-sulphate were < 0.01 mg/kg in milk and < 0.03 mg/kg in liver and kidney.

Analytical methods for animal matrices are available for the analysis of residues convertible to FA-1-1 or for analysis of FA-1-5-sulphate.

The Meeting agreed that residues converted to 4-chloro-2-(trifluoromethyl)aniline (FA-1-1) and expressed as parent triflumizole are suitable for enforcement and dietary risk purposes in animal commodities.

The log P_{OW} of the residues in animal commodities will be determined by the composition and relative amounts of the metabolite residues in the various matrices. In the lactating cattle feeding study, total residues of triflumizole in fat were, on average, approximately 3.5 times greater than those in muscle.

The Meeting concluded that the residue is fat soluble.

In metabolism studies with apple (leaves only), pear, cucumber, and grape, triflumizole and FM-6-1 constitute the vast majority of the identified residues, and no other residues consistently occur at greater than 10% TRR. The FM-6-1 metabolite was observed in the rat.

The analytical method for plant matrices is a common-moiety method that quantifies all residues convertible to FA-1-1, including FM-6-1.

The Meeting agreed that residues converted to 4-chloro-2-(trifluoromethyl)aniline (FA-1-1) and expressed as parent triflumizole are suitable for enforcement and dietary risk purposes in plant commodities.

The Meeting recommended the following residue definition:

For plants and animals:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake):

Residues analysed as 4-chloro-2-(trifluoromethyl)aniline and expressed as parent triflumizole.

Results from supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of triflumizole on apple, pear, cherry, grape, strawberry, papaya, broccoli, cabbage, cucumber, muskmelon, squash, tomato, lettuce (head and leaf), mustard green, Swiss chard, turnip green, hazelnuts and hops. Residue trial data were made available from the USA for all crops as well as Japan (cucumber, apple and pear), Netherlands (cucumber and tomato), and Belgium (tomato).

Labels for end-use products containing triflumizole were available from the USA and the Netherlands (glasshouse use on tomato and cucumber only) describing the registered uses of triflumizole.

For most crops, the residues were determined by the common-moiety method and reflect the sum of all residues convertible to the aniline moiety, expressed as triflumizole. For the trials in Japan (cucumber), the Netherlands (cucumber and tomato) and Belgium (tomato), the reported residues are the sum of triflumizole and FM-6-1. Side-by-side data reflecting analysis by both common-moiety and residue-specific (triflumizole + FM-6-1) methods are available for hops. In those samples, residues from the common-moiety method were considerably greater than those from the residue-specific method. As the residue definition is for residues convertible to the FA-1-1 metabolite, only residues determined as total triflumizole are suitable for making residue estimates.

Pome fruits

The GAP in the USA on pome fruit is foliar application at a maximum rate of 0.56 kg ai/ha and a PHI of 14 days. Applications may be made at a 7- to 10-day interval with a maximum seasonal rate of 2.24 kg ai/ha.

Apple

Data were available from supervised trials on apple in the USA. Data were also provided from Japan; however, there is no GAP in Japan.

Eighteen supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. Using trials with a supported storage interval, the Meeting selected the following data for consideration (n=3): < 0.02, 0.26, 0.30 mg/kg.

The meeting agreed that the data for apples were insufficient to make a recommendation.

Pear

Data were available from supervised trials on pear in the USA. Data were also provided from Japan; however, there is no GAP in Japan.

Seven supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. Using trials with a supported storage interval, the Meeting selected the following datum for consideration (n=1): 0.28 mg/kg.

The Meeting agreed that the data for pear were insufficient to make a recommendation.

Cherries

The GAP in the USA on cherry is foliar application at a maximum rate of 0.56 kg ai/ha and a PHI of 1 day. Applications may be made at a 7- to 14-day interval with a maximum seasonal rate of 3.36 kg ai/ha.

Eight supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. The residue results are supported by the available storage stability data. The trials resulted in the following eight independent residue values: 0.59, 0.96, 1.1, 1.1, 1.2 (2), 1.3, 1.5 mg/kg.

The Meeting estimated a maximum residue level, STMR, and HR for triflumizole residues in cherries (Subgroup 003A) of 4 mg/kg, 1.2 mg/kg, and 1.5 mg/kg, respectively.

Berries and other small fruits

The GAP in the USA on grapes and strawberries is foliar application at a maximum rate of 0.28 kg ai/ha and a PHI of 7 days for grapes and 1 day for strawberry. Applications may be made at a minimum interval of 14 days, with a maximum seasonal rate of 1.12 kg ai/ha.

Grape

Nineteen supervised trials were conducted in the USA at GAP for grapes and residues were obtained by the common-moiety method. The residue results are supported by the available storage stability data. The trials resulted in the following 12 independent residue values: 0.09, 0.1, 0.15, 0.16, 0.18, 0.21, 0.31, 0.50, 0.90, 0.94, 1.4, and 2.0 mg/kg.

Based on these data, the Meeting estimated a maximum residue level, STMR, and HR for triflumizole residues in grapes of 3 mg/kg, 0.26 mg/kg, and 2.0 mg/kg, respectively.

Strawberry

Eight supervised trials were conducted in the USA. The residue data for strawberry are not supported by the available storage stability data.

The Meeting agreed not to make a recommendation for strawberry.

Papaya

The GAP in the USA on papaya is foliar application at a maximum rate of 0.35 kg ai/ha on a 14-day interval, with a PHI of 0 days. The maximum seasonal rate is 0.84 kg ai/ha.

Four supervised trials were conducted in the USA. Each treated plot received five applications at approximately 0.42 kg ai/ha on a 12- to14-day interval for a total rate of approximately 2.1 kg ai/ha. Papaya were harvested 0 DAT.

The Meeting agreed that although application in the papaya field trials reflects an over dosing relative to GAP due to the number of applications, the retreatment interval (12 to14 days) combined with residue decline and the rapid growth of the papaya fruit which would be occurring during the first two applications, the early applications are unlikely to have contributed significantly to the residue level at harvest. Therefore, the available trials are suitable for estimation of residue levels resulting from GAP applications. The residue data (n=4) are: 0.22, 0.53, 0.88, and 0.89 mg/kg.

Based on the trials for papaya in the USA, the Meeting estimated a maximum residue level, STMR, and HR for triflumizole residues in papaya of 2 mg/kg, 0.71 mg/kg, and 0.89 mg/kg, respectively.

Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassica

The GAP in the USA on Brassica vegetables is foliar application at a maximum rate of 0.28 kg ai/ha, at a 14-day interval, and a PHI of 1 day. The maximum seasonal rate is 0.63 kg ai/ha.

Broccoli

Ten supervised trials were conducted in the USA. The residue data for broccoli are not supported by the available storage stability data.

The Meeting agreed not to make a recommendation for broccoli.

Cabbage

Nine supervised trials were conducted in the USA. The residue data for cabbage are not supported by the available storage stability data.

The Meeting agreed not to make a recommendation for cabbage.

Fruiting vegetables, Cucurbits

The GAP in the USA on cucurbit vegetables is foliar application at a maximum rate of 0.28 kg ai/ha at a 7- to 14-day interval, and a PHI of 0 days. The maximum seasonal rate is 1.4 kg ai/ha.

The GAP in the Netherlands on cucumber and summer squash is foliar application at a maximum rate of 0.225 kg ai/ha and a PHI of 1 day. One to six applications may be made per season at a 7-day interval.

Cucumber-Outdoor Crops

Six supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. The residue results supported by the available storage stability data are as follows (n=4): 0.13 (2), 0.17, and 0.18 mg/kg.

The Meeting agreed that the data from field-grown cucumber were insufficient to make a recommendation.

Cucumber-Indoor Crops

Four supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. The residue results supported by the available storage stability data are as follows (n=3): 0.10, 0.11, and 0.21 mg/kg.

An additional four trials were conducted in the Netherlands according to the Netherlands GAP. The trials measured only triflumizole and FM-6-1; therefore the residues did not match the residue definition.

The Meeting agreed that the data from indoor-grown cucumber were insufficient to make a recommendation.

Residues from the indoor and outdoor trials on cucumbers from the USA matching the USA GAP are from the same population (Mann-Whitney U test). Using the combined data set (n=7): 0.10, 0.11, 0.13 (2), 0.17, 0.18 and 0.21 mg/kg, the Meeting estimated a maximum residue level, STMR, and HR for triflumizole residues in cucumber of 0.5 mg/kg, 0.13 mg/kg, and 0.21 mg/kg, respectively.

Melon

Six supervised trials were conducted on muskmelon in the USA at GAP. The residue data for muskmelon are not supported by the available storage stability data.

The Meeting agreed not to make a recommendation for muskmelon.

Summer squash

Five supervised trials were conducted in the USA at GAP at GAP and residues were obtained by the common-moiety method. The residue data for summer squash are not supported by the available storage stability data.

The Meeting chose not to make a recommendation for summer squash.

Tomato

The GAP in the USA on tomato is foliar application at a maximum rate of 0.28 kg ai/ha at a 7- to 14-day interval and a PHI of 0 days. The maximum seasonal rate is 1.4 kg ai/ha. The USA GAP is the Critical GAP.

Four supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. The residue data for tomato are marginally supported by the storage stability data and are as follows (n=4): < 0.5 (2), 0.59, and 0.76 mg/kg.

The Meeting agreed that the data for tomato from the USA were insufficient to make a recommendation.

The GAP in the Netherlands on tomato is foliar application at a maximum rate of 0.015 kg ai/hL and a PHI of 1 day. One to five applications may be made per season at a 7-day interval.

Four supervised trials were conducted in each of the Netherlands and Belgium. Treated plot received three applications each at 0.016 kg ai/hL on a 7-day interval and fruits were harvested 3 DAT.

The trials in the Netherlands and Belgium were conducted according to the Netherlands GAP. The Netherlands trials measured only triflumizole and FM-6-1; therefore the residues do not reflect the residue definition.

The Meeting agreed not to make a recommendation for tomato.

Leafy vegetables (including Brassica leafy vegetables)

The GAP in the USA on leafy vegetables is foliar application at a maximum rate of 0.28 kg ai/ha at a 14-day interval and a PHI of 0 days. The maximum seasonal rate is 0.63 kg ai/ha.

Lettuce

Data were available from supervised trials on lettuce in the USA.

Seventeen supervised trials were conducted in the USA on lettuce (head lettuce = seven field trials; leaf lettuce = seven field trials + two greenhouse trials; unspecified = one field trial). In 16 trials, treated plot received four applications each at approximately 0.28 kg ai/ha on a 6- to 9-day interval. The total application rate was approximately 1.12 kg ai/ha. In the remaining trial on the unspecified lettuce variety, five applications were made at approximately 0.57 kg ai/ha, for a total rate of approximately 2.85 kg ai/ha. Lettuce was harvested 0 DAT.

The Meeting agreed that in both the number and interval of applications, the residue trials for lettuce did not match the GAP for lettuce in the USA, and that the proportionality concept could not be used.

The Meeting agreed not to make a recommendation for lettuce.

Mustard greens

Ten supervised trials were conducted in the USA on mustard greens at GAP. The residue trials are not supported by the available storage stability data.

The Meeting agreed not to make a recommendation for mustard greens.

Swiss chard

Data were available from supervised trials on Swiss chard in the USA at GAP. The residue trials are not supported by the available storage stability data.

The meeting agreed not to make a recommendation for Swiss chard.

Turnip greens

The GAP in the USA for triflumizole on turnip greens is foliar application at a maximum rate of 0.28 kg ai/ha and a PHI of 1 day. Two to three applications may be made per season at a 14-day interval with a maximum seasonal rate of 0.63 kg ai/ha.

In one trial conducted in USA, where five applications of 0.27–0.3 kg ai/ha triflumizole were made on a 7-day schedule, total residues in turnip greens 1 day after the last application were 7.1 mg/kg.

The Meeting agreed that the supporting data for turnip greens did not match the GAP in USA and are of insufficient quantity. The Meeting agreed not to make a recommendation for turnip greens.

Hazelnuts/Filberts

The GAP in the USA on hazelnut is foliar application at a maximum rate of 0.21 kg ai/ha and a PHI of 18 days. Four to six applications may be made per season at a 10- to 14-day interval with a maximum seasonal rate of 0.84 kg ai/ha.

All of the field trials in the USA (n=3) were conducted at an exaggerated rate of 6.25× GAP. Per-trial average triflumizole residues (via common moiety) were < 0.05 mg/kg for all samples. The Meeting noted that all three trials were conducted in the same year and in the same orchard complex.

The Meeting noted that the trials are not independent and agreed not to make a recommendation for hazelnuts.

Hops

The GAP in the USA on hops is foliar application at a maximum rate of 0.42 kg ai/ha and a PHI of 7 days. Three applications may be made per season at a 14-day interval with a maximum seasonal rate of 1.26 kg ai/ha.

Data were available from supervised trials on hops in the USA.

Four supervised trials were conducted in the USA at GAP and residues were obtained by the common-moiety method. The residue results are supported by the available storage stability data. The trials resulted in the following 4 independent residue values for dried hops: 7.0, 7.8, 10, and 11 mg/kg.

Based on the trials for hops in the USA, the Meeting estimated a maximum residue level, STMR, and HR for triflumizole residues in hops, dried of 30 mg/kg, 8.9 mg/kg, and 11 mg/kg, respectively.

Fate of residues during processing

The Meeting received data depicting the effects of processing on residue levels in apple (juice, sauce, and wet/dry pomace) and grape (juice, raisins (and related commodities), stems, and wet/dry pomace). The estimated processing factors for grape commodities are 0.42 for juice; 0.22 for grape, dried; and 4.3 for wet pomace.

The Meeting estimated an STMR and HR for grapes of 0.41 mg/kg and 2.0 mg/kg, respectively. Application of the estimated processing factors results in an estimated STMR-P and HR-P, respectively, of 0.06 mg/kg and 0.44 mg/kg for grape, dried; an STMR-P of 0.17 mg/kg for grape juice; and an STMR-P of 1.2 mg/kg for wet grape pomace.

Residue in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of triflumizole 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 MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities.

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). The diets are based on residues in wet grape pomace.

Livestock dietary burden, triflumizole, ppm of dry matter diet [a]

	US-Canada	EU	Australia	Japan

	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	–	–	–	–	1.49 [a]	1.49 [c]	–	–
Dairy cattle	–	–	–	–	1.49 [b]	1.49 [d]	–	–
Poultry—broiler	–	–	–	–	–	–	–	–
Poultry—layer	–	–	–	–	–	–	–	–

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

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

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

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

Farm animal feeding studies

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

Lactating dairy cows

Lactating dairy cows were dosed with triflumizole for 28 days at the equivalent of 10 or 50 ppm in the diet. Analysis was for residues convertible to FA-1-1 and FA-1-5 (high-dose group only). Residues for all analyses and tissues were < 0.02 for control animals.

From the high-dose group, milk samples from even-numbered treatment days (except Day 24) were analysed. Residues of FA-1-5 were < 0.01 mg/kg in all milk samples and < 0.03 mg/kg in liver and kidney, which were the only tissue analysed for this compound. Total triflumizole residues were < 0.02 mg/kg in milk on Day 0 and were then rather consistent, ranging from, on average, 0.03 to 0.06 mg/kg. In the low- and high-dose tissue samples, respectively, average residues were 0.06 to 0.33 mg/kg in fat, 0.31 to 1.55 mg/kg in kidney, 0.48 to 4.25 mg/kg in liver, < 0.02 to 0.94 in muscle, and < 0.02 to 0.04 mg/kg in milk.

Animal commodities maximum residue levels

For MRL estimation, the residue in the animal commodities is residues analysed as 4-chloro-2-(trifluoromethyl)aniline and expressed as parent triflumizole.

Residues in tissues and milk at the expected dietary burden for beef cattle in Australia are shown in the Table below. The residue in milk was relatively consistent from Day 2 through Day 28 and the mean estimated residue in milk was calculated using the residue values from Day 4 to the final day.

Triflumizole feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg) in			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study [a]	10 50	< 0.02 0.041	10 50	< 0.02 0.106	0.496 4.602	0.460 1.717	0.115 0.478
Dietary burden and high residue	1.49	< 0.02	1.49	< 0.02	0.07	0.69	0.03
STMR beef or dairy cattle							
Feeding study [b]	10 50	< 0.02 0.041	10 50	< 0.02 0.094	0.484 4.248	0.307 1.552	0.056 0.330
Dietary burden and residue estimate	1.49	< 0.02	1.49	< 0.02	0.072	0.046	0.008

[a] Highest residues for tissues and mean residues for milk

[b] Mean residues for tissues and mean residues for milk

The Meeting estimated the following maximum residue levels: Milk = 0.02(*) mg/kg, meat = 0.03 (fat) mg/kg and edible offal = 0.1 mg/kg.

The Meeting estimated the following STMR levels: Milk = 0 mg/kg, meat = 0 mg/kg, fat = 0.008 mg/kg and edible offal = 0.072 mg/kg.

The Meeting estimated the following HR levels: Milk = 0mg/kg, meat = 0 mg/kg, fat = 0.017 mg/kg and edible offal = 0.074 mg/kg.

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 assessments.

Plant and animal commodities:

Definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary intake): *Residues analysed as 4-chloro-2-(trifluoromethyl)aniline and expressed as parent triflumizole.*

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		
FS 0013	Cherries	4	1.17	1.5
VC0424	Cucumber	0.5	0.13	0.21
FB 0269	Grapes	4	0.41	2.0
FI 0350	Papaya	2	0.71	0.89
DH 1100	Hops, Dried	30	8.9	11
ML 0106	Milks	0.02(*)	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.05 (fat)	0 Muscle 0.008 Fat	0 Muscle 0.017 Fat
MO 0105	Edible Offal (mammalian)	0.2	0.072	0.072
JF 0269	Grape, Juice	Covered by grapes	0.11	
	Grape, pomace (wet)	–	1.2	
DF 0269	Dried grapes (=currants, raisins and sultanas)	Covered by grapes	0.06	0.44

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of triflumizole were calculated for the 13 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (see Annex 3 of

the 2013 JMPR Report). The ADI is 0–0.04 mg/kg bw and the calculated IEDIs were 0–2% of the maximum ADI (0.04 mg/kg bw). The Meeting concluded that the long-term intakes of residues of triflumizole, resulting from the uses considered by the current JMPR, are unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of triflumizole 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 2013 JMPR Report). The ARfD is 0.3 mg/kg bw and the calculated IESTIs were ≤ 100% of the ARfD for all commodities. The Meeting concluded that the short-term intake of residues of triflumizole, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
UR 1305	Ball, JO	1986a	Analytical Methodology for Determining the Magnitude of Triflumizole Residue in Food Crops. Uniroyal Chemical Company, Inc. Report No. UR 1305. Unpublished.
UR1304	Ball, JO	1986b	Magnitude of the Residue in Grapes (fresh & processed). Uniroyal Chemical Company, Inc. Report No. UR1304. Unpublished.
UR1302	Ball, JO	1986c	Magnitude of the Residue of Triflumizole in Apples (fresh and processed). Uniroyal Chemical Company, Inc. Report No. UR1302. Unpublished.
UR1303	Ball, JO	1986d	Magnitude of the Residue of Triflumizole in Pears. Uniroyal Chemical Company, Inc. Report No. UR1303. Unpublished.
RP-88036	Ball, JO	1988a	Magnitude of the Residue: Storage Stability of Residues of Triflumizole and Its Metabolites in Apples and Grapes. Uniroyal Chemical Company, Inc. Report No. RP-88036. Unpublished.
UR1306	Ball, JO	1988b	Magnitude of the Residue Triflumizole in Apples. Uniroyal Chemical Company, Inc. Report No. UR1306. Unpublished.
UR1308	Ball, JO	1988c	Magnitude of the Residue Triflumizole in Grapes. Uniroyal Chemical Company, Inc. Report No. UR1308. Unpublished.
UR1307	Ball, JO	1988d	Magnitude of the Residue Triflumizole in Pears. Uniroyal Chemical Company, Inc. Report No. UR1307. Unpublished.
RP-88036 Addendum 1	Ball, JO	1989	Storage Stability of Triflumizole and Three of Its Metabolites in Frozen Apples and Grapes—12 Month Interval. Uniroyal Chemical Company, Inc. Report No. RP-88036 Addendum 1. Unpublished.
08865	Barney, WP	2006	Triflumizole: Magnitude of the Residue on Mustard Greens. Rutgers, The State University of NJ. Report No. 08865. Unpublished.
2006-008	Barney, WP	2007a	Procure® 50WS and Procure® 480 SC on Hops: Magnitude of the Residue Study. Chemtura Corporation. Report No. 2006-008. Unpublished.
08967/RP-02011	Barney, WP	2007b	Procure® 50WS on Hops: Magnitude of the Residue Study. Rutgers, The State University of NJ. Report No. 08967/RP-02011. Unpublished.
09319	Barney, WP	2007c	Triflumizole: Magnitude of the Residue on Broccoli. Rutgers, The State University of NJ. Report No. 09319. Unpublished.
09143	Barney, WP	2007d	Triflumizole: Magnitude of the Residue on Cabbage. Rutgers, The State University of NJ. Report No. 09143. Unpublished.
08993	Barney, WP	2007e	Triflumizole: Magnitude of the Residue on Lettuce (Head and Leaf, Field and Greenhouse). Rutgers, The State University of NJ. Report No. 08993. Unpublished.
09332	Barney, WP	2007f	Triflumizole: Magnitude of the Residue on Papaya. Rutgers, The State University of NJ. Report No. 09332. Unpublished.
08867	Barney, WP	2007g	Triflumizole: Magnitude of the Residue on Swiss Chard. Rutgers, The State University of NJ. Report No. 08867. Unpublished.
09299	Barney, WP	2012	Triflumizole: Magnitude of the Residue on Tomato (Greenhouse). Rutgers, The State University of NJ. Report No. 09299. Unpublished.
8945	Batorewicz, W	1990	Storage Stability Study: Determination of Stability of Triflumizole, FA-1-1, and FA-1-5 in Cow Tissue and Milk: Freezer Storage Stability of FA-1-5 Sulfate Ester in Beef Liver and Milk. Uniroyal Chemical Company, Inc. Report No. 8945. Unpublished.

Code	Author	Year	Title, Institute, Report reference
9196	Batorewicz, W	1991	Amendment to Final Report, Project No. 9196: Triflumizole (Procure). Extended Freezer Storage Stability of FA-1-5 Sulfate Ester in Beef Liver and Milk. Uniroyal Chemical Company, Inc. Report No. 9196. Unpublished.
9019	Batorewicz, W & Bakker, RW	1990	Analytical Method—Triflumizole (Procure)—Determination of FA-1-5 Metabolite Residues in Meat and Milk. Uniroyal Chemical Company, Inc. Report No. 9019. Unpublished.
10798	Corley, J	2012	Triflumizole: Magnitude of the Residue on Hops. IR-4 Project Headquarters. Report No. 10798. Unpublished.
UR-02-89	Eckert, J	1989	Analysis of FA-1-1 in Cow Muscle and Milk from 2, 4, and 6 Hour Refluxes. Uniroyal Chemical Company, Inc. Report No. UR-02-89. Unpublished.
UR-01-89	Eckert, J	1990	Freezer Storage Stability Analysis of Triflumizole (A-815) and Metabolite FA-1-1 in Cow Tissues and Milk. Uniroyal Chemical Company, Inc. Report No. UR-01-89. Unpublished.
GRL-FR-11321	Friedlander, BT	1998	Determination of the Oxidizing and Reducing Characteristics of Procure 50 WG. Uniroyal Chemical Co. Report No. GRL-FR-11321. Unpublished.
RP-97007	Gaydosh, K	1999	Procure®50WS on Squash: Magnitude of Residue Study. Uniroyal Chemical Company, Inc. Report No. RP-97007. Unpublished.
RP-97005	Gaydosh, K, Maselli, C & Puhl, J	1999	Procure® 50WS on Muskmelon: Magnitude of the Residue Study. Uniroyal Chemical Company, Inc. Report No. RP-97005. Unpublished.
RP-95012	Gaydosh, KA	1997	Procure 50WS® on Sweet Cherries: Magnitude of the Residue Study. Uniroyal Chemical Company, Inc. Report No. RP-95012. Unpublished.
RP-98023	Gaydosh, KA	2000a	Residue Analytical Method Validation for Triflumizole and its Metabolites in Wheat, Turnip, and Lettuce. Uniroyal Chemical Company, Inc. Report No. RP-98023. Unpublished.
RP-98017	Gaydosh, KA	2000b	Procure 50WS Rotational Crop Study: Procure 50WS Treated Cucumbers Rotated with Wheat, Lettuce, and Turnips 30, 60, 90, and 180 Days After Harvest. Uniroyal Chemical Co., Inc. Report No. RP-98017. Unpublished.
RP-99005	Gaydosh, KA	2001	Procure® 50WS on Strawberries: Magnitude of the Residue Study. Uniroyal Chemical Company, Inc. Report No. RP-99005. Unpublished.
RP-03006	Gaydosh, KA	2004a	PROCURE® 50WS in Broccoli: Magnitude of the Residue Study. Crompton Corp. Uniroyal Chemical. Report No. RP-03006. Unpublished.
RP-03005	Gaydosh, KA	2004b	PROCURE® 50WS in Lettuce: Magnitude of the Residue Study. Crompton Corp. Uniroyal Chemical. Report No. RP-03005. Unpublished.
RP-03004	Gaydosh, KA	2004c	PROCURE® 50WS in Turnip: Magnitude of the Residue Study. Crompton Corp. Uniroyal Chemical. Report No. RP-03004. Unpublished.
F-93-30-NL-00/02	Geuijen, I	1994	Determination of the magnitude of residues triflumizole on the raw agricultural commodity cucumber, from Rocket EC by ground-equipment in the Netherlands (field phase). Certis. Report No. F-93-30-NL-00/02. Unpublished.
EC-326; RD-9118	Gomyo, T	1991	Analytical Method for Total Content of Triflumizole and Its Degradates in Water by Gas Chromatography and Gas Chromatography Mass Spectrometry. Nippon Soda Co., Ltd. Report No. EC-326; RD-9118. Unpublished.
RD-9235	Gomyo, T	1992	Residue Analysis of Triflumizole in Pome Fruits. Nippon Soda Co., Ltd. Report No. RD-9235. Unpublished.
RD-9331	Gomyo, T	1993	The Results of the Supervised Residue Trials of Triflumizole on Cucumber in Japan. Nippon Soda Co., Ltd. Report No. RD-9331. Unpublished.
EC-311; RD-9112	Gomyo, T, Morishima, Y & Ono, S	1990	Analytical Method for Individual and Total Content of Residues of Triflumizole Fungicide and Its Metabolites in Crops by High Performance Liquid and Gas Chromatography. Nippon Soda Co., Ltd. Report No. EC-311; RD-9112. Unpublished.
EC-322; RD-9114	Gomyo, T, Morishima, Y, Tachibana, T, Kobayashi, S & Ono, S	1991	Analytical Method for Residues of Triflumizole Fungicide and Its Main Metabolite in Crops by High Performance Liquid and Gas Chromatography. Nippon Soda Co., Ltd. Report No. EC-322; RD-9114. Unpublished.
EC-323; RD-9115	Gomyo, T, Morishima, Y, Yamazaki, S, Dairiki, H & Ono, S	1991	Analytical Method for Total Content of Residues of Triflumizole Fungicide and Its Metabolites in Crops by High Performance Liquid and Gas Chromatography. Nippon Soda Co., Ltd. Report No. EC-323; RD-9115. Unpublished.
84253	Harned, WH,	1985	Aerobic Soil Metabolism study of [^{14}C]A815 in Sandy Loam Soil.

Code	Author	Year	Title, Institute, Report reference
	Tortora, NJ & Sullivan, EA		Uniroyal Chemical. Report No. 84253.
RD-9862	Higashida, S & Tanaka, T	1998	Partition Coefficient (n-octanol/water) of Triflumizole. Nippon Soda Co., Ltd. Report No. RD-9862. Unpublished.
09300	Homa, K	2012	Triflumizole: Magnitude of the Residue on Greenhouse Cucumber. Rutgers, The State University of NJ. Report No. 09300. Unpublished.
TL-6362, RD-8841	Iguchi, K	1988a	Triflumizole—Density, bulk density or specific gravity. Nippon Soda Co., Ltd. Report No. TL-6362, RD-8841. Unpublished.
TL-6361, RD-8840	Iguchi, K	1988b	Triflumizole—Melting Point. Nippon Soda Co., Ltd. Report No. TL-6361, RD-8840. Unpublished.
RD-II02245	Knight, LJL	2002	Triflumizole—Photodegradation in Water. Nippon Soda Co., Ltd. Report No. RD-II02245. Unpublished.
8517-A	Knipe, JO	1986	Further Analysis of the Metabolism of (^{14}C)-A815 (Triflumizole) in a Lactating Goat. Uniroyal Chemical Company, Inc. Report No. 8517-A. Unpublished.
8517	Knipe, JO & Harned, WH	1985	The Metabolism of (^{14}C)-A815 in a Lactating Goat. Uniroyal Chemical Company, Inc. Report No. 8517. Unpublished.
8635	Knipe, JO & Lengen, MR.	1986	The Disposition of (^{14}C)-A815 (Triflumizole) in Chickens. Uniroyal Chemical Company, Inc. Report No. 8635. Unpublished.
2010-020	Marin, JE	2011	Storage Stability of Triflumizole Metabolites in Plant Matrices. PTRL Report No. 2122W, Chemtura Report No. 2010-020. Unpublished.
RP-95013	Maselli, CV	1997	Procure 50WS on Tart Cherries: Magnitude of the Residue. Uniroyal Chemical Company, Inc. Report No. RP-95013. Unpublished.
RP-97006	Maselli, CV	1999	Procure® 50WS on Cucumbers: Magnitude of the Residue Study. Uniroyal Chemical Company, Inc. Report No. RP-97006. Unpublished.
EC-99, RD-8762	Nomura, O & Nakashima, N	1987	Triflumizole—Octanol/Water Partition Coefficient. Nippon Soda Co., Ltd. Report. No. EC-99, RD-8762. Unpublished.
20	Noon, P	1993	Method Validation: Determination of Residues of 2-Hydroxy-4-Chloro-6-Trifluoromethylaniline, FA-1-5, and Its Conjugates in Meat and Milk. Uniroyal Chemical Company, Inc. Report No. 20.028: 9308. Unpublished.
8772	Parkins, MD	1988	Triflumizole Cow Feeding Study. Uniroyal Chemical Company, Inc. Report No. 8772. Unpublished.
9308: EPA3/EH/H701/3	Parkins, MD	1993	Proposed Enforcement Procedure for the Determination of Conjugates of 2-hydroxy-4-chloro-6-trifluoromethylaniline, FA-1-5, in Liver and Milk. Uniroyal Chemical Company, Inc. Report No. 9308: EPA3/EH/H701/3. Unpublished.
8659	Parkins, MD & Polakoff, BM	1986	Methods for Analyzing Meat, Milk and Egg Residues Resulting from Feed Containing Triflumizole (A815). Uniroyal Chemical Company, Inc. Report No. 8659. Unpublished.
94181	Pierce, JP	1994	Calculation of Henry's Law Constant. Uniroyal Chemical Company. Report No. 94181. Unpublished.
2006/004, 1512W	Ponte, M	2006	Photodegradation of [14 Carbon] Triflumizole in/on Soil by Simulated Sunlight: Final Report. PTRL West, Inc. Report No. 2006/004, 1512W. Unpublished.
RP-03008	Puhl, J	2005	ProcureÒ 50WS Treated Squash Rotated with Wheat, Onion, Tomato, Cabbage, and Cotton: Rotational Crop Study. Crompton Corporation. Report No. RP-03008. Unpublished.
RD-01201	Rijsbergen, L	2002	Determination of the Vapour Pressure of TRIFLUMIZOLE. Nippon Soda. Co., Ltd. Report No. RD-01201.
EC-211, RD-9004	Saito, H	1990	Triflumizole—Solubility in Organic Solvents. Nippon Soda Co., Ltd. Report No. EC-211, RD-9004. Unpublished.
V 94	Schouten, .	1994	The determination of residues of triflumizole (NF-114) and its main metabolite (FM-6-1) in cucumber by high performance liquid chromatography. Certis. Report No. V 94.326. Unpublished.
RD-8246	Shiotani, H	1982	Fate of ^{14}C-NF-114 in Soil. Nippon Soda Co., Ltd. Report No. RD-8246. Unpublished.
ML-1, RD-8359	Soeda, Y	1983	Fate of NF-114 in Cucumber Plants. Nippon Soda Co., Ltd. Report No. ML-1, RD-8359. Unpublished.
RD-8845	Soeda, Y	1988	Triflumizole—Oxidizing or reducing. Nippon Soda Co., Ltd. Report No. RD-8845. Unpublished.
EC-80, RD-8708	Soeda, Y & Hashimoto, Y	1987a	Triflumizole Fate in Pear Plants. Nippon Soda Co., Ltd. Report No. EC-80, RD-8708. Unpublished.
EC-81, RD-8709	Soeda, Y & Hashimoto, Y	1987b	Triflumizole Fate in Apple Plants. Nippon Soda Co., Ltd. Report No. EC-81, RD-8709. Unpublished.
EC-82, RD-8710	Soeda, Y & Hashimoto, Y	1987c	Triflumizole Fate in Grape Plants. Nippon Soda Co., Ltd. Report No. EC-82, RD-8710. Unpublished.

Code	Author	Year	Title, Institute, Report reference
EC-129, RD-8833	Soeda, Y & Nomura, O	1988	Triflumizole—Solubility in Organic Solvents. Nippon Soda Co., Ltd. Report No. EC-129, RD-8833. Unpublished.
EC-111	Soeda, Y & Shiotani, H	1987a	Triflumizole—Hydrolysis. Nippon Soda Co., Ltd. Report No. EC-111. Unpublished.
EC-113, RD-8783	Soeda, Y & Shiotani, H	1987b	Triflumizole—Photodegradation in Water. Nippon Soda Co., Ltd. Report. No. EC-113, RD-8783. Unpublished.
EC-130, RD-8834	Soeda, Y & Shiotani, H	1988	Triflumizole—Dissociation Constant. Nippon Soda Co., Ltd. Report No. EC-130, RD-8834. Unpublished.
RD-II02419	Tanaka, T	2002	Validation of Analytical Method for Technical Grade Triflumizole. Nippon Soda Co., Ltd. Study No. 1-0206; Report No. RD-II02419. Unpublished.
07996	Thompson, DC	2002	Triflumizole: Magnitude of the Residue on Filbert. Rutgers, The State University of NJ. Report No. 07996. Unpublished.
91469	Tutty, DG	1994	Determination of the Solubility of Triflumizole in Water at 25 °C. Uniroyal Chemical Ltd. Report No. 91469. Unpublished.
GRL-FR-11330	Tutty, DG	1998	Thermal Explodability of Procure 50WG. Uniroyal Chemical Co. Report No. GRL-FR-11330. Unpublished.
RD-8225N	Unknown	1983	Physical and Chemical Properties of NF-114. Nippon Soda Co., Ltd. Report No. RD-8225N. Unpublished.
RD-8409N	Unknown	1984	Physical and Chemical Properties of NF-114 Technical. Nippon Soda Co., Ltd. Report No. RD-8409N. Unpublished.
3096040731	van de Ruit, ANR	1997	Field residue study of Rocket EC (a.i.: 150 g/L triflumizole) in greenhouse tomatoes in the Netherlands and Belgium. Certis. Report No. 3096040731. Unpublished.
98201	Yu, WC, Dzialo, DG, Whitright, RR & Regis, RR	2001	Confined Accumulation Study on Rotational Crops with ^{14}C-Triflumizole (Procure®). Uniroyal Chemical Company, Inc. Report No. 98201. Unpublished.