TRIFLUMURON (317)

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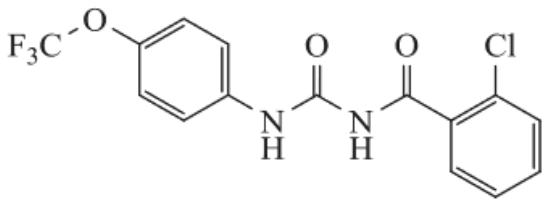
EXPLANATION

Triflumuron is a benzoylurea insecticide. It is an insect growth regulator which acts by inhibiting the synthesis of chitin in insect larvae that are about to moult by interfering with the moulting hormone system. It is registered on a variety of crops worldwide, including fruit trees, potatoes, vegetables, mushrooms, soya beans, cotton, cereals, tobacco and ornamentals. It also has pest control uses against termites, silverfish, fleas and cockroaches, and veterinary uses against lice and flies. Triflumuron has never been evaluated by JMPR.

At the Fiftieth Session of CCPR (2018) triflumuron was scheduled for consideration by the 2019 JMPR as a new compound for the use in soya bean. The Meeting received the data for triflumuron on plant and animal metabolism, environmental fate, methods of analysis, use pattern, residues resulting from supervised trials in soya bean, fate of residues in storage and processing and residues in animal products.

IDENTITY

Table 1 Identity

ISO common name	Triflumuron
IUPAC name	1-(2-chlorobenzoyl)-3-[4-trifluoromethoxyphenyl]urea
Chemical Abstract name	2-chloro-N-[[[4-(trifluoromethoxy)phenyl]amino]carbonyl]benzamide
CAS No.	64628-44-0
CIPAC No.	548
Synonyms	AE F067232, SIR8514, Alsystin®
Molecular formula	$C_{15}H_{10}ClF_3N_2O_3$
Structural formula	
Molecular weight	358.7 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

Table 2 Pure active ingredient (except where noted as technical grade)

Property	Results	Test material purity	Reference
Triflumuron			
Melting point	194 °C (1 atm)	99.6%	Olenik, B., 2008 (M-300905-01-1)
Boiling point	254 °C Decomposition: >254 °C	99.6%	Olenik, B., 2008 (M-300905-01-1)
Relative density	1.55 g/mL (at 20 °C)	99.8%	Gruener, R., 2001(M-091466-01-1)
Vapour pressure	Extrapolated $< 10^{-3}$ Pa at 20.0 °C $< 10^{-3}$ Pa at 25.0 °C $< 10^{-3}$ Pa at 50.0 °C	99.8%	Olf, 2002(M-052058-01-1)

Property	Results	Test material purity	Reference
	1.73×10^{-2} Pa at 112.09 °C 8.86×10^{-1} Pa at 146.51 °C		
Description of the physical state, colour and odour, purity of the ai, and of technical grade	Colourless to white crystalline powder Odourless	99.8%	Stoecker, R., 2001(M-041424-01-1)
	Colourless to white crystalline powder Odourless	99.2%	Stoecker, R., 2001(M-041424-01-1)
Solubility of purified ai in water	0.04 mg/L (at 20 °C)	99.8%	Schneider, J., 2002(M-060752-01-1)
Solubility in organic solvent	Acetone: 22.6 g/L Acetonitrile: 4.5 g/L Dichloromethane: 11.7 g/L Dimethyl sulfoxide: 127.4 g/L Ethyl acetate: 23.3 g/L n-Heptane: < 0.1 g/L 1-Octanol: 1.2 g/L Polyethyeneglycol: 9.6 g/L 2-Propanol: 1.3 g/L Xylene: 1.7 g/L (at 20 °C)	99.8%	Gruener, R., 2001(M-091466-01-1)
n-Octanol / water partition coefficient (log P_{ow})	3.6 at 10 °C 3.5 at 20 °C 3.5 at 30 °C	99.6%	Eyrich, U., Ziemer, F., 2012(M-429618-01-1)
Direct phototransformation in sterile water using artificial light	DT_{50} (Half-life at 25 °C) At pH 7: 32.8 days The only major degradation product is 2-chlorobenzamide (M01). Under environmental conditions solar radiation does not significantly contribute to the degradation of triflumuron in aqueous solution.	^{14}C-Chlorophenyl ring-AE F067232, Radiochemical purity: > 99% ^{14}C-Trifluorophenyl ring-AE F067232, Radiochemical purity: > 99%	Hellpointner, Kloeppner, 2003(M-088318-01-1
Dissociation in water of purified active ingredient	Triflumuron shows no basic properties in aqueous systems. It is not possible to specify a pK value of Triflumuron in water	99.5%	Placke, F.J., 1987(M-025794-01-2

Specifications

Specifications for triflumuron have been developed by JMPS in FAO Specifications 548 / TC (July 2018).

Table 3 Technical material

Chemical/physical property	Results
Minimum purity	Triflumuron not less than 955 g/kg
Melting range	Not specified
Stability	Not specified
Physical state, colour	Colourless powder

Formulations

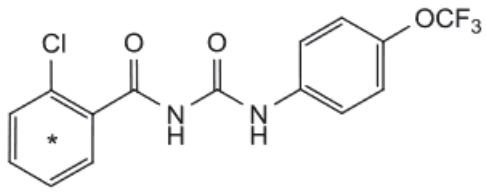
Various triflumuron formulations are available in the following products.

Table 4 Formulations

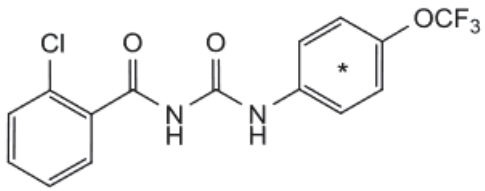
Purpose	ai content	Type
Crop protection	480 g ai/L	SC
	25%	WP
Seed treatment	4 g/L 150 g/L triadimenol	LS
	4 g/L 150 g/L triadimenol	LS
Veterinary use	2.5%	PO

METABOLISM AND ENVIRONMENTAL FATE

The studies on plant metabolism (apples, tomatoes, potatoes and soya bean) and confined rotational crops, as well as animal metabolism (rat, lactating goats and laying hens) were conducted with the test material shown with the label position indicated in Figure 1.



Ring 1 label:
[2-chlorophenyl-UL-^{14}C] triflumuron

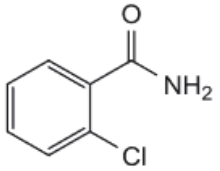
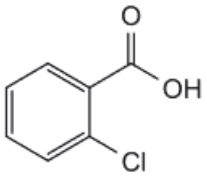


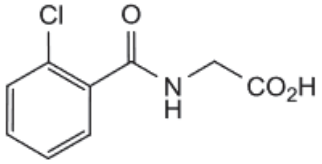
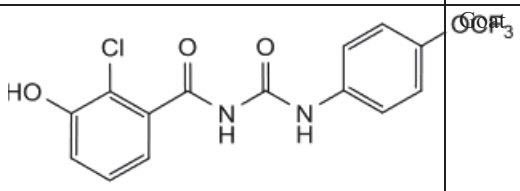
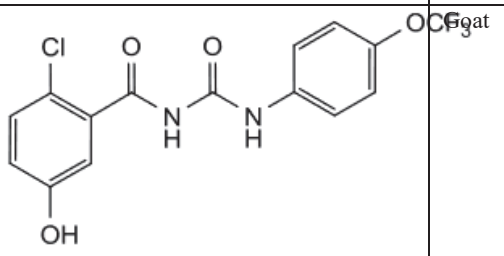
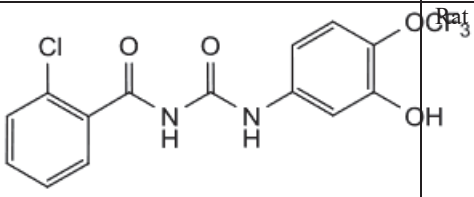
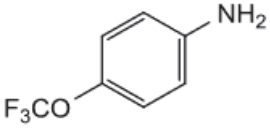
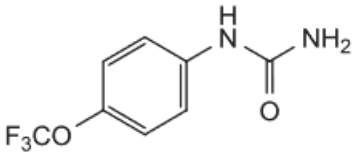
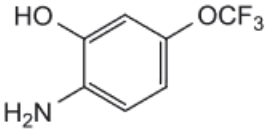
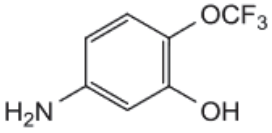
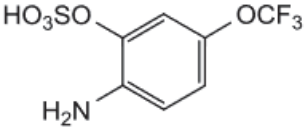
Ring 2 label:
[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron
[4-trifluoromethoxyaniline-UL-^{14}C] triflumuron
[phenoxy-UL-^{14}C] triflumuron

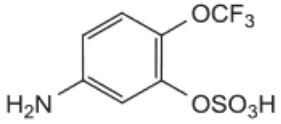
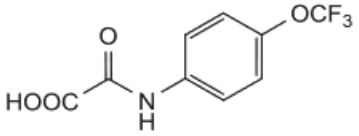
Figure 1 Radiolabelled test materials used in the metabolism studies

Table 5 summarizes the names, codes, and structures of the parent and principal metabolites found in plant, livestock, rat, rotational crop and environmental studies.

Table 5 Triflumuron and metabolites/degradates found in metabolism and environmental fate studies

Compound code number, chemical name, synonym	Structure	Found in
M01 2-Chlorobenzamide AE F092117		Soya bean, potato Rotational crop Rat, goat, hen
M02 2-Chlorobenzoic acid AE F091982		Soya bean, potato Rotational crop Rat, hen

Compound code number, chemical name, synonym	Structure	Found in
M03 2-Chlorohippuric acid		Rat, goat
M04 1-(2-chloro-3-hydroxybenzoyl)-3-[4-trifluoromethoxyphenyl]urea SIR 8514-3-hydroxy-2-chlorophenyl		Goat
M05 1-(6-chloro-3-hydroxybenzoyl)-3-[4-trifluoromethoxyphenyl]urea SIR 8514-5-hydroxy-2-chlorophenyl		Goat
M06 1-(2-chlorobenzoyl)-3-[4-trifluoromethoxy-3-hydroxyphenyl]urea SIR 8514-3-hydroxyaniline		Rat
M07 4-Trifluoromethoxyaniline AE F069069		Soya bean, potato Goat (as conjugates), rat
M08 4-Trifluoromethoxyphenyl urea AE 1111065		Soya bean, potato Rat, goat
M09 2-Hydroxy-4-trifluoromethoxyaniline		Rat, goat (as conjugates)
M10 3-Hydroxy-4-trifluoromethoxyaniline		Rat
M16 2-amino-5-trifluoromethoxybenzensulfonic acid		Rat

Compound code number, chemical name, synonym	Structure	Found in
M17 3-amino-6-trifluoromethoxybenzensulfonic acid		Rat
M19 4-trifluoromethoxy-oxalanilide		Rat

PLANT METABOLISM

The Meeting received information on metabolism of triflumuron using [2-chlorophenyl-UL-^{14}C] triflumuron or [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Figure 1) in apples, tomatoes, soya beans and potatoes. In the following texts, TRR is expressed in mg-triflumuron equivalents/kg.

Apple (Moellhoff, 1985, M-137273-01-2)

Translocation study

Two greenhouse experiments to investigate the translocation of triflumuron in apple trees and shoot cuttings were available (Moellhoff, 1985, M-137273-01-2).

In the first experiment, shoots (10 cm long) were cut from an apple tree (Morgenduft, 1 year old) and immediately placed into a beaker filled with an aqueous solution of [2-chlorophenyl-UL-^{14}C] triflumuron or [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron at the concentration of between 0.07 and 0.12 mg ai/L. The beakers were wrapped in aluminium foil and the water was replenished to a volume of 25 mL. The shoots had been kept at 20 ± 2 ºC for 13 days. Cuttings of apple seedlings over a 13-day period absorbed 7.7% for [2-chlorophenyl-UL-^{14}C]-triflumuron and 7.5% for [4-trifluoromethoxy-phenyl-UL-^{14}C]-triflumuron.

In the second greenhouse experiment, ten leaves of apple trees (Morgenduft, 1 year old) were topically treated with 0.02–0.03 mg of [2-chlorophenyl-UL-^{14}C] triflumuron or 0.03–0.04 mg of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron as aqueous solution. After 13 days, these leaves and the stem section on which they had been growing were harvested, and the leaves and the stem of the upper third and the stem of the bottom third. In the experiments, 0.05% of the applied radioactivity was translocated into upper plant parts over a 13-day period. There was no downward movement of radioactivity.

Based on these studies, triflumuron was not absorbed extensively into the peel and pulp as between 90% and 99% of the recovery of radioactivity was found in the acetone wash of the apples. Degradation of triflumuron on the surface was not observed.

Metabolism study

The metabolism of triflumuron under outdoor conditions was studied on apples (James Grieve, 5 or 6 years old). Each apple plant was grown in conical 90-L containers with a top diameter of 55 cm and protected from rain by a transparent canopy.

Either the [2-chlorophenyl-UL-^{14}C] or [4-trifluoromethoxy-phenyl-UL-^{14}C] solution (10 g ai/L for first year and 1 or 0.2 g ai/L for second year) were directly topically applied to apples using a pipette in two consecutive years at 20-30 mg ai/kg (first year) and 0.15–0.26 mg ai/kg or 0.89-1.04 mg ai/kg (second year).

Apples (three for each sampling time) were harvested 5, 11, 19, 24, (27) and 31 days after treatment (DAT) for the first year and 7, 14, 21, 28, 32 and 35 DAT for the second year.

Apples were dipped in acetone for two hours to remove the dip and then washed with acetone. The washing solutions were combined for each crop fraction for quantifying radioactivity and analysis of metabolites. The washed apples were peeled. The peels were homogenized with acetone and filtered. The filtrates were evaporated and extracted with chloroform. The fruit pulp was extracted with acetone and filtered, and then the aqueous residue was extracted with chloroform.

The radioactivity in the surface wash and acetonitrile extracts was determined by liquid scintillation counting (LSC). The surface wash was analysed by HPLC.

Low penetration into peel and pulp was observed as most of radioactivity was found in the acetone surface wash of the apples (89.9–99.2% of total recovered radioactivity). The distribution of radioactivity in the surface wash solution, peel and pulp is summarized in Table, 6, 7, 8 and 9.

Table 6 Distribution of radioactivity in apples following a single application of [2-chlorophenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 10 g ai/L)

Days after application	[2-chlorophenyl-UL-^{14}C] triflumuron Application solution concentration approximately 10 g ai/L						
	Acetone wash		Peel		Pulp		total
	%AR	% TRR	% AR	% TRR	%AR	% TRR	% AR
5	89.1	99.0	0.59	0.66	0.29	0.32	90.0
11	88.6	98.9	0.84	0.94	0.17	0.19	89.6
14	89.1	99.2	0.56	0.62	0.16	0.17	89.8
19	84.8	98.8	0.87	1.01	0.16	0.19	85.8
24	83.2	98.7	0.81	0.96	0.30	0.35	84.3
31	77.7	98.8	0.81	1.03	0.17	0.21	78.7

Table 7 Distribution of radioactivity in apples following a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 10 g ai/L)

Days after application	[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron Application solution concentration approximately 10 g ai/L						
	Acetone wash		Peel		Pulp		total
	%AR	% TRR	%AR	% TRR	%AR	% TRR	%AR
5	86.6	99.2	0.57	0.65	0.11	0.13	87.3
11	76.2	98.9	0.63	0.82	0.18	0.23	77.0
14	82.6	99.0	0.59	0.71	0.27	0.32	83.5
19	91.4	98.6	0.88	0.95	0.45	0.49	92.7
24	70.1	98.2	0.92	1.29	0.38	0.53	71.4
27	79.9	97.9	1.03	1.26	0.72	0.88	81.7
31	88.0	98.5	0.84	0.94	0.50	0.56	89.3

Table 8 Distribution of radioactivity in apples following a single application of [2-chlorophenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) and 1 g ai/L (day 32))

Days after application	[2-chlorophenyl-UL-^{14}C] triflumuron Application solution concentration approximately 0.2 g ai/L (days 7-28 and 35) and 1 g ai/L (day 32)						
	Acetone wash		Peel		Pulp		total
	%AR	% TRR	%AR	% TRR	%AR	% TRR	%AR
7	81.8	98.2	1.16	1.39	0.38	0.46	83.3

Days after application	[2-chlorophenyl-UL-^{14}C] triflumuron Application solution concentration approximately 0.2 g ai/L (days 7-28 and 35) and 1 g ai/L (day 32)						
14	57.3	94.6	2.66	4.39	0.63	1.04	60.6
21	66.1	93.8	3.30	4.68	1.08	1.53	70.5
28	57.6	90.3	4.81	7.54	1.36	2.13	63.8
32	70.1	93.6	3.55	4.74	1.28	1.71	74.9
35	63.0	91.5	3.72	5.40	2.17	3.15	68.9

Table 9 Distribution of radioactivity in apples following a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) and 1 g ai/L (day 32))

Days after application	[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) and 1 g ai/L (day 32)						
	Acetone wash		Peel		Pulp		% of total applied
	% of applied	% TRR	% of applied	% TRR	% of applied	% TRR	
7	60.3	97.6	1.00	1.62	0.51	0.83	61.8
14	56.0	95.8	2.00	3.42	0.46	0.79	58.5
21	58.6	93.4	3.29	5.25	0.83	1.32	62.7
28	62.3	92.1	4.52	6.68	0.86	1.27	67.7
32	90.4	96.0	2.85	3.03	0.96	1.02	94.2
35	74.1	89.9	6.00	7.28	2.31	2.80	82.4

The results as percentage of the applied radioactivity and percentage of peel (Tables, 10, 11, 12 and 13) or pulp (Tables 14, 15, 16 and 17) residues were summarized. The organo-soluble radioactivity in peel and pulp consisted mainly of triflumuron at all sampling times and trace of 2-chlorobenzamine and 4-trifluromethoxyphenyl urea were detected in the experiments conducted at the high application rate. The summary of distribution of radioactivity in the samples (31 days after high dose application) is shown in Table 18. Due to the low levels of radioactivity, the water-soluble and the insoluble fractions were not further analysed.

Table 10 Characterization of radioactivity in peel of apples following a single application of [2-chlorophenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 10 g ai/L)

Days after application	Peel: Organo-soluble		Peel: Water-soluble		Peel: Insoluble	
	%AR	%TRR of peel residue	%AR	%TRR of peel residue	%AR	%TRR of peel residue
5	0.31	52.6	0.24	40.7	0.04	6.8
11	0.42	50.0	0.38	45.2	0.04	4.8
14	0.03	5.4	0.50	89.3	0.03	5.4
19	0.03	3.5	0.81	93.1	0.03	3.5
24	0.04	4.9	0.73	90.1	0.04	4.9
31	0.04	4.9	0.73	90.1	0.04	4.9

Table 11 Characterization of radioactivity in peel of apples following a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 10 g ai/L)

Days after application	Peel: Organo-soluble		Peel: Water-soluble		Peel: Insoluble	
	%AR	%TRR of peel residue	%AR	%TRR of peel residue	%AR	%TRR of peel residue
5	0.35	61.4	0.08	14.0	0.14	24.6
11	0.33	52.4	0.18	28.6	0.12	19.1
14	0.36	61.0	0.10	17.0	0.13	22.0
19	0.53	60.2	0.11	12.5	0.24	27.3
24	0.52	56.5	0.18	19.6	0.22	23.9
27	0.65	63.1	0.14	13.6	0.24	23.3
31	0.51	60.7	0.09	10.7	0.24	28.6

Table 12 Characterization of radioactivity in peel of apples following a single application of [2-chlorophenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) or 1 g ai/L (day 32))

Days after application	Peel: Organo-soluble		Peel: Water-soluble		Peel: Insoluble	
	%AR	%TRR of peel residue	%AR	%TRR of peel residue	%AR	%TRR of peel residue
7	1.0	86.2	0.04	3.5	0.12	10.3
14	2.4	90.2	0.07	2.6	0.19	7.1
21	3.0	90.9	0.13	3.9	0.17	5.2
28	4.2	87.3	0.21	4.4	0.40	8.3
32	2.8	78.8	0.18	5.1	0.57	16.1
35	2.8	75.3	0.15	4.0	0.77	20.7

Table 13 Characterization of radioactivity in peel of apples following a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) or 1 g ai/L (day 32))

Days after application	Peel: Organo-soluble		Peel: Water-soluble		Peel: Insoluble	
	%AR	%TRR of peel residue	%AR	%TRR of peel residue	%AR	%TRR of peel residue
7	0.69	69.0	0.01	1.0	0.30	30.0
14	1.4	70.0	0.06	3.0	0.54	27.0
21	2.2	66.9	0.13	4.0	0.96	29.2
28	2.8	62.0	0.12	2.7	1.6	35.4
32	1.6	56.1	0.15	5.3	1.1	38.6
35	3.6	60.0	0.10	1.7	2.3	38.3

Table 14 Characterization of radioactivity in pulp of apples following a single application of [2-chlorophenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 10 g ai/L)

Days after application	Pulp: Organo-soluble		Pulp: Water-soluble		Pulp: Insoluble	
	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue
5	0.11	37.9	0.17	58.6	0.01	3.5
11	0.08	46.0	0.09	51.7	0.004	2.3
14	0.06	38.5	0.09	57.7	0.006	3.9
19	0.06	36.6	0.10	61.0	0.004	2.4
24	0.11	37.2	0.18	60.8	0.006	2.0
31	0.06	36.1	0.10	60.2	0.006	3.6

Table 15 Characterization of radioactivity in pulp of apples following a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 10 g ai/L)

Days after application	Pulp: Organo-soluble		Pulp: Water-soluble		Pulp: Insoluble	
	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue
5	0.07	63.6	0.02	18.2	0.02	18.2
11	0.14	77.8	0.03	16.7	0.01	5.6
14	0.22	81.5	0.04	14.5	0.01	3.7
19	0.38	84.4	0.05	11.1	0.02	4.4
24	0.31	81.6	0.05	13.2	0.02	5.3
27	0.63	87.5	0.06	8.3	0.03	4.2
31	0.42	84.0	0.06	12.0	0.02	4.0

Table 16 Characterization of radioactivity in pulp of apples following a single application of [2-chlorophenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) or 1 g ai/L (day 32))

Days after application	Pulp: Organo-soluble		Pulp: Water-soluble		Pulp: Insoluble	
	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue
7	0.30	79.0	0.03	7.9	0.05	13.2
14	0.56	88.9	0.04	6.4	0.03	4.8
21	0.96	88.9	0.07	6.5	0.05	4.6
28	1.2	88.2	0.08	5.9	0.08	5.9
32	1.1	85.9	0.11	8.6	0.07	5.5
35	1.9	87.6	0.18	8.3	0.09	4.2

Table 17 Characterization of radioactivity in pulp of apples following a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Application solution concentrations: approximately 0.2 g ai/L (days 7-28 and 35) or 1 g ai/L (day 32))

Days after application	Pulp: Organo-soluble		Pulp: Water-soluble		Pulp: Insoluble	
	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue	%AR	%TRR of pulp residue
7	0.40	78.4	0.03	5.9	0.08	15.7
14	0.38	82.6	0.03	6.5	0.05	10.9
21	0.71	85.5	0.04	4.8	0.08	9.6
28	0.70	81.4	0.05	5.8	0.11	12.8
32	0.73	76.0	0.08	8.3	0.15	15.6
35	1.90	82.3	0.17	7.4	0.24	10.4

Table 18 Distribution of radioactivity in ^{14}C triflumuron treated apples at day 31 after the application (application solutions: approximately 10 g ai/L)

Compounds	[2-chlorophenyl-UL-^{14}C] triflumuron		[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron	
	%TRR	mg eq/kg	%TRR	mg eq/kg
Acetone wash of apples	98.8	20.1	98.5	25.7
-triflumuron	98.4	20.0	97.6	25.5
-other compounds	0.4	0.08	0.9	0.23
Peel	1.0	0.21	0.9	0.25
Organic extract	0.05	0.01	0.6	0.15
-triflumuron	0.05	0.01	0.41	0.11
-other compounds			0.2	0.04
Aqueous phase	0.9	0.19	0.1	0.03
Unextracted residues	0.05	0.01	0.3	0.07
Pulp	0.2	0.04	0.6	0.15
Organic extract	0.08	0.02	0.5	0.12
-triflumuron	0.04	0.01	0.2	0.04
-other compounds	0.04	0.01	0.3	0.08
Aqueous phase	0.1	0.03	0.07	0.02
Unextracted residues	0.01	0	0.02	0.01
Sum identified	98.4	20.05	98.2	25.59
Sum characterized	1.5	0.31	1.5	0.39
Unextracted residue	0.06	0.01	0.3	0.08
Balance	100	83.82	100	26.06

Tomato (Spiegel & Sur, 2006, MEF-06/376, M-279281-01-1)

Metabolism study

The metabolism of triflumuron was studies on tomato plants. Each plant was cultivated under greenhouse conditions in 35 L containers were filled with soil (Einheitserde Typ T®).

One tomato plant was used for each experiment. Both the [2-chlorophenyl-UL-^{14}C] and [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron were formulated as 480 g ai/L SC and applied as a foliar spray a rate of 383 g ai/ha and 391 g ai/ha, respectively, twice (first: BBCH 75, second: BBCH 87 and 21 days of interval).

Tomatoes were harvested at BBCH 89 at least 7 days after the last application. The red, ripe fruits were separated from the stalks. The green tomatoes were sampled separately and kept in reserve with the stems and stalks.

The ripe tomatoes were washed with dichloromethane. The washing solution was combined for each crop and analysed by liquid scintillation counting (LSC), followed by the analysis by HPLC.

The washed fruits were homogenized in liquid nitrogen, and sub-samples stored at approximately -20 °C until analysis. All samples were analysed within one month of collection. Homogenised washed tomato samples were successively extracted by homogenisation three times with acetonitrile/water (80/20, v/v) and once with acetonitrile (1 × 300 mL). After each extraction step, the suspension was filtered under suction and the extracts combined and measured by LSC. The remaining solids were air-dried, and aliquots analysed by combustion and LSC. The combined acetonitrile/water extract was concentrated and partitioned against dichloromethane yielding an organic and an aqueous phase. The organic phase was concentrated after addition of an emulsifier (VPHOT 5902) and a small amount of slightly acidified water, to prevent losses of radioactivity due to adsorption effects of triflumuron and analysed by HPLC.

Metabolite profiles were obtained for the dichloromethane surface wash and the dichloromethane phase from the extraction procedure. Aliquots of the concentrated dichloromethane surface wash (after solvent exchange) were either subjected to structure elucidation by LC-MS/MS or were analysed by HPLC co-chromatography with authentic reference items. The concentrated dichloromethane extracts were analysed by TLC and HPLC with radiodetection (Table 19).

The TRR in tomatoes was 1.087 mg eq/kg and 1.383 mg eq/kg in the experiments of the application of [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron and [2-chlorophenyl-UL-^{14}C] triflumuron, respectively. By washing the fruits with dichloromethane, 96-98% of radioactivity was removed. In the acetonitrile/water extracts of the washed fruits, radioactivity was detected at 0.026 mg eq/kg (2.4% TRR) for [4-trifluoromethoxyphenyl-UL-^{14}C] and 0.045 mg eq/kg (3.2% TRR) for [2-chlorophenyl-UL-^{14}C] triflumuron. Most of this partitioned into the dichloromethane phase (2.2% and 3.0% TRR, respectively). For both radiolabels, less than 0.001 mg eq/kg remained in the post-extraction solids.

For both radiolabels, parent triflumuron was the only compound detected in the dichloromethane surface wash. In the experiment with [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron, identification of the parent compound in the surface washes was confirmed by LC-MS and LC-MS/MS. For [2-chlorophenyl-UL-^{14}C] triflumuron, identification of parent compound was confirmed by HPLC co-chromatography. The total amount of parent triflumuron in ripe tomatoes corresponded to 1.382 mg eq/kg (99.8% TRR) and 1.085 mg eq/kg (99.8% TRR) for [2-chlorophenyl-UL-^{14}C] triflumuron and [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron, respectively.

Table 19 Distribution and identification of radioactivity in [2-chlorophenyl-UL-^{14}C] triflumuron and [2-chlorophenyl-UL-^{14}C] triflumuron treated tomato foliage following foliar treatment at 0.38 and 0.39 kg ai/ha

Fraction	[4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron		[2-chlorophenyl-UL-^{14}C] triflumuron	
	%TRR	mg eq/kg	%TRR	mg eq/kg
Surface wash	97.59	1.061	96.75	1.338
-Triflumuron	97.59	1.061	96.75	1.338
Acetonitrile/water extract	2.39	0.026	3.23	0.045
Organosoluble phase	2.23	0.024	3.01	0.044
-Triflumuron	2.23	0.024	3.01	0.044
Water soluble phase	0.16	0.002	0.22	0.001
Total extracted	99.98	1.087	99.97	1.383
Total identified	99.81	1.085	99.75	1.382
Total unidentified	0.16	0.002	0.22	0.001

Fraction	[4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron		[2-chlorophenyl-UL-^{14}C] triflumuron	
	%TRR	mg eq/kg	%TRR	mg eq/kg
Total unextracted	0.02	< 0.001	0.03	< 0.001
TRR	100	1.087	100	1.383

Based on these studies, recovered radioactivity was predominantly found in the dichloromethane surface washes (96-98% TRR). The remaining part of radioactivity was detected in the extract of the fruit homogenates (2.4-3.2% TRR) and unextracted residues were very low (0.02–0.03% TRR). At harvest, 99.8% TRR was identified as parent triflumuron (corresponding to 1.085 mg eq/kg or 1.382 mg eq/kg for the 4-trifluoromethoxyphenyl-UL-^{14}C label or chlorophenyl-UL-^{14}C label, respectively) and no metabolites were not found either in surface washes or fruits.

Soya beans (Cain & Clay, 1983, MR86335, M-074440-01-1).

Translocation study

The leaf surface of approximately 30 soya bean plants in greenhouse was painted with a high dose of [2-chlorophenyl-UL-^{14}C] triflumuron (196 mg in total) or of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (206 mg in total). The mature soya beans were harvested 101 days post-treatment. The plants were watered by irrigating the soil, but not the treated leaves. The plants were maintained under greenhouse conditions and were harvested 101 days post-treatment.

In addition, 5 mg of [2-chlorophenyl-UL-^{14}C] triflumuron or [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron was injected into the stems of 15 soya bean plants. In these studies, at highest 0.11% of the applied radioactivity were recovered in the mature soya beans.

At harvest, parent triflumuron accounted for 72-99% TRR in foliage, 58-98% TRR in pods, and 14-40% TRR in mature seed. The amounts of non-extractable residue in pod (9.4-18.0% TRR) and mature seed (7.0-16.3% TRR) were higher than that in foliage (2.6-3.0% TRR) (Table 20**Error! Reference source not found.**).

Table 20 Amounts of triflumuron applied to soya bean plants and the radioactivity recovered in the mature soya bean seed

Position of ^{14}C-label	Applied radioactivity [Bq]	Recovered radioactivity in mature soya beans [Bq]	% of dose in mature soya beans
Foliar painting study			
[2-chlorophenyl-UL-^{14}C]	2.90×10^7	4.59×10^3	0.02
[4-trifluoromethoxy-phenyl-UL-^{14}C]	3.05×10^7	7.33×10^3	0.02
Stem injection study			
[2-chlorophenyl-UL-^{14}C]	7.40×10^5	7.03×10^2	0.10
[4-trifluoromethoxy-phenyl-UL-^{14}C]	7.40×10^5	7.77×10^2	0.11

Metabolism study

The metabolism of triflumuron was studied on soya beans (*Glycine max*) under field conditions using [2-chlorophenyl-UL-^{14}C] and [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron. Each soya bean plant was grown on an outdoor vegetation area.

Both [2-chlorophenyl-UL-^{14}C] and [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron was formulated as 480 g/L SC and applied as a foliar spray a rate of 1.12 kg ai/ha at full bloom. In the study with [2-chlorophenyl-UL-^{14}C] triflumuron, soya bean foliage was sampled at 0, 7, 14, 28, 63 and 77 days after treatment (BBCH not specified). The entire soya bean pod (beans plus outer hull) was sampled at 14, 28, 63 and 77 days after treatment. Mature soya beans were harvested 77 days

post-treatment. In the study with the 4-trifluoromethoxyphenyl-^{14}C label, soya bean foliage and pods were sampled at 0, 7, 14, 28 and 60 days after treatment; the mature soya beans were harvested 60 days post-treatment. Soil samples were collected in the study with the [2-chlorophenyl-UL-^{14}C] label at 7, 63 and 77 days after treatment.

Plant tissues were extracted by homogenizing three times with methanol, and the combined extracts were partitioned between hexane and water, to give hexane-soluble and water-soluble fractions. For the mature harvest samples, the water-soluble fraction was acidified to pH 1 before partitioning between water and ethyl acetate. The post-extraction solids were refluxed with 1 M HCl, and the filtrate partitioned between water and ethyl acetate.

Following a single application, the radioactive residue decreased in soya bean foliage over time. The highest residues were found in foliage, followed by the residues in pods. The residues in the mature beans were 0.21 mg eq/kg and 0.31 mg eq/kg at harvest. A summary of total radioactive residues (TRR) in foliage, pods and mature beans from treatment until harvest is shown in Tables 21 and 22.

Table 21 TRR of [2-chlorophenyl-UL-^{14}C] triflumuron in soya bean following foliar application at 1.12 kg ai/ha

	TRR (mg eq/kg)					
Days after treatment	0	7	14	28	63	77[b]
Foliage	83.82	30.31	24.21	14.47	6.94	5.75
Pods [a]	Ns	ns	1.40	0.86	0.42	0.82
Mature beans	Ns	ns	ns	ns	ns	0.21
Soil	Ns	0.33	ns	ns	0.44	0.43

[a] Outer hull plus immature bean

[b] Harvest sampling period for the raw agricultural commodity

ns Not sampled

Table 22 TRR of [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron in soya bean following foliar application at 1.12 kg ai/ha

	TRR (mg eq/kg)				
Days after treatment	0	7	14	28	60 [b]
Foliage	40.50	69.74	14.04	34.16	10.75
Pods [a]	8.98	6.04	1.03	1.21	1.47
Mature beans	ns	Ns	ns	ns	0.31

[a] Outer hull plus immature bean

[b] Harvest sampling period for the raw agricultural commodity

ns Not sampled

In foliage and pods, the main portion of radioactivity was extractable with hexane and mainly comprised parent triflumuron. Besides the parent compound, 2-chlorobenzoic acid (M02), 2-chlorobenzamide (M01), and 4-trifluoromethoxyphenyl urea (M08) were the only identified metabolites.

In mature beans, only 15.4 or 14.3% of TRR was extractable with hexane, the main portion of the radioactivity remained in the unextracted residues (44.8% or 70.6%). Acid hydrolysis (6 M HCl, reflux for 16 hours) released further parent triflumuron and additionally 2-chlorobenzoic acid (M02) after the treatment with the [2-chlorophenyl-UL-^{14}C] label and 4-trifluoromethoxyaniline (M07) after the treatment with the [4-trifluoromethoxy-phenyl-UL-^{14}C] label. Both compounds were most probably formed to a major extent during the hydrolysis procedure. The distribution of the radioactivity in all samples analysed is shown in Tables 23 and 24 for foliage, Tables 25 and 26 for

pods, and Table 27 for seed. Identification of triflumuron and its metabolites was performed by HPLC and TLC. The identity of triflumuron was confirmed by mass spectroscopy of the 28-day [2-chlorophenyl-UL-^{14}C] labelled foliage sample.

Table 23 Distribution and identification of radioactivity in [2-chlorophenyl-UL-^{14}C] triflumuron treated soya bean foliage following foliar treatment at 1.12 kg ai/ha

Days after treatment	0		7		14		28		63		77	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Hexane soluble compounds [a]	99.4	83.32	98.9	29.98	97.6	23.63	94.8	13.72	81.0	5.62	73.9	4.25
-triflumuron	98.9	82.90	98.4	29.83	94.3	22.83	94.4	13.66	80.8	5.61	71.9	4.13
-M01	0.1	0.08	0.1	0.03	-	-	-	-	-	-	-	-
-M02	0.1	0.08	-	-	-	-	-	-	0.1	0.01	1.3	0.07
-unknown A	-	-	-	-	0.5	0.12	-	-	-	-	-	-
-unknown B	-	-	0.4	0.12	0.3	0.07	0.2	0.03	-	-	0.2	0.01
-unknown C	0.2	0.17	-	-	0.3	0.07	0.1	0.01	0.1	0.01	0.2	0.01
-unknown D	0.1	0.08	-	-	2.2	0.53	0.1	0.01	-	-	0.3	0.02
Water soluble compounds [b]	0.4	0.34	0.8	0.24	0.6	0.15	3.9	0.56	14.1	0.98	16.9	0.97
Acid extracted: -triflumuron -origin	na	na	na	na	na	na	na	na	na	na	 6.1 7.3	 0.35 0.42
Acid non-extracted	na	na	na	na	na	na	na	na	na	na	1.7	0.10
Unextracted residue [c]	0.2	0.17	0.3	0.09	1.8	0.44	1.3	0.19	4.9	0.34	9.2	0.53
HCl released (organo-soluble)[a] -M02 -origin	na	na	na	na	na	na	na	na	na	na	 3.5 1.0	 0.20 0.06
HCl released (water soluble)[a]	na	na	na	na	na	na	na	na	na	na	1.7	0.10
Not released	na	na	na	na	na	na	na	na	na	na	3.0	0.17
Sum identified	99.1	83.07	98.5	29.86	94.3	22.83	94.4	13.66	80.9	5.61	82.8	4.76
Sum characterized	0.7	0.59	1.2	0.36	3.9	0.94	4.3	0.62	14.2	0.99	14.2	0.82
Non-extracted residue	0.2	0.17	0.3	0.09	1.8	0.44	1.3	0.19	4.9	0.34	3.0	0.17
Balance	100.0	83.82	100.0	30.31	100.0	24.21	100.0	14.47	100.0	6.94	100.0	5.75

M01 2-chlorobenzamide

M02 2-chlorobenzoic acid

[a] analysed by HPLC

[b] analysed by TLC

[c] at day 77: additional extraction with acid led to an acid-extractable fraction[2] and an acid non-extractable fraction

na – not analysed

– <LOD

Table 24 Distribution and identification of radioactivity in [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron treated soya bean foliage following foliar treatment at 1.12 kg ai/ha

Days after treatment	0		7		14		28		60	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Hexane soluble compounds [a]	92.2	37.34	96.0	66.95	95.2	13.37	91.8	31.36	90.8	9.76
-triflumuron	92.1	37.30	95.4	66.53	95.0	13.34	91.1	31.12	90.5	9.73
-origin	0.1	0.04	0.6	0.42	0.2	0.03	0.7	0.24	0.3	0.03
Water soluble compounds [b]	7.7	3.12	3.4	2.37	3.2	0.45	6.3	2.15	4.7	0.51
Base-extracted:	na	na	na	na	na	Na	na	na		
-triflumuron									0.6	0.06
-M08									1.1	0.12
-unknown K									0.2	0.02
-origin									2.2	0.24
Base non-extracted	na	na	na	na	na	Na	na	na	0.6	0.06
Unextracted residue [c]	0.1	0.04	0.6	0.42	1.6	0.22	1.9	0.65	4.5	0.48
HCl released (organo-soluble)[a]	na	na	na	na	na	Na	na	na		
-triflumuron									-	-
-M07									0.4	0.04
-origin									0.5	0.05
HCl released (water soluble)[a]	na	na	na	na	na	Na	na	na	1.0	0.11
Not released	na	na	na	na	na	Na	na	na	2.6	0.28
Sum identified	92.1	37.30	95.4	66.53	95.0	13.34	91.1	31.12	90.5	9.73
Sum characterized	7.8	3.16	4.0	2.79	3.4	0.48	7.0	2.39	5.0	0.54
Unextracted	0.1	0.04	0.6	0.42	1.6	0.22	1.9	0.65	2.6	0.28
Balance	100.0	40.50	100.0	69.74	100.0	14.04	100.0	34.16	100.0	10.75

M07 trifluoromethoxy aniline

M08 4-trifluoromethoxyphenyl urea

[a] analysed by HPLC

[b] analysed by TLC

[c] at day 60: additional extraction with acid led to an acid-extractable fraction[2] and an acid non-extractable fraction

na not analysed

– <LOD

Table 25 Distribution and identification of radioactivity in [2-chlorophenyl-UL-^{14}C] triflumuron treated soya bean pods following foliar treatment at 1.12 kg ai/ha

Days after treatment	14		28		63		77	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Hexane soluble compounds [a]	72.3	1.012	83.8	0.721	57.5	0.242	73.7	0.604
-triflumuron	72.1	1.009	83.1	0.715	57.5	0.242	71.7	0.588

Days after treatment	14		28		63		77	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
-M01	-	-	-	-	-	-	0.6	0.005
-unknown B	-	-	0.5	0.004	-	-	-	-
-unknown C	0.2	0.003	0.2	0.002	-	-	-	-
-unknown D	-	-	-	-	-	-	1.4	0.011
Water soluble compounds	12.1	0.169	11.9	0.102	16.4	0.069	16.9	0.139
Unextracted residue	15.6	0.218	4.3	0.037	26.1	0.110	9.4	0.077
Sum identified	72.1	1.009	83.1	0.715	57.5	0.242	72.3	0.593
Sum characterized	12.3	0.172	12.6	0.108	16.4	0.069	18.3	0.150
Unextracted	15.6	0.218	4.3	0.037	26.1	0.110	9.4	0.077
Balance	100.0	1.400	100.0	0.860	100.0	0.420	100.0	0.820

M01 2-chlorobenzamide

[a] analysed by HPLC

- <LOD

Table 26 Distribution and identification of radioactivity in [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron treated soya bean pods following foliar treatment at 1.12 kg ai/ha

Days after treatment	0		7		14		28		60	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Hexane soluble compounds [a]	97.8	8.78	95.0	5.74	90.4	0.93	84.4	1.02	72.9	1.07
-triflumuron	97.8	8.78	93.7	5.66	88.6	0.91	82.3	1.00	68.9	1.01
-unknown H	-	-	1.3	0.08	1.8	0.02	1.4	0.02	2.6	0.04
-unknown I	-	-	-	-	-	-	0.7	0.01	-	-
-unknown J	-	-	-	-	-	-	-	-	1.4	0.02
Water soluble compounds	2.1	0.19	3.9	0.24	7.2	0.07	9.7	0.12	9.1	0.13
Unextracted	0.1	0.01	1.1	0.07	2.4	0.02	5.9	0.07	18.0	0.26
Sum identified	97.8	8.78	93.7	5.66	88.6	0.91	82.3	1.00	68.9	1.01
Sum characterized	2.1	0.19	5.2	0.31	9.0	0.09	11.8	0.14	13.1	0.19
Unextracted	0.1	0.01	1.1	0.07	2.4	0.02	5.9	0.07	18.0	0.26
Balance	100.0	8.98	100.0	6.94	100.0	1.03	100.0	1.21	100.0	1.47

[a] analysed by TLC

– <LOD

Table 27 Distribution and identification of radioactivity in [2-chlorophenyl-UL-^{14}C] triflumuron treated mature soya bean seed following foliar treatment at 1.12 kg ai/ha

	[2-chlorophenyl-UL-^{14}C] triflumuron		[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron	
Days after treatment	77		60	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg
Hexane soluble compounds [a]	15.4	0.032	14.3	0.04

	[2-chlorophenyl-UL-^{14}C] triflumuron		[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron	
Days after treatment	77		60	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg
-triflumuron	14.4	0.030	14.3	0.04
-M01	0.4	0.001		
-M02	0.4	0.001		
-unknown A	0.2	0.000		
Water soluble compounds	14.0	0.026	40.9	0.13
Acid extractable [b]			np	np
-triflumuron	3.2	0.007		
-origin	3.3	0.007		
Acid unextracted	7.5	0.016	np	np
Base extracted [a]	np	np	26.0	0.08
Base unextracted	np	np	14.9	0.05
Unextracted residue [c]	70.6	0.080	44.8	0.14
HCl released				
-triflumuron	2.1	0.004		
-M02	30.2	0.063		
-M07[b]			33.1	0.10
-unknown E	14.7	0.031		
-unknown F	3.4	0.007		
-unknown G	3.9	0.008		
HCl released (water soluble)[a]			4.7	0.01
Unextracted residue	16.3	0.034	7.0	0.02
Sum identified	50.7	0.106	73.4	0.23
Sum characterized	33.0	0.069	19.6	0.06
Unextracted residue	16.3	0.034	7.0	0.02
Balance	100.0	0.210	100.0	0.31

M01:2-chlorobenzamide

M02:2-chlorobenzoic acid

M07:trifluoromethoxyaniline

[a] analysed by HPLC

[b] analysed by TLC

[c] additional extraction with acid led to an acid-extractable fraction and an acid non-extractable fraction

np not performed

Metabolites identified in soya beans were 2-chlorobenzamide (M01), 2-chlorobenzoic acid (M02), 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08). The proposed metabolism pathway for triflumuron in soya beans was as shown in Figure 2.

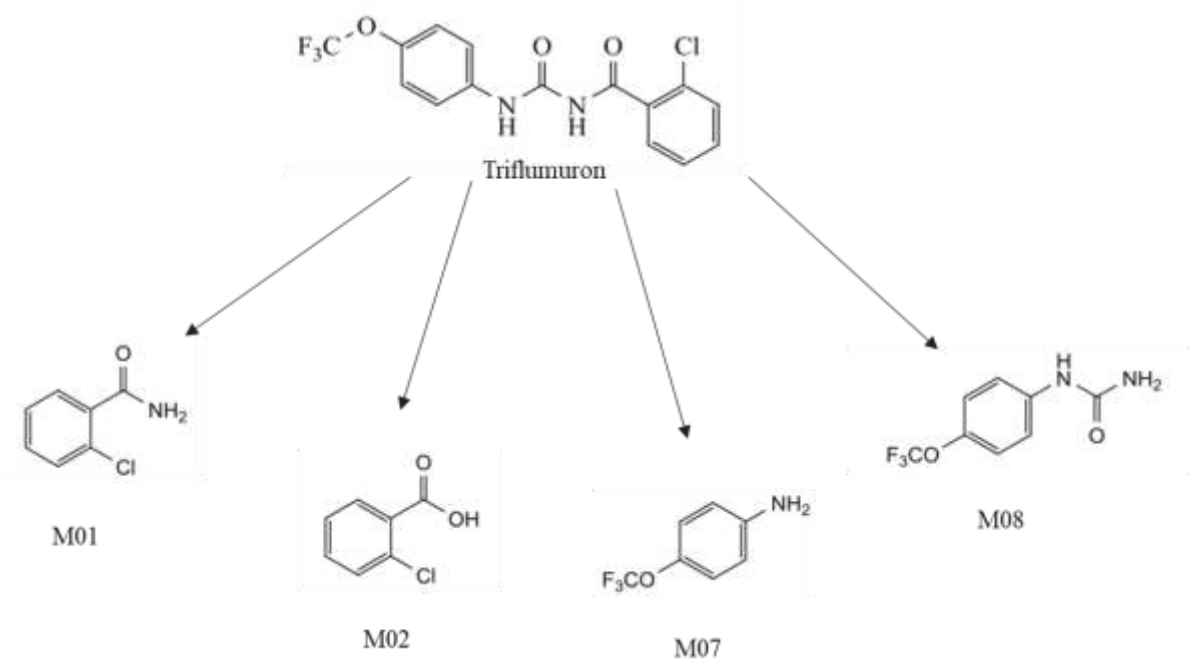


Figure 2 Proposed metabolic pathway for triflumuron in soya beans

Potatoes (Whitfield & Clay, 1983, MR86121, M-074433-01-1)

Translocation study

The leaf surface of four potato plants under greenhouse condition was treated with a high dose of [2-chlorophenyl-UL-^{14}C] triflumuron (221 mg in total) or [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (217 mg in total). The potato tubers were harvested 42 days post-treatment. The plants were watered only by irrigating the soil, not the treated leaves. 0.005–0.008% of AR was detected in potato tubers (Table 28).

[2-chlorophenyl-UL-^{14}C] triflumuron or [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron was injected into the stems of two potato plants. The plants were maintained under greenhouse conditions and were harvested 6 days post-treatment. Stem injection resulted in the low level of residue in the tubers (0.7% at the highest of the applied radioactivity; Table 28).

Table 28 Translocation studies on potatoes

Position of ^{14}C label	%AR in tubers
High dose foliar application study	
2-chlorophenyl-UL-^{14}C	0.005
4-trifluoromethoxyphenyl-UL-^{14}C	0.008
Stem injection study	
2-chlorophenyl-UL-^{14}C	0.39, 0.17
4-trifluoromethoxyphenyl-UL-^{14}C	0.02, 0.65

In another experiment seed potatoes were injected with [2-chlorophenyl-UL-^{14}C] triflumuron and stored in the dark. Analysis of the radioactive residue was performed 12 days post-treatment and 97% of the applied radioactivity was recovered as triflumuron.

In potato foliage, between 64% and 99% TRR were parent triflumuron and less than 1% TRR was represented by 2-chlorobenzamide (M01), 2-chlorobenzoic acid (M02) and 4-trifluoro-methoxyaniline (M07), and between 1% and 6% TRR was identified as 4-trifluoromethoxyphenyl

urea (M08). In potato tuber, 42-49% TRR was identified as triflumuron and M01, M02 and M08 were also identified (2.9% TRR, 7.5% TRR and 13.8-19.0% TRR, respectively).

Metabolism study

The Meeting received the metabolism study of triflumuron in potatoes (*Solanum tuberosum*) using [2-chlorophenyl-UL-^{14}C] triflumuron and [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron (Figure 1).

The study with the [2-chlorophenyl-UL-^{14}C] label was conducted as an outdoor study. Potato foliage and tubers were sampled at 7, 14, 21, 28 and 42 days after a single application of [2-chlorophenyl-UL-^{14}C] triflumuron at bloom with an application rate of 1.12 kg ai/ha and an additional sample of foliage was taken at day 0. Soil samples were collected at day 7, 21, 28 and 42 after the single treatment.

The study with the [4-trifluoromethoxyphenyl-UL-^{14}C] label was a greenhouse experiment. Potato foliage and tubers were sampled at 7, 14, 28 and 42 days after a single application of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron at bloom with an application rate of 1.12 kg ai/ha and an additional sample of foliage was taken at day 0.

Following the single application of [2-chlorophenyl-UL-^{14}C] triflumuron, the residue levels in foliage declined from 83.54 mg eq/kg at day 0 to 23.06 mg eq/kg at day 28, but a high level of residue (75.03 mg eq/kg) was observed at day 42. The residue in tuber increased from 0.01 to 0.08 mg eq/kg. The residue levels of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron in foliage were similar from day 7 to 42 (8.03-16.63 mg eq/kg) and consistently low in tuber (0.01–0.02 mg eq/kg). The data is summarized in Tables 29 and 30.

Table 29 TRR of [2-chlorophenyl-UL-^{14}C] triflumuron in potato following foliar application at 1.12 kg ai/ha (outdoor experiment)

	Total radioactive residue (TRR) (mg eq/kg)					
Days after treatment	0	7	14	21	28	42[a]
Foliage	83.54	42.91	97.95	37.55	23.06	75.03
Tuber	ns	0.01	0.04	0.07	0.05	0.08
Soil	ns	0.21	Ns	0.32	0.22	0.12

[a] harvest sampling period for the raw agricultural commodity

ns-not sampled

Table 30 TRR of [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron in potato following foliar application at 1.12 kg ai/ha (green house experiment)

	Total radioactive residue (TRR) (mg eq/kg)				
Days after treatment	0	7	14	28	42[a]
Foliage	40.99	8.03	8.25	16.63	10.76
Tuber	ns	0.02	0.01	0.01	0.01

[a] harvest sampling period for the raw agricultural commodity

ns-not sampled

The main portion of the radioactivity in foliage was extractable with acetone and dichloromethane ([2-chlorophenyl-UL-^{14}C] triflumuron) or methanol ([4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron) and comprised mainly triflumuron. Analysis of the organo-soluble extracts showed the presence of 2-chlorobenzoic acid (M02), 2-chlorobenzamide (M01), 4 trifluoromethoxyphenyl urea (M08) and 4-trifluoromethoxyaniline (M07)(Tables 31, 32 and 33).

In potato tuber, between 46.0% and 80.9% of the radioactive residue was organo-extractable and 19.1% to 39.6% remained in the post-extraction solids. Enzymatic hydrolysis (glucosidase and cellulase) of the fraction was ineffective. Acid hydrolysis experiments (2 M HCl, reflux for 6 hours) in the study with the [2-chlorophenyl-UL-^{14}C] label showed that the main part of the unextracted residues could be released and 4.6% of the radioactive residue remained as non-extractable residue. The distribution of the radioactivity in all samples analysed is shown in Table 19 and Table 20 for foliage, and Table 21 for tubers. Identification of triflumuron and its metabolites was performed by co-chromatography with authentic reference compounds in at least two different TLC systems and in one HPLC system for several extracts. The structure of triflumuron was confirmed by mass spectroscopy in the extract of the 28-day [2-chlorophenyl-UL-^{14}C] labelled foliage sample.

Table 31 Distribution and identification of radioactivity in [2-chlorophenyl-UL-^{14}C] triflumuron treated potato foliage following foliar treatment at 1.12 kg ai/ha

Days after treatment	0		7		14		21		28		42	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Organo-soluble compounds [a]	99.7	83.29	99.6	42.74	98.6	96.58	96.2	36.12	94.7	21.84	94.2	70.68
-triflumuron	99.2	82.87	98.9	42.44	98.1	96.09	95.0	35.67	93.2	21.49	92.3	69.25
-M01	0.3	0.25	0.3	0.13	0.1	0.10	0.1	0.04	0.2	0.05	0.2	0.15
-M02	0.1	0.08	0.2	0.09	0.1	0.10	0.3	0.11	0.4	0.09	0.8	0.60
-unknown E	-	-	0.1	0.04	0.2	0.20	0.8	0.30	0.8	0.18	0.7	0.53
-unknown F	0.1	0.08	0.2	0.09	0.1	0.10			0.4	0.09	0.8	0.60
Water soluble compounds	0.1	0.08	0.1	0.04	0.4	0.39	1.8	0.68	3.0	0.69	3.5	2.63
Unextracted	0.2	0.17	0.3	0.13	1.0	0.98	2.0	0.75	2.3	0.53	2.3	1.73
Sum identified	99.6	83.21	99.3	42.61	98.3	96.28	95.4	35.82	93.5	21.56	92.7	69.55
Sum characterized	0.2	0.17	0.4	0.17	0.7	0.69	2.6	0.98	4.2	0.97	5.0	3.75
Balance	100.0	83.54	100.0	42.91	100.0	97.95	100.0	37.55	100.0	23.06	100.0	75.03

M01 2-chlorobenzamide

M02 2-chlorobenzoic acid

[a] analysed by HPLC

Table 32 Distribution and identification of radioactivity [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron treated potato foliage following foliar treatment at 1.12 kg ai/ha

Days after treatment	0		7		14		21		42	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Organo-soluble compounds [a]	99.8	40.91	99.4	7.98	98.3	8.11	96.8	16.10	78.5	8.45
-triflumuron	92.9	38.08	84.6	6.79	80.3	6.62	83.1	13.82	64.3	6.92
-M07	0.2	0.08	0.7	0.06	0.7	0.06	0.2	0.03	0.3	0.03
-M02	5.1	2.09	5.7	0.46	3.8	0.31	2.7	0.45	1.0	0.11
-unknown D	0.1	0.04	4.2	0.34	1.5	0.12	1.4	0.23	2.8	0.30
-origin	1.5	0.61	4.2	0.34	12.0	0.99	9.4	1.56	10.1	1.09
Unextracted	0.2	0.08	0.6	0.05	1.7	0.14	3.2	0.53	21.5	2.31

Days after treatment	0		7		14		21		42	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Sum identified	98.2	40.25	91.0	7.31	84.8	7.00	86.0	14.30	65.6	7.06
Sum characterized	1.6	0.66	8.4	0.67	13.5	1.11	10.8	1.80	12.9	1.39
Balance	100.0	40.99	100.0	8.03	100.0	8.25	100.0	16.63	100.0	10.76

M07 trifluoromethoxy aniline

M08 4-trifluoromethoxyphenyl urea

[a] analysed by TLC

Table 33 Distribution and identification of radioactivity in [2-chlorophenyl-UL-^{14}C] triflumuron and [4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron treated potato tuber following foliar treatment at 1.12 kg ai/ha

	[2-chlorophenyl-UL-^{14}C] triflumuron		[4-trifluoromethoxyphenyl-UL-^{14}C] triflumuron							
Days after treatment	42		7		14		28		42	
Compounds	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Organo-soluble compounds [a d]	46.0	0.037	80.9	0.016	76.6	0.008	77.9	0.008	78.5	0.008
-triflumuron	25.6	0.020					43.2	0.004	41.5	0.004
-M02	13.6	0.011								
-unknown A	0.9	0.001								
-unknown B	3.3	0.003								
-unknown C	0.9	0.001								
-M08							19.0	0.002	13.8	0.001
-origin	1.7	0.001					15.7	0.002	23.2	0.002
Water soluble compounds	14.4	0.012								
Organic phase after HCl hydrolysis [a]	13.2	0.011								
-triflumuron	8.3	0.007								
-origin	4.9	0.004								
Aqueous phase	1.2	0.001								
Unextracted [c]	39.6	0.032	19.1	0.004	23.4	0.002	22.1	0.002	21.5	0.002
HCl released [b]										
-triflumuron	15.3	0.012								
-M01	2.9	0.002								
-M02	13.9	0.011								
-unknown E	2.9	0.002								
Unextracted with HCl	4.6	0.004								
Sum identified	79.6	0.064					62.2	0.006	55.3	0.006
Sum characterized	15.8	0.013					15.7	0.002	23.2	0.002
Not released with HCl	4.6	0.004					22.1	0.002	21.5	0.002
Balance	100.0	0.080	100.0	0.004	100.0	0.010	100.0	0.010	100.0	0.010

M01 2-chlorobenzamide
M02 2-chlorobenzoic acid
M08 4-trifluoromethoxyphenyl urea
[a] analysed by TLC
[b] analysed by HPLC
[c] additional extraction with acid led to an acid-extractable fraction[1] and an acid non-extractable fraction
[d] only the 28-day and 42-day samples for the [^{14}C-Trifluoromethoxyphenyl] label were analysed by TLC

The metabolic pathway for triflumuron in potato was proposed to be identical to that for soya bean (Figure 2).

Environmental Fate

Hydrolysis

The Meeting received a hydrolysis study on triflumuron (Lenz, M.F., 1984, M-019861-02-1). [2-chlorophenyl-UL-^{14}C] triflumuron (purity ~99%) was incubated in sterile buffer solutions (pH 5, 7 and 9, containing 1% of acetonitrile) at a concentration of 0.02, 0.04, 0.2 and 0.4 mg/L. These solutions were autoclaved for one hour and incubated in the dark at 25 °C for 0, 3, 7, 14, 21 and 30 days. Each sample was co-chromatographed by silica gel TLC with non-labelled standards. The solvent systems were benzene/ethyl acetate/acetic acid (50:25:1) and hexane/ethyl acetate/acetic acid (25:25:1). No degradation after 30 days was observed at pH 5 and DT_{50} at pH 7 (by extrapolation) and pH 9 were 465 and 57 days, respectively. The primary degradation product in solution at pH 9 was 2-chloro-benzoic acid (M02) (Table 34).

Table 34 Hydrolysis study of triflumuron

Compound	Day 0	Day 3	Day 7	Day 14	Day 21	Day 30
	% AR					
pH 5						
Triflumuron	97.4	91.2	94.4	92.2	93.9	96.0
M02	1.5	5.2	2.8	4.1	2.7	2.4
M01	0.7	2.6	2.1	2.3	2.0	0.7
Origin	0.4	1.0	0.7	1.4	1.4	0.9
Total	100.0	100.0	100.0	100.0	100.0	100.0
pH 7						
Triflumuron	98.0	93.0	92.7	92.7	93.8	91.5
M02	0.9	2.9	4.4	4.3	2.2	3.0
M01	1.1	2.9	2.9	1.7	1.6	2.2
Origin	ND	1.2	ND	1.3	2.4	3.3
Total	100.0	100.0	100.0	100.0	100.0	100.0
pH 9						
Triflumuron	07.9	90.8	86.3	77.8	76.1	66.2
M02	1.3	7.1	10.9	17.2	20.4	28.9
M01	0.7	2.1	1.2	2.5	1.9	2.7
Origin	0.1	ND	1.6	2.5	1.6	2.2
Total	100.0	100.0	100.0	100.0	100.0	100.0

ND: < 0.02 mg eq/mL

Aerobic degradation

The Meeting received a study on the aerobic degradation of triflumuron in silt (Hoefchen, pH 6.7 in $CaCl_2$, organic carbon 2.11%) and a sandy loam soil (Laacher Hof, pH 6.3 in $CaCl_2$, organic carbon 1.02%) for a maximum period of 120 days in the dark at 20 °C (Hellpointner & Kloeppner, 2002, MR-365/02, M-082409-01-1). [4-Trifluoromethoxy-phenyl-UL-^{14}C] triflumuron or [2-chlorophenyl-UL-^{14}C] triflumuron was applied at a nominal rate of 0.24 mg ai/kg dry soil (5 cm depth of soil, equivalent to 0.18 kg ai/ha). The test system consisted of Erlenmeyer flasks attached with traps containing polyurethane foam and soda lime for adsorption of volatile organic compounds and CO_2, respectively. Entire flasks were taken for processing and analysis 0.1 (approximately 2 hours), 1, 3, 7, 16, 30, 56, 91 and 120 days after treatment. Soil samples were each extracted three times at room temperature with acetonitrile. Combined extracts were analysed by TLC. For identification of the transformation products co-chromatography and spectroscopic methods (LC/MS and LC/MS/MS) were used. Polyurethane foam was extracted with acetonitrile for the analysis of volatile organic compounds. The $^{14}CO_2$ adsorbed by soda lime was liberated with HCl and purged into LSC cocktail with nitrogen. Non-extractable radioactivity in soil was determined by combustion (Table 35).

Table 35 Physiochemical characteristics of soil

Soil characteristic type	pH	Organic matter	Microbial biomass (mg microbial C/kg soil dry wt)	Field moisture capacity at 1/3 bar	WHCmax (g water/100 soil)	Bulk density
Silt Hoefchen	7.6 (water) 6.7 ($CaCl_2$) 7.1-6.6 (KCl)	3.6%	1166 (day 0) 792 (day 120)	36%	63%	2.09 g/cm^3
sandy loam soil Laacher Hof	7.2 (water) 6.3 ($CaCl_2$) 6.7-5.0 (KCl)	1.8%	690 (day 0) 253 (day 120)	29%	32%	2.5 g/cm^3

In the silt soil, the total recovery of radioactivity was between 92.3% and 101.9% of the applied radioactivity (Table 36). In the sandy loam soil, the total recovery of radioactivity was between 93.9% and 100.9% (Table 37).

In the test with [2-chlorophenyl-UL-^{14}C] triflumuron, volatile organic compounds were not detected at any sampling interval. In the test with [4-Trifluoromethoxy-phenyl-UL-^{14}C] triflumuron in both soils the amount of volatile organic compounds increased at the maximum of 1.0-1.2% TRR on day 30 and then decreased to 0.4–0.6% (day 120). For both compounds, $^{14}CO_2$ levels consistently increased throughout the experiments (64.6% TRR (silt) and 65.9% TRR (sandy loam) for [2-chlorophenyl-UL-^{14}C] triflumuron and 18.1% TRR (silt) and 12.8% TRR (sandy loam) for [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron after 120 days).

The extraction efficiency (expressed as % of the applied radioactivity) decreased with increasing incubation period in all test series. Higher amounts of unextracted residues resulted from [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron than [2-chlorophenyl-UL-^{14}C] triflumuron. Unextracted residues reached a maximum between day 16 (silt: 35.1%) and day 56 (sandy loam: 26.6%) for [2-chlorophenyl-UL-^{14}C] triflumuron and between day 91 (silt: 76.8%) and day 120 (sandy loam: 68.7%) for [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron.

During the course of the study, triflumuron residues in soil extracts declined from ≥93% of the applied radioactivity (day 0) to values of 2.3% and 1.6% (silt) and 8.2% and 6.0% (sandy loam) for the [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron and [2-chlorophenyl-UL-^{14}C] triflumuron, respectively. Apart from parent triflumuron, two metabolites were detected and quantified (4-Trifluoromethoxyphenyl urea (M08) and 2-chlorobenzoic acid (M02)). Residues of 4-Trifluoromethoxyphenyl urea (M08), found in the test using [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron, reached a maximum between 3 and 7 days post treatment (13.5% and 12.3% of the

applied radioactivity for silt and sandy loam, respectively), and then declined towards the end of the study, yielding 0.3% to 2.8% of the applied radioactivity at day 120. The metabolite 2-chlorobenzoic acid (M02) originated from the application of [2-chlorophenyl-UL-^{14}C] triflumuron reached a maximum of 5.9% (silt) and 3.9% (sandy loam) of the applied radioactivity at day 3 and 7, respectively.

The degradation curve and regression analysis were computed with the evaluation programme ModelManager version 1.1. For determination of the degradation kinetics of triflumuron in soil, the mean values of both labels were calculated for each soil. According to the simple first order model (SFO), the DT_{50} values of triflumuron were calculated to be 6.9 days (silt soil) and 18.8 days (sandy loam soil). The respective DT_{90} values were 23.0 and 62.5 days (**Error! Reference source not found.**).

Table 36 Material balance and distribution of residues for the silt soil (expressed as % of AR)

Sampling interval (days)	0.1 d	1 d	3 d	7 d	16 d	30 d	56 d	91 d	120 d
[4-trifluoromethoxy-phenyl-UL-^{14}C] applied at 0.24 mg ai/kg dry soil									
Volatiles		0.1	0.5	1.5	5.7	9.4	13.7	15.9	18.5
Organic		0.1	0.1	0.1	1.0	1.0	0.9	0.5	0.4
CO_2		0.1	0.4	1.4	4.7	8.4	12.8	15.4	18.1
Extract	93.6	90.1	85.9	63.0	31.6	11.3	7.8	4.1	3.6
Triflumuron	93.0	80.5	71.5	49.3	21.8	6.3	5.4	2.8	2.3
4-trifluoromethoxyphenyl urea (M08)	-	9.4	13.5	13.0	7.2	2.1	1.1	0.4	0.3
Unextracted [a]	4.8	8.4	15.5	31.9	62.0	71.6	74.1	76.8	70.9
Balance	98.3	98.6	101.9	96.4	99.4	92.3	95.6	96.8	93.0
[2-chlorophenyl-UL-^{14}C] triflumuron applied at 0.24 mg ai/kg dry soil									
Volatiles		1.9	9.4	23.8	43.5	46.9	61.6	62.7	64.6
Organic		< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
CO_2		1.9	9.4	23.8	43.5	46.9	61.6	62.7	64.6
Extract	95.8	87.2	72.4	44.8	19.9	19.3	3.0	2.2	2.1
Triflumuron	94.7	82.8	65.8	40.1	17.0	17.4	2.2	1.6	1.6
2-chlorobenzoic acid (M02)	-	4.1	5.9	4.4	1.7	0.8	-	-	-
Unextracted [a]	5.2	11.0	20.0	26.7	35.1	31.1	33.3	32.5	30.0
Balance	100.9	100.1	101.9	95.3	98.6	97.4	97.9	97.4	96.8

[a] soil + polyurethane filter

- <LOD (0.001 mg eq/kg)

Table 37 Material balance and distribution of residues for the sandy loam soil (expressed as % of AR)

Sampling interval (days)	0.1 d	1 d	3 d	7 d	16 d	30 d	56 d	91 d	120 d
[4-trifluoromethoxy-phenyl-UL-^{14}C] applied at 0.24 mg ai/kg dry soil									
Volatiles		0.1	0.3	0.9	3.1	5.8	8.2	10.1	13.4
Organic		< 0.1	0.1	< 0.1	0.6	1.2	1.0	0.5	0.6
CO_2		0.1	0.3	0.9	2.4	4.6	7.2	9.6	12.8
Extract	94.7	93.5	86.2	74.9	59.6	37.0	28.7	24.6	12.2
Triflumuron	94.0	87.8	75.6	62.5	46.4	24.5	21.3	20.1	8.2
4-trifluoromethoxyphenyl urea (M08)	-	5.5	10.3	12.3	11.3	9.8	6.0	3.8	2.8
Unextracted [a]	3.4	5.6	11.0	19.6	36.2	52.3	60.8	61.6	68.7
Balance	98.2	99.2	97.6	95.5	98.9	95.2	97.6	96.3	94.2
[2-chlorophenyl-UL-^{14}C] triflumuron applied at 0.24 mg ai/kg dry soil									
Volatiles		1.2	4.1	14.5	24.7	39.2	54.0	55.6	65.9

Sampling interval (days)	0.1 d	1 d	3 d	7 d	16 d	30 d	56 d	91 d	120 d
Organic		< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
CO_2		1.2	4.1	14.5	24.7	39.2	54.0	55.6	65.9
Extract	95.7	92.3	78.5	64.3	52.5	33.0	17.8	16.6	6.2
Triflumuron	95.6	88.9	74.3	60.2	48.3	30.8	17.1	16.2	6.0
2-chlorobenzoic acid (M02)	-	2.1	3.4	3.9	3.3	1.5	0.3	-	-
Unextracted [a]	4.4	7.5	11.3	18.6	22.1	24.1	26.6	24.9	24.0
Balance	100.1	100.9	93.9	97.4	99.3	96.2	98.5	97.1	96.1

[a] soil + polyurethane filter

- <LOD (0.001 mg eq/kg)

The Meeting received a study on the biotransformation of radiolabelled triflumuron in one European soil (Laacher Hof AIIIa: silt loam, pH 6.7 in $CaCl_2$, organic carbon 0.94% under aerobic conditions for 49 days at 20 °C±1 °C in the dark, at about 50% of the maximum water holding capacity (Heinemann & Dehner, 2005, MEF-05-043, Bayer Ref: M-248322-01-1)). [Phenoxy-UL-^{14}C] triflumuron was applied in acetonitrile at a nominal test rate of 240 µg/kg soil, equivalent to a virtual field application rate of 180 g/ha. The test systems consisted of Erlenmeyer flasks, each containing 100 g of soil dry matter equivalents, which were fitted with solid phase traps for the collection of CO_2 and volatile organic compounds (static test system design). Duplicate samples were analysed at intervals of 0, 1, 3, 6, 10, 21, 35, and 49 days post application. Soil samples were extracted three times by shaking with acetonitrile, and the combined extracts were analysed by normal phase TLC. Representative samples were analysed by reversed phase (R18) TLC for confirmatory purposes. Identity confirmation of the test item and the main transformation product 4-trifluoromethoxyphenyl urea (M08) was performed by co-chromatography with certified unlabelled reference items in both chromatography systems and by HPLC-MS/MS.

The mean material balance was 98.1 of the applied radioactivity. Parent triflumuron decreased from 95.9% at day 0 to 10.9% of the applied radioactivity by the end of the study (day 49). Non-extractable ^{14}C-residues increased from 3.1% of the applied radioactivity on day 0 to 72.2% AR by the end of the study. At study termination, evolved $^{14}CO_2$ accounted for 6.6% of the applied radioactivity. No significant amounts of volatile organics were detected (maximum abundances of 1.2% of the applied radioactivity on day 10 and 35). One major transformation product was identified as 4-trifluoromethoxyphenyl urea (M08), found at a maximum abundance of 19.1% of the applied radioactivity on day 6, and declining towards the end of the study to 4.3% of the applied radioactivity. Further unknown metabolites were observed, but in sum did not exceed 2.9% of the applied radioactivity in any sample. The simple first order (SFO) fitting of an exponential decline function using ModelManager Version 1.1 led to acceptable results with an R^2 of 0.98. The laboratory DT_{50} and DT_{90} of triflumuron in the aerobic soil tested was calculated to be 7.3 and 24.1 days, respectively (Table 39). All data indicated that triflumuron and its metabolite 4-trifluoromethoxyphenyl urea (M08) degraded continuously. In addition, it was found that no metabolite accumulated, and that the unextracted residues formed participated in the natural carbon cycle of soil.

In an additional study, the degradation of radiolabelled triflumuron was tested in three different soils of European origin (Stroech, 2009, MEF-09/188, M-348643-01-1). Triflumuron was applied to a loam (Laacher Hof "Wurmwiese": pH($CaCl_2$) 5.6, 1.7% OC) and two clay loam type soils (Dollendorf II: pH($CaCl_2$) 7.3, 5.4% OC; Les Cayades: pH($CaCl_2$) 7.6, 0.9% OC) from Germany and France and incubated for up to 32 days under aerobic laboratory conditions in the dark, at 20 ± 1 °C and 55 ± 5% of MWHC soil moisture. The amount of the test item for the treatment of the incubation flasks was 240 µg triflumuron per kg of soil dry weight. This study application rate was based on the single maximum field use rate (180 g/ha), assuming a homogeneous distribution of triflumuron in a topsoil layer of 5 cm depth at a generic soil bulk density of 1.5 g/cm^3. The test systems consisted of open Erlenmeyer glass flasks each containing 100 g of soil dry weight equivalents (static test system design). Duplicate samples were analysed after 0, 0.25, 1, 2, 4 and 7 days of incubation for all soils. In addition, samples for soil Laacher Hof "Wurmwiese" were analysed on day 14 and for soil Les

Cayades on days 14, 21 and 32. Soil samples were extracted twice with acetonitrile at room temperature, and once with acetonitrile using microwave-accelerated solvent extraction. The combined extracts were analysed for triflumuron residues by reversed phase HPLC-MS/MS in multiple reaction monitoring (MRM) mode using matrix-matched non-labelled triflumuron standards (Table 38).

Table 38 Degradation of triflumuron in the 3 European test soils

Soil type	Recovery of triflumuron (% of AR)								
DAT (days)	0	0.25	1	2	4	7	14	21	32
Laacher Hof "Wurmwiese"	98.7	88.9	74.9	65.2	48.8	20.0	5.0	na	na
Dollendorf II	96.0	83.0	65.7	44.6	16.1	3.5	na	na	na
Les Cayades	97.2	91.4	86.9	82.9	74.7	53.3	20.5	11.0	2.2

During study incubation the concentration of triflumuron decreased rapidly in all soils, dropping below 10% of the applied at 7 to 32 days after application. At study termination, the residue levels were 5.0, 3.5 and 2.2% of the applied amount for soils Laacher Hof "Wurmwiese", Dollendorf II and Les Cayades, respectively. In all three soils the degradation of triflumuron followed single first order (SFO) kinetics according to the lowest chi-square values. A summary of the kinetics results of triflumuron degradation, using KinGUI software, is presented below in Table 39.

Table 39 Single first order (SFO) half-life of triflumuron in different soil types

Soil type		Organic carbon (%)	SFO fit		
			r^2	DT_{50} (days)	DT_{90} (days)
Sandy loam	Laacher Hof	1.02	0.95	18.8	62.5
Silt	Höfchen	2.11	0.99	6.9	23.0
Silt loam	Laacher Hof AIIIa	0.94	0.98	7.3	24.1
Loam	Laacher Hof "Wurmwiese"	1.7	0.988	3.5	11.6
Clay loam	Dollendorf II	5.4	0.994	1.7	5.7
Clay loam	Les Cayades	0.9	0.986	7.3	24.4

Soil photolysis

The Meeting received a study on the photolytic degradation of triflumuron on a silt loam soil (pH 6.4, organic carbon 1.57%)(Coody, 1986, MR91983, M-041075-02-1). A slurry of sieved (2 mm) soil was uniformly distributed onto the surface of petri dishes. After air drying for several days each sample was treated with [2-chlorophenyl-UL-^{14}C] triflumuron at a rate corresponding to 560 g ai/ha. The samples were incubated in two closed chambers at 25 ± 1 °C. Each system was connected to three different traps in order to avoid losses of radioactivity by evaporation. Petri dishes in one chamber were exposed for 41 days to artificial light with a wave-length spectrum similar to that of natural sun light, whereas the other chamber was not illuminated. Duplicate irradiated and non-irradiated samples were taken 0, 4, 8, 14, 22, and 32 days post treatment and analysed separately. On day 41 post treatment, 4 replicate samples were taken. At each sampling point, soils were extracted with MeOH/H_2O (4:1) and the extracts analysed by LSC and HPLC. At each sampling time, gas washing solutions were replaced and assayed for ^{14}C by LSC. The exchange resin from each system was also replaced and assayed for ^{14}C by combustion followed by LSC.

The average light intensity measured was 1062 ± 54 μW/cm^2. Considering the reported sunlight intensity of 2745 μW/cm^2 for 12 hours/day in Kansas during July and the artificial irradiation time of 24 hours per day, a factor of 0.77 relative light energy is obtained. Utilizing this factor, the sampling times of 0, 4, 8, 14, 22, 32 and 41 days post treatment resulted in the soil receiving UV radiation equivalent to 0, 3.1, 6.2, 10.8, 16.9, 24.6, and 31.6 days of natural sunlight.

Quantitative radioactive recovery (98.5% to 104.8% of the applied radioactivity) was obtained from the soil at all sampling times (Table 40). No significant concentrations (≤0.1% of the applied radioactivity) of volatiles containing the ^{14}C label were observed in the gas-scrubbing traps at any time. Regarding both irradiated and dark samples, the bulk of radioactivity (96.6% to 103.9% of the applied radioactivity) could be extracted, whilst only a minor fraction (0.9% to 4.0% of the applied radioactivity) remained bound to the soil. At the end of the study (day 41), 97% of the extracted radioactivity in irradiated samples was identified as unchanged triflumuron, compared to 99% in the non-irradiated samples. The two metabolites identified, 2-chlorobenzamide (M01) and 2-chlorobenzoic acid (M02), did not exceed 1% of the recovered radioactivity, except 2-chlorobenzamide (M01) on day 22 (3%). No statistically significant differences in the radioactive recovery or decomposition of triflumuron were observed between the illuminated vs. non-illuminated systems (Tables 40 and 41).

Table 40 Material balance and distribution of radioactivity in a silt loam after irradiation on Petri dishes

	% of AR						
Sampling intervals (day)	0	4	8	14	22	32	41
Extracted	96.7	101.8	100.6	100.7	100.7	97.4	97.2
Triflumuron (in % of extracted activity)	100	95	97	98	95	97	97
Unextracted	4.0	1.5	2.4	2.8	3.3	3.8	2.3
Volatiles	NS	0.0	0.1	0.0	0.1	0.0	0.1
Recovery	100.7	103.3	103.1	103.5	104.1	101.2	99.6

NS – not sampled

Table 41 Material balance and distribution of radioactivity in a silt loam in dark control samples

	% of AR						
Sampling intervals (day)	0	4	8	14	22	32	41
Extracted	96.7	103.9	103.1	99.9	97.0	96.6	97.6
Triflumuron (in % of extracted activity)	100	98	98	98	98	99	99
Unextracted	4.0	0.9	1.3	1.7	1.6	1.9	1.7
Volatiles	NS	0.0	0.0	0.0	0.0	0.0	0.0
Recovery	100.7	104.8	104.4	101.6	98.6	98.5	99.3

NS – not sampled

Rotational crop studies (confined)

The Meeting received information on a confined accumulation study on rotational crops with [2-chlorophenyl-UL-^{14}C] triflumuron using kale (*Brassica oleracea acephla*, "Dwarf Scotch"), red beets (*Beta goosefoot*, "Detroit dark red"), and wheat (*Triticum aestivum*, "Triumph") (Lenz & Clay, 1984, MR86749, M-074443-01-1).

[2-chlorophenyl-UL-^{14}C] triflumuron was formulated as a 25% WP and applied to a container of the sandy loam soil (pH 6.4, organic matter 1.8%) as a coarse spray a first and second application rate of 0.28 kg/ha and 0.84 kg/ha, respectively, with an interval of 7 days between applications. The total application rate was 1.12 kg/ha. After each application the sprayed triflumuron was incorporated

into the top layer (5.1 cm) of the soil, and after the second application the entire tub was planted with wheat as a cover crop.

Approximately 1, 4 and 9 months after the second treatment, the soil was tilled to a depth of about 15 cm, and kale, red beets and wheat were planted (1-month PBI: May, 4-month PBI: August-September, 9-month PBI: January). The crops were grown outdoors under cover crops (wheat) until cold weather necessitated moving the container inside a greenhouse (at 23 °C). Kale (leaves), beet (tops and roots), mature wheat (stalks and heads: unseparated wheat grain and chaff) were harvested at maturity, with additional immature forage wheat stalks taken from immature wheat plants.

Each sample was homogenized in liquid nitrogen and Soxhlet extracted with methanol for at least 42 hours. The remaining solids were radioassayed by oxidation. The methanol extract was radioassayed directly by LSC and then evaporated, re-dissolved in water and partitioned with ethyl acetate. Both aqueous and organic phases were radioassayed by LSC. The organic phase from the 1-month re-plant samples were concentrated and analysed by TLC.

Total radioactivity and the metabolic profiles were determined for all matrices of the crops under investigation. Soil samples were taken after the last application (0 days after treatment) and after collection of the last crop sample and were analysed.

The TRR in the tested plant commodities ranged between 0.01 mg eq/kg and 0.66 mg eq/kg. The highest residues were found in tops and roots of red beets (0.58 and 0.66 mg eq/kg, respectively) in the first plant back interval (PBI). Low TRR values were found in the second and third PBI. For kale and red beets, the TRR decreased to 0.01 mg eq/kg; for wheat values decreased to between 0.01 mg eq/kg and 0.08 mg eq/kg. A decrease in the concentration of radioactivity in soil was also observed. The summary of the result is shown in Table 42**Error! Reference source not found.**.

Table 42 Total radioactive residues in rotational crops grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron and in the soil itself

Crops	TRR (shown as [2-chlorophenyl-UL-^{14}C] triflumuron in mg eq/kg)		
	First PBI (1 month)	Second PBI (4 months)	Third PBI (9 months)
Kale	0.25	0.01	0.01
Red beets			
Tops	0.58	0.01	0.01
Roots	0.66	0.01	0.01
Wheat			
Forage	0.25	0.01	0.03
Heads	0.26	0.03	0.07
Straw	0.53	0.03	0.08
Soil [a]			
At planting	0.63	0.25	0.14
At harvest [b]	0.27	0.24	0.17

[a] Soil was treated at a rate equivalent to 1.12 kg/ha approximately 1 month before the first crop was planted; the residue level in the soil immediately after application of triflumuron was 1.40 mg/kg

[b] Residue level in soil when the last crop sample was collected, 336 DALA

The non-extractable radioactivity increased with increasing plant back interval, except for wheat forage where no change was seen. Extractability was between 30.8% (wheat heads, 4-month crop) and 88.5% (beet tops, 1-month crop) of the TRR. Organo-soluble compounds were released from a plant by 42-hour Soxhlet extraction with methanol.

For the non-extractable fraction, acid hydrolysis (HCl, 6 M) at 60-70 °C for up to 60 hours resulted in some amount of 2-chlorobenzoic acid (M02). Treatment with cellulase, hemicellulose or β-glucosidase (incubated for 16 hours at 37 °C) or with milder acid (1.5 and 3.0 M of HCl at 60-70 °C

for up to 60 hours) did not produce significant additional identified activity. The data is summarized in **Error! Reference source not found.**the following tables**Error! Reference source not found.**.

Table 43 Characterization and identification of the total radioactive residues in rotational crops (kales and beets) grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (1st PBI, 1 month; % of TRR and ai equivalent in mg/kg)

	Kale		Beet tops		Beet roots	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract [a]						
Organo-soluble						
-triflumuron	5.7	0.01	4.1	0.02	2.3	0.02
-2-chlorobenzoic acid(M02)	14.5	0.04	19.9	0.12	13.6	0.09
-2-chlorobenzamide (M01)	7.4	0.02	19.8	0.11	2.1	0.01
-unknown	0.4	< 0.01	-	-	-	-
-origin	19.7	0.05	14.1	0.08	21.4	0.14
Subtotal organo-soluble	47.7	0.12	57.9	0.34	39.4	0.26
Water soluble [b]						
-triflumuron	2.9	0.01	7.3	0.04	2.5	0.02
-2-chlorobenzoic acid (M02)	20.7	0.05	16.0	0.09	32.5	0.21
-origin	5.2	0.01	7.3	0.04	7.7	0.05
Subtotal water-soluble	28.8	0.07	30.6	0.18	42.7	0.28
Unextracted [b]	23.5	0.06	11.5	0.07	17.9	0.12
HCl released organic phase						
-2-chlorobenzoic acid (M02)	1.6	< 0.01	0.9	0.01	2.1	0.01
-origin	1.0	< 0.01	1.0	0.01	2.5	0.02
Aqueous phase	20.8	0.05	5.3	0.03	8.4	0.06
Non-released	0.1	< 0.01	4.3	0.02	4.9	0.03
Total	100.0	0.25	100.0	0.58	100.0	0.66

[a] Residues from the extractable fractions were subjected to TLC analysis for identification of the compounds

[b] Residues from the water-soluble and non-extractable fractions were released by HCl; released compounds were analysed by TLC

Table 44 Characterization and identification of the total radioactive residues in rotational crops (wheat) grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (1st PBI, 1 month; % of TRR and ai equivalent in mg/kg)

	Wheat forage		Wheat heads		Wheat straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract [a]						
Organo-soluble						
-triflumuron	4.3	0.01	1.4	< 0.01	4.7	0.02
-2-chlorobenzoic acid(M02)	16.5	0.04	12.6	0.03	15.0	0.08
-2-chlorobenzamide (M01)	3.7	0.01	2.5	0.01	1.1	0.01
-unknown	0.3	< 0.01	0.3	< 0.01	1.9	0.01
-origin	3.8	0.01	0.9	< 0.01	4.5	0.02
Subtotal organo-soluble	28.6	0.07	17.7	0.05	27.2	0.14
Water extracted [b]						
-triflumuron	1.7	< 0.01	4.7	0.01	4.9	0.03
-2-chlorobenzoic acid (M02)	19.5	0.05	36.2	0.09	30.2	0.16
-origin	3.5	0.01	3.2	0.01	6.3	0.03
Subtotal water-soluble	24.7	0.06	44.1	0.11	41.4	0.22
Non-extractable [b]						

	Wheat forage		Wheat heads		Wheat straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
HCl released organic phase						
-2-chlorobenzoic acid (M02)	5.2	0.01	15.3	0.04	7.9	0.04
-origin	1.2	< 0.01	1.7	< 0.01	1.8	0.01
Aqueous phase	32.3	0.08	12.3	0.03	6.4	0.03
Non-released	8.0	0.02	8.9	0.02	15.3	0.08
Subtotal unextracted	46.7	0.12	38.2	0.10	31.4	0.17
Total	100.0	0.25	100.0	0.26	100.0	0.53

[a] Residues from the extractable fractions were subjected to TLC analysis for identification of the compounds

[b] Residues from the water-soluble and non-extractable fractions were released by HCl; released compounds were analysed by TLC

Table 45 Characterization and identification of the total radioactive residues in rotational crops (kales, beets) grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (2nd PBI, 4 month; % of TRR and ai equivalent in mg/kg)

	Kale		Beet tops		Beet roots	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract						
Organo-soluble	44.4	< 0.01	46.3	< 0.01	47.7	< 0.01
Water extractable	30.9	< 0.01	8.8	< 0.01	7.3	< 0.01
Unextracted	24.7	< 0.01	44.9	< 0.01	45.0	< 0.01
Total	100.0	0.01	100.0	0.01	100.0	0.01

Table 46 Characterization and identification of the total radioactive residues in rotational crops (wheat) grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (2nd PBI, 4-month; % of TRR and ai equivalent in mg/kg)

	Wheat forage		Wheat heads		Wheat straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract						
Organo-soluble	31.6	< 0.01	18.2	0.01	19.0	0.01
Water extractable	22.8	< 0.01	12.6	< 0.01	30.4	0.01
Unextracted	45.6	< 0.01	69.2	0.02	50.2	0.02
Total	100.0	0.01	100.0	0.03	100.0	0.03

Table 47 Characterization and identification of the total radioactive residues in rotational crops (kales, beets) grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (3rd PBI, 9 month; % of TRR and ai equivalent in mg/kg)

	Kale		Beet tops		Beet roots	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract						
Organo-soluble	23.8	< 0.01	47.9	< 0.01	26.2	< 0.01
Water extractable	28.5	< 0.01	15.6	< 0.01	28.5	< 0.01
Unextracted	47.7	< 0.01	36.5	< 0.01	45.3	< 0.01
Total	100.0	0.01	100.0	0.01	100.0	0.01

Table 48 Characterization and identification of the total radioactive residues in rotational crops (wheat) grown in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (3rd PBI, 9 month; % of TRR and ai equivalent in mg/kg)

	Wheat forage		Wheat heads		Wheat straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract						
Organo-soluble	27.8	0.01	25.6	0.02	29.0	0.02
Water extractable	25.0	0.01	12.5	0.01	26.5	0.02
Unextracted	47.2	0.01	61.9	0.04	44.5	0.04
Total	100.0	0.03	100.0	0.07	100.0	0.08

Table 49 Characterization and identification of the total radioactive residues in soil treated with [2-chlorophenyl-UL-^{14}C] triflumuron (% of TRR and ai equivalent in mg/kg)

	1-month PBI				4-month PBI				9-month PBI			
	Planting		Harvest		Planting		Harvest		Planting		Harvest	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extract												
Organo-soluble												
-triflumuron	60.7	0.38	30.1	0.08	26.8	0.07	18.8	0.05	11.9	0.02	20.4	0.03
-2-chlorobenzoic acid (M02)	1.6	0.01	1.5	< 0.01	1.4	< 0.01	0.9	< 0.01	0.7	< 0.01	1.5	< 0.01
-2-chlorobenzamide (M01)	2.2	0.01	1.3	< 0.01	1.5	< 0.01	1.5	< 0.01	0.9	< 0.01	1.7	< 0.01
-origin	0.5	< 0.01	0.8	< 0.01	1.1	< 0.01	0.7	< 0.01	0.5	< 0.01	0.9	< 0.01
Subtotal organo-soluble	65.0	0.41	33.7	0.09	30.8	0.08	21.9	0.05	14.0	0.02	24.5	0.04
Water-soluble	20.6	0.13	7.3	0.02	2.5	0.01	1.5	< 0.01	0.9	< 0.01	1.2	< 0.01
Unextracted	14.4	0.09	59.0	0.16	66.7	0.17	76.6	0.18	85.1	0.12	74.3	0.13
Total	100.0	0.63	100.0	0.27	100.0	0.25	100.0	0.24	100.0	0.14	100.0	0.17

The metabolites of triflumuron found in succeeding crops were also found in the plant metabolism studies. Parent compound was assumed to be the major residue, and 2-chlorobenzoic acid (M02) and 2-chlorobenzamide (M01) were present at low levels after conventional extraction in the plant studies and their amounts increased after acid hydrolysis of the unextracted residues. The Meeting estimated the metabolic pathway of [2-chlorophenyl-UL-^{14}C] triflumuron in rotational crops as shown in Figure 3**Error! Reference source not found.**.

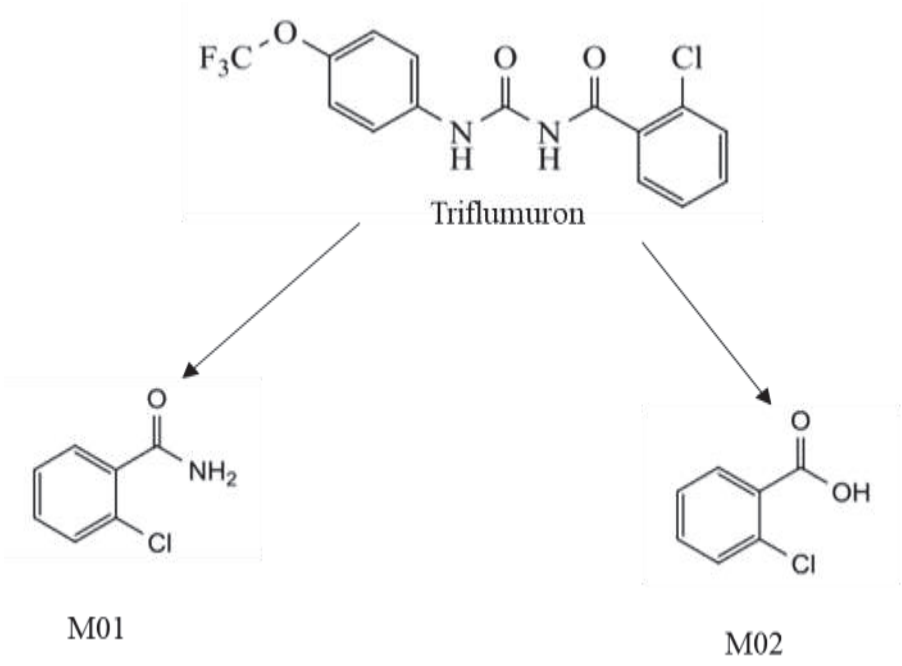


Figure 3 Pathway for the metabolism of triflumuron in rotational crops

ANIMAL METABOLISM

Information was available on metabolism of triflumuron in rats, lactating goats and laying hens.

Lactating goats (Obrist & Sietsema, 1984, 86889, M-074563-01-1)

The metabolism of triflumuron in the lactating goats was studied using [2-chlorophenyl-UL-^{14}C] and [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron administered orally in gelatine capsules, once daily immediately after milking.

A lactating goat (mixed breed alpine; 70 kg) was administered one time orally with 3.0 mg/kg bw of [2-chlorophenyl-UL-^{14}C] triflumuron in a gelatine capsule (165 ppm assuming the feed intake of lactating goat as 1.27 kg). The blood concentration of [2-chlorophenyl-UL-^{14}C] triflumuron reached the maximum concentration of 0.027 mg eq/kg at 20 hours after dosing. The goat was then given a single oral dose of 25.1 mg/kg bw of [2-chlorophenyl-UL-^{14}C] triflumuron in a gelatine capsule at 56 hours after the first dose in order to generate "high" residues in tissues and milk and sacrificed 20 hours after the final dose.

Approximately 60% of the AR was excreted within 56 hours after a single administration of [2-chlorophenyl-UL-^{14}C] triflumuron (25.1 mg/kg bw) via the faeces (53.7%) and urine (6.2%)**Error! Reference source not found.**.

Table 50 Excretion of radioactivity in excreta from lactating goat after administration of a single dose of [^{14}C-chlorophenyl] triflumuron

Time (hour)	Total excretion from administration (% AR)	
	Faeces	Urine
within 12	0.02	0.43
within 24	8.37	2.82
within 32	27.96	4.14
within 48	49.20	5.67

within 56	53.72	6.19

In the second experiment, a lactating goat (mixed breed alpine; 31 kg) was administered three consecutive daily oral doses of [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron at 17.8 mg/kg bw in a gelatine capsule (439 ppm assuming the feed intake of lactating goat as 1.27 kg). Milk samples were collected every morning throughout the study. The animal was sacrificed 20 hours after the final dose.

Tissues and organs collected after sacrifice were homogenized and extracted with methanol for liver, kidney and muscle and with hexane for fat. Milk was extracted by dichloromethane. Urine and faeces were also collected and extracted with methanol. Analysis of TRR was carried out using combustion and liquid scintillation counting (LSC). Metabolite identification was based on co-chromatography with authentic reference compounds using TLC and HPLC or on spectroscopic evidence.

A low amount was secreted with the milk, and residue levels increased throughout the multiple dose experiment to a maximum concentration of 0.76 mg/kg. The residue level hierarchy was liver > fat > kidney > milk > muscle. Summary is shown in Table 51.

Table 51 Total radioactive residues in milk and edible products of lactating goats after administration of triflumuron

Matrix	Residue (mg eq/kg, expressed as triflumuron)	
	[2-chlorophenyl-UL-^{14}C] triflumuron [a]	[4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron [b]
Milk	0.43	0.76
Liver	3.33	6.12
Kidneys	0.87	1.60
Muscle	0.30	0.18
Fat	1.83	4.82

[a] first dose of 3.0 mg/kg bw & second dose of 25.1 mg/kg bw

[b] three daily doses of 17.8 mg/kg bw each

Unchanged triflumuron was found in all samples and accounted for 15.3-96.1% of the TRR. The lowest concentration of triflumuron was found in kidney and liver, and the highest concentration in fat. 4-Trifluoromethoxyaniline (M07), a likely metabolite of triflumuron, was not detected in tissues and milk.

Higher levels of 2-chlorohippuric acid (M03) than SIR 8514-3-hydroxy-2-chlorophenyl (M04) was found in milk and tissues in the experiment with the [2-chlorophenyl-UL-^{14}C] triflumuron and free and conjugated 2-chlorohippuric acid (M03 and M21) was found in urine at 56 hours after treatment. The summary is given in Table 52, 53, 54 and 55. The summary of metabolism in lactating goat is shown in Figure 3.

Table 52 [2-chlorophenyl-UL-^{14}C] triflumuron: Extractability in the edible tissues, organs and milks of the lactating goat

	Milk		Liver		Kidney		Muscle		Fat	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Extracted by solvent [a]	95.8	0.41	31.2	1.04	71.4	0.62	83.3	0.25	99.0	1.81
Hexane phase	< 0.1		-	-	-	-	-	-	1.0	0.02
Water-soluble	4.2	0.02	50.1	1.67	21.3	0.19	7.1	0.02	-	-
Unextracted	< 0.1		18.7	0.62	7.3	0.06	9.6	0.03	< 0.1	
Total	100.0	0.43	100.0	3.33	100.0	0.87	100.0	0.30	100.0	1.83

[a] methanol for liver, kidney and muscle, hexane for fat and dichloromethane for milk.

Table 53 [2-chlorophenyl-UL-^{14}C] triflumuron: Quantitative distribution of triflumuron and its metabolites in the edible tissues, organs and milks of the lactating goat

	Milk		Liver		Kidney		Muscle		Fat	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
TRR		0.43		3.33		0.87		0.30		1.83
triflumuron	75.0	0.32	15.3	0.51	20.2	0.18	58.4	0.18	96.1	1.76
2-chlorobenzoic acid (M02)	-	-	-	-	-	-	-	-	-	-
2-chlorobenzamide (M01)	7.5	0.03	4.5	0.15	5.3	0.05	20.4	0.06	0.9	0.02
2-chlorohippuric acid										
Free acid (M03)	6.2	0.03	-	-	18.3	0.16	-	-	-	-
Conjugate (M21)	-	-	-	-	17.6	0.15	-	-	-	-
3-hydroxyl derivative										
Free acid (M04)	-	-	-	-	0.8	0.01	-	-	-	-
Conjugate (M22)	4.1	0.02	-	-	-	-	-	-	-	-
5-hydroxyl derivative										
Free acid (M05)	-	-	1.6	0.05	-	-	-	-	-	-
Conjugate (M23)	-	-	2.4	0.08	-	-	-	-	-	-
Polar metabolites [a]	2.0	0.01	3.9	0.13	4.7	0.04	2.5	0.01	1.6	0.03
Origin [a]	1.0	0.00	3.5	0.12	4.5	0.04	2.0	0.01	0.4	0.01
Subtotal	95.8	0.41	31.2	1.04	71.4	0.62	83.3	0.25	99.0	1.81
Hexane phase	< 0.1		-	-	-	-	-	-	1.0	0.02
Water-soluble	4.2	0.02	50.1	1.67	21.3	0.19	7.1	0.02	-	-
Unextracted	< 0.1		18.7	0.62	7.3	0.06	9.6	0.03	< 0.1	
Total	100.0	0.43	100.0	3.33	100.0	0.87	100.0	0.30	100.0	1.83
Sum identified	92.8	0.40	23.8	0.79	62.2	0.54	78.8	0.24	97.0	1.78

[a] Characterized by TLC

<LOD (0.001 mg eq/kg)

Table 54 [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron: extractability in the edible tissues, organs and milks of the lactating goat

	Milk		Liver		Kidney		Muscle		Fat	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
TRR		0.76		6.12		1.60		0.18		4.82
Extract by solvent[1]	88.6	0.67	52.4	3.21	85.9	1.37	91.8	0.17	97.6	4.70
Hexane phase	3.2	0.02	-	-	-	-	-	-	0.2	0.01
Water-soluble	4.9	0.04	3.0	0.18	8.8	0.14	3.0	0.01	-	-
Unextracted	3.3	0.03	44.6[4]	2.73	5.3	0.08	5.2	0.01	2.2	0.11
Total	100.0	0.76	100.0	6.12	100.0	1.60	100.0	0.18	100.0	4.82

[a] methanol for liver, kidney and muscle, hexane for fat and dichloromethane for milk.

Table 55 [4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron: Quantitative distribution of triflumuron and its metabolites in the edible tissues, organs and milks of the lactating goat

	Milk		Liver		Kidney		Muscle		Fat	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
TRR		0.76		6.12		1.60		0.18		4.82
triflumuron	59.6	0.45	19.9	1.22	27.0	0.43	80.3	0.14	95.4	4.60
Unknown 1										
Free acid	1.6	0.01	1.3	0.08	0.3	< 0.01	1.6	< 0.01	-	-
Conjugate	-	-	0.9	0.06	-	-	-	-	-	-
3-hydroxyl derivative										
Free acid (M04)	4.5	0.03	2.5	0.15	1.5	0.02	1.6	< 0.01	0.9	0.04
Conjugate (M22)	6.4	0.05	5.7	0.35	27.0	0.43	-	-	-	-
Unknown 2										
Free acid	1.3	0.01	2.1	0.13	2.0	0.03	1.9	< 0.01	-	-
Conjugate	-	-	0.2	0.01	6.1	0.10	-	-	-	-
Unknown 3										
Free acid	0.3	0.00	0.6	0.04	0.6	0.01	1.5	< 0.01	-	-
Conjugate	-	-	0.3	0.02	-	-	-	-	-	-
4-trifluoromethoxyphenyl urea										
Free acid (M08)	-	-	0.5	0.03	1.4	0.02	0.9	< 0.01	-	-
Conjugate (M24)	4.8	0.04	1.0	0.06	1.0	0.02	-	-	-	-
Unknown 4										
Free acid	0.5	0.00	0.3	0.02	0.8	0.01	-	-	-	-
Conjugate	3.1	0.02	1.7	0.10	-	-	-	-	-	-
Polar metabolites	6.5[b]	0.05	15.4[a]	0.94	18.2[b]	0.29	1.7[c]	< 0.01	1.0[c]	0.05
Origin							2.3[c]	< 0.01	0.3[c]	0.01

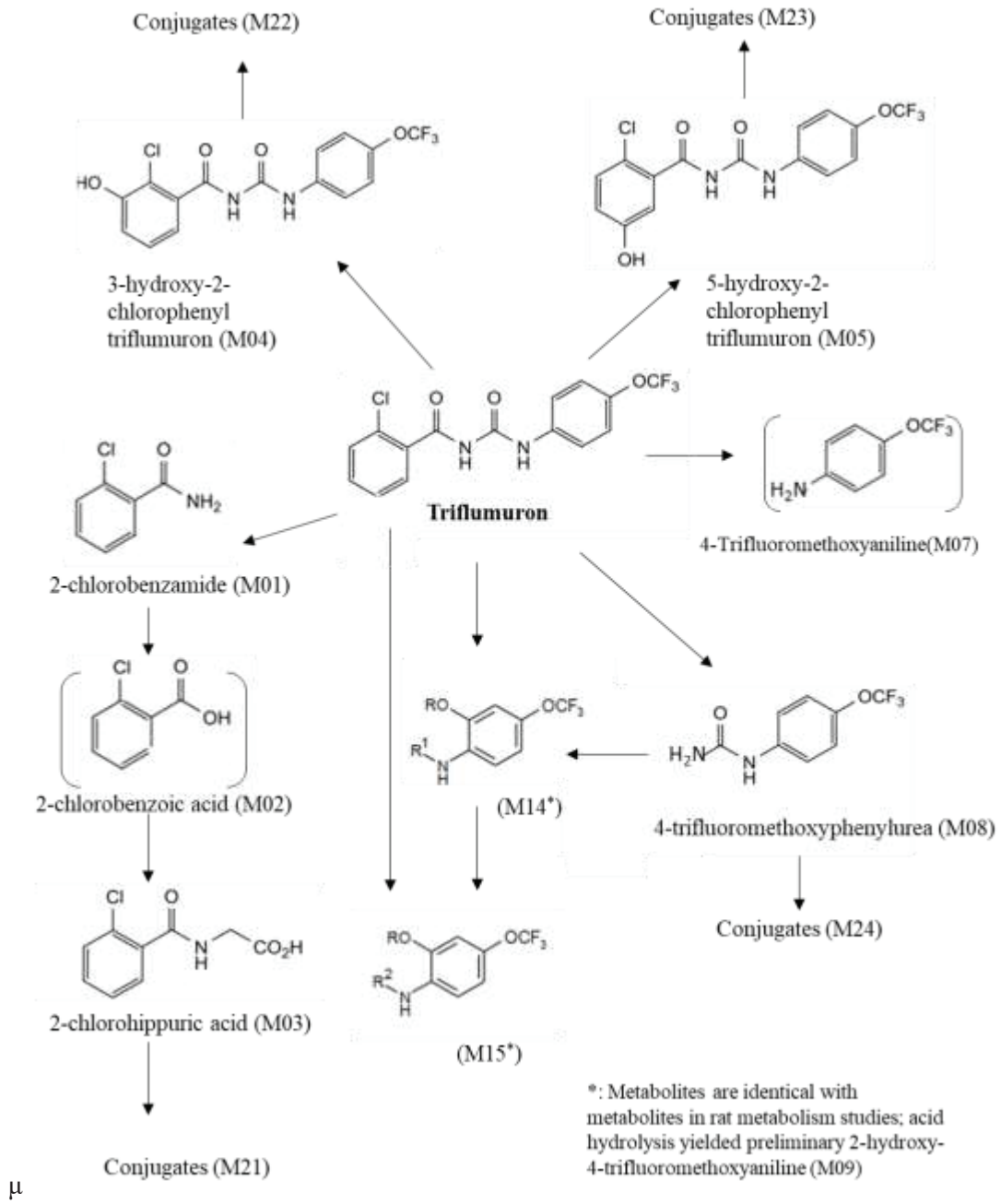
[a] determined by TLC; It was found that 14.3% could be hydrolysed under acidic conditions to form 4-trifluoromethoxyaniline (M07)

[b] determined by TLC; these samples were not hydrolysed with acid, but TLC behaviour was very similar to the polar liver metabolites

[c] determined by TLC

[d] determined from acid hydrolysis that 29.9% could be converted to 4-trifluoromethoxyaniline (M07) with 14.7% associated with the aqueous fraction

<LOD (< 0.001 mg eq/kg)



μ

Figure 3 Proposed metabolism pathway for triflumuron in lactating goat

Laying hens

The Meeting received a study on excretion and metabolism of triflumuron by laying hens using [2-chlorophenyl-UL-^{14}C] triflumuron (Obrist & Sietsema, 1983, 86243, M-074584-01-1).

The study involved three groups of laying hens (white Leghorn, approximately 10 months old and weighing an average of 1.43 kg):

Group A: Five test animals received a single oral dose of 2.5 mg/kg bw (in gelatine capsule) for determination of the excretion pattern (33 ppm assuming that the feed intake is 110 g/bird/day). Eggs and excreta of each individual test animal were collected 3, 6, 9, 24, 48, 72, and 96 hours after dosing. Eggs, if available, were collected every morning throughout the study. Animals were

sacrificed 4 days after dosing, and the following tissues collected: liver, kidney, muscle (composite of white and dark muscle from the breast and leg), skin, fat composite, gizzard muscle and heart.

Group B: Three test animals received a single oral dose of 2.5 mg/kg bw for blood level determination. Blood samples were taken by heart puncture from each animal at 0.5, 1, 1.5, 2, 3, 4 and 5 hours after dosing, and the radioactive residue concentration determined in plasma. Radioactive residues in plasma were seen to vary considerably from bird to bird, however the average plasma level peaked at 3 hours after dosing.

Group C: Three test animals received five daily oral doses of 8.0 mg/kg bw for investigation of tissue residues (104 ppm assuming that the feed intake is 110 g/bird/day). Excreta and eggs were collected each day just before administration of the daily dose. Animals were sacrificed 3 hours after the final dose, and the following tissues collected: liver, kidney, muscle (composite of white and dark muscle from the breast and leg), skin, fat composite, gizzard muscle and heart.

Eggs of group A and C were collected 24, 48, 72 and 96 hours after administration and radioactive residues was measured (Table 56).

Table 56 Total radioactive residues (mg/kg, expressed as triflumuron) in eggs after administration of single or multiple oral doses of [2-chlorophenyl-UL-^{14}C] triflumuron

Interval from first dosing (hours)	Group A 1 × 2.5 mg/kg bw	Group C 5 × 8.0 mg/kg bw
24	< 0.002	0.025
48	0.019	0.206
72	0.067	0.274
96	0.075	0.662

Liver and kidney were extracted with methanol. Muscles, including gizzard muscle and heart, and eggs were extracted with acetone and dichloromethane. Fat and skin were extracted with hexane and acetonitrile. Radioactivity was also measured in two composite samples of eggs (96-hour sample of three birds of group A and 96-hour sample of three birds of group C), as well as in the mentioned tissues and organs at sacrifice. Following oral administration of triflumuron to hens, excretion was rapid and extensive, amounting to 94.1% of the total administered radioactivity at termination. Most of the administered radioactivity (93.9%) was recovered in the excreta, and only an additional 0.2% in the eggs. At sacrifice (4 days after the first dose), relative tissue residue levels were: fat > skin > liver > kidney > muscle. Fat and skin contained the highest residues, while breast and leg muscle contained the lowest residues (Table 57).

Table 57 Total radioactive residues (mg/kg, expressed as triflumuron) in hen tissues after administration of single or multiple oral doses of [2-chlorophenyl-UL-^{14}C] triflumuron

Matrix	Group A 1 × 2.5 mg/kg bw	Group C 5 × 8.0 mg/kg bw
Fat	0.611	26.56
Skin	0.222	13.79
Liver	0.144	7.27
Kidney	0.046	3.08
Leg muscle	0.012	0.98
Breast muscle	0.007	0.61

Following administration of a single dose of 2.5 mg/kg bw (Group A), total radioactive residues in eggs were 0.019 mg eq/kg, 0.067 mg eq/kg, and 0.075 mg eq/kg at 48, 72 and 96 hours, respectively. The values of the multiple dose study at 8.0 mg/kg bw (Group C) were higher (0.025 mg eq/kg, 0.206 mg eq/kg, 0.274 mg eq/kg and 0.662 mg eq/kg at 24, 48, 72 and 96 hours after administration of the first dose, respectively).

Identification of the significant components extracted from kidneys was undertaken by TLC and HLPC co-chromatography with authentic standards. Following isolation, methylation and purification, N,N'-2-chloro-dibenzoyl ornithine (M11) was identified by mass spectroscopy, which was considered to be conjugation of 2-chlorobenzoic acid and ornithine. The compound was also detected in excreta.

Analyses of tissues collected 3 hours after administration of the last of five daily doses resulted in the identification of 85% to 98% of the TRR in all tissues investigated, while 89% to 94% of the egg residue was identified. Parent triflumuron was found in all samples, with the highest amounts being detected in fat. Triflumuron accounted for 58.8% of the TRR in kidney, 85.5% in liver, 86.2% in eggs (multiple dose study), 90.9% in muscle, 97.2% in skin, and 97.4% in fat. In muscle and eggs, the residue of parent triflumuron ranged from 0.57–0.73 mg/kg, whereas the residues in fat and skin reached values of 13.40-25.87 mg/kg. The only metabolites detected were 2-chlorobenzoic acid (M02) and 2-chlorobenzamide (M01). 2-Chlorobenzoic acid (M02) was present in free and conjugated forms, whereas 2-chlorobenzamide (M01) was found only in its free form. Detailed information is given in Tables 58. 59 and 60. Proposed metabolism in laying hen is shown in Figure 4.

Table 58 Distribution of [2-chlorophenyl-UL-^{14}C] triflumuron and its metabolites in the muscle, fat and skin of laying hens (Group C: 5 × 80 mg/kg bw)

	Muscle [b]		Fat		Skin	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
TRR		0.80		26.56		13.79
Extract	98.8	0.79	98.6	26.19	98.6	13.60
Triflumuron	90.9	0.73	97.4	25.87	97.2	13.40
2-chlorobenzamide (M01)	6.3	0.05	0.3	0.08	0.5	0.07
2-chlorobenzoic acid (M02)	0.3	< 0.01	< 0.1	< 0.03	0.1	0.01
Polar metabolites [a]	1.0	0.01	0.8	0.21	0.7	0.10
Origin [a]	0.3	< 0.01	0.1	0.03	0.1	0.01
Hexane phase	0.1	< 0.01	1.4	0.37	< 0.1	< 0.01
Water-soluble	0.1	< 0.01	< 0.1	< 0.03	< 0.1	< 0.01
Unextracted	1.0	0.01	< 0.1	< 0.03	1.4	0.19
Total	100.0	0.80	100.0	26.56	100.0	13.79
Sum identified	97.5	0.78	97.7	25.95	97.8	13.49

[a] characterized by TLC and hydrolysis investigations

[b] muscle composite (TRR value was estimated as the mean of the single values of breast and leg muscle)

Table 59 Distribution of [2-chlorophenyl-UL-^{14}C] triflumuron and its metabolites in the kidney and liver of laying hens (Group C: 5 × 80 mg/kg bw)

	kidney		Liver	
	%TRR	mg eq/kg	%TRR	mg eq/kg
TRR		3.08		7.27
Extract	77.8	2.40	95.8	6.96
Triflumuron	58.6	1.80	85.5	6.22
2-chlorobenzamnide (M01)	4.4	0.14	8.9	0.65
2-chlorobenzoic acid	13.7	0.42	0.3	0.02
- Free acid (M02)	9.5	0.29	0.3	0.02
- Ornithine conjugate (M11)	1.7	0.05		
- Other conjugates	2.5	0.08		
Polar metabolites [a]	0.6	0.02	0.6	0.04
Origin [a]	0.5	0.02	0.5	0.04
Hexane phase	13.6	0.42	0.8	0.06

	kidney		Liver	
	%TRR	mg eq/kg	%TRR	mg eq/kg
Triflumuron	0.2	0.01		
2-chlorobenzamide (M01)	0.1	< 0.01		
2-chlorobenzoic acid	12.8	0.39		
Free acid (M02)	8.4	0.26		
Ornithine conjugate (M11)	1.7	0.05		
Other conjugates	2.7	0.08		
Polar metabolites [a]	0.5	0.02		
Origin				
Water-soluble	6.8	0.21	0.1	0.01
Unextracted	1.8	0.06	3.3	0.24
Total	100.0	3.08	100.0	7.27
Sum identified	84.6	2.61	94.7	6.88

[a] Characterized by TLC and hydrolysis investigations

Table 60 Distribution of [2-chlorophenyl-UL-^{14}C] triflumuron and its metabolites in the eggs of laying hens (eggs at 96 hours after dosing)

	Group A 1 × 2.5 mg/kg bw		Group C 5 × 8.0 mg/kg bw	
	%TRR	mg eq/kg	%TRR	mg eq/kg
TRR		0.075		0.662
Extract	91.7	0.069	95.2	0.630
Triflumuron	85.2	0.064	86.2	0.571
2-chlorobenzamnide (M01)	3.3	0.002	7.6	0.050
2-chlorobenzoic acid (M02)	-	-	-	-
Polar metabolites [a]	2.2	0.002	0.9	0.006
Origin [a]	1.0	0.001	0.5	0.003
Hexane phase	2.3	0.002	0.6	0.004
Water-soluble	0.8	0.001	0.4	0.003
Unextracted	5.2	0.004	3.8	0.025
Total	100.0	0.075	100.0	0.662
Sum identified	88.5	0.066	93.8	0.621

[a] Characterized by TLC and hydrolysis investigations

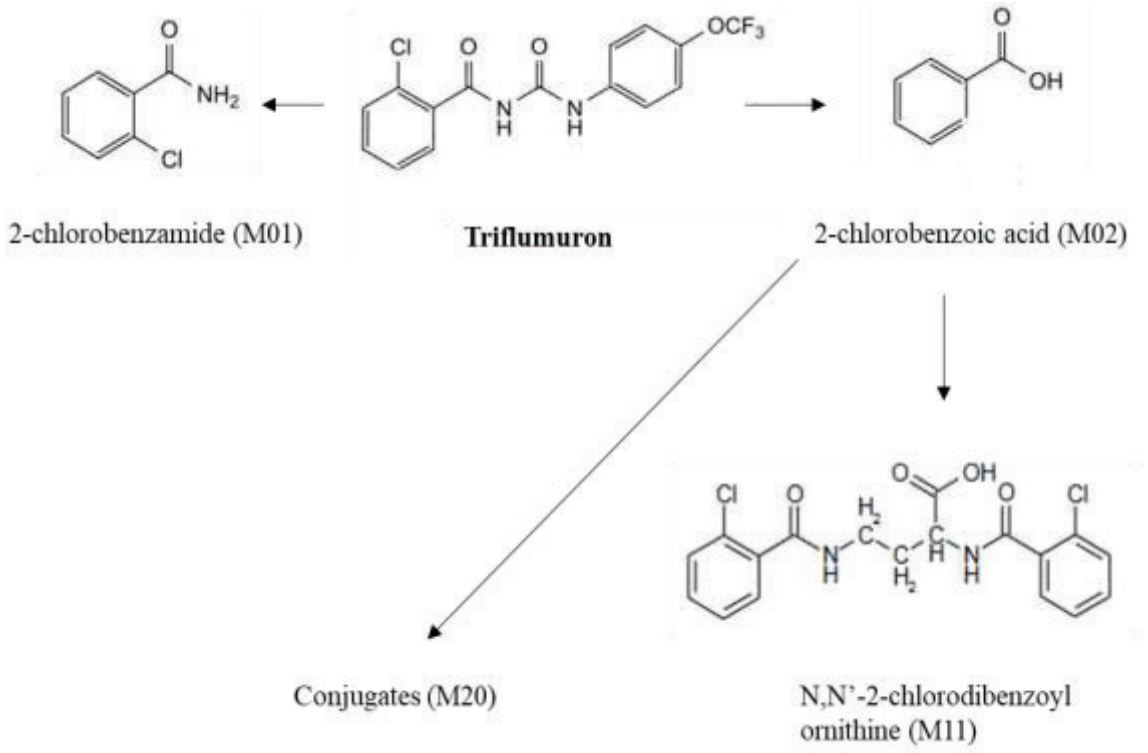


Figure 4 Proposed metabolic pathway for triflumuron in laying hens

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received three methods of analysis for plant commodities and two methods for animal commodities for supervised residue trials and animal feeding studies. Conversion to triflumuron equivalents for 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) were calculated by multiplying by a conversion factor of 2.0251 and 1.6294, respectively.

Methods for plants

Method 00722/M002 (Bomke) (2011, MR-10/048, M-398587-01-1)

Triflumuron residues are extracted from sunflower seed samples (5 g) by soaking with acetonitrile/hexane (3:2 v/v, 50 mL) and then blending. The extracts are filtered and transferred to a separator funnel for phase separation. The lower acetonitrile phase is collected and evaporated to approximately 5 mL. The samples are diluted with acetonitrile (5 mL) and then water (to 25 mL final volume). Final determination is by LC-MS/MS with C18 column (Water/acetonitrile containing 1.0 mL/L of acetic acid, 81:19→9:91) using external matrix-matched standards. MRM transitions: Triflumuron: m/z 357→154 (quantification) and m/z 357→85 (confirmation). LOQ is 0.01 mg/kg for triflumuron (RSD ≤ 9%). The correlation between the injected amount of substances and the detector response was linear for solvent standards between 0.125 µg/L to 500 µg/L for triflumuron (R^2> 0.98). The summary data is shown in Table 61.

Table 61 Method validation data for Method 00722/M002 (Bomke)

Matrix	Analyte	Fortification mg/kg	n	Quantification		Confirmation	
				Recovery range mg/kg (mean)	RSD %	Recovery range mg/kg (mean)	RSD %
Sunflower seed	Triflumuron	0.01	5	74-92 (86)	9	75-94 (87)	8
		0.10	5	79-89 (84)	6	79-89 (83)	6

00722/M002 with modifications 1 and 2 (Harbin) (2017, RASIN006, M-606533-01-1)

Triflumuron residues are extracted from soya bean seed (5 g) by soaking and then blending with acetonitrile (30 mL). The extracts are filtered under vacuum and the volume adjusted to 50 mL with acetonitrile. Final determination is by LC-MS/MS with C18 column (water/acetonitrile containing 1.0 mL/L acetic acid; 85.5:14.5→9:91) using external matrix-matched standards. MRM transitions: Triflumuron: m/z 357→154 (quantification) and m/z 357→85 (confirmation); 4-trifluoromethoxyaniline (M07): m/z 178→93 (quantification) and m/z 178→75 (confirmation) ; and 4-trifluoromethoxyphenyl urea (M08): m/z 219→176(quantification) and m/z 219→85 (confirmation). LOQs are 0.01 mg/kg for triflumuron (RSD ≤ 10%), and 4-trifluoromethoxyaniline (M07; 0.01 mg eq/kg; RSD ≤ 16%) and 4-trifluoromethoxyphenyl urea (M08; 0.008 mg eq/kg; RSD ≤ 11%). The correlation between the injected amount of substances and the detector response was linear for solvent standards of 0.5 µg/L to 100 µg/L triflumuron and its metabolites analysed (R^2> 0.99). The summary data is shown in Table 62.

Table 62 Method validation data for Method 00722/M002 with modifications 1 and 2 (Harbin)

Matrix	Analyte	Fortification mg/kg	N	Quantification	
				Recovery range mg/kg (mean)	RSD %
Soya bean seed	Triflumuron	0.01	13	86-113 (98)	9
		0.10	11	73-108 (97)	10
		0.20	5	89–99 (95)	4
	4-trifluoromethoxyaniline (M07)	0.005	13	79–96 (91)	6
		0.05	6	89–95 (92)	3
		0.10	5	69-107 (94)	16
	4-trifluoromethoxyphenyl urea (M08)	0.005	13	79–97 (89)	6
		0.05	6	72-95 (88)	9
		0.10	5	71-93 (88)	11

00722/M002 with modifications 1 and 2 (Brungardt) (2017, RASIN007, M-599624-01-1)

Triflumuron residues are extracted from seed or processed commodities (5 g) by blending with acetonitrile/hexane (3:2 v/v, 50 mL). The extracts are filtered and transferred to a separator funnel for phase separation. The acetonitrile phase is collected and evaporated to approximately 5 mL (2 mL for aspirated grain fractions). The samples are diluted with acetonitrile (5 mL, or 2 mL for aspirated grain fractions) and then water (to 25 mL, or 10 mL for aspirated grain fractions). Final determination is by LC-MS/MS with C18 column (water/acetonitrile containing 1.0 mL/L acetic acid; 85.5:14.5→9:91)

using external matrix-matched standards. MRM transitions: Triflumuron: m/z 357→154 (quantification) and m/z357→85 (confirmation); 4-trifluoromethoxyaniline (M07): m/z 178→93 (quantification) and m/z 178→75 (confirmation); and 4-trifluoromethoxyphenyl urea (M08): m/z 219 →176(quantification) and m/z 219→85 (confirmation). LOQs for triflumuron are 0.01 mg/kg in soya bean seed (RSD ≤ 10%) and processed commodities (RSD ≤ 18%), and 0.10 mg/kg for aspirated grain fractions (RSD ≤ 5%). LOQs are 0.005 mg/kg for 4-trifluoromethoxyaniline (M07; 0.01 mg eq/kg; RSD ≤ 17%) and 4-trifluoromethoxyphenyl urea (M08; 0.008 mg eq/kg; RSD ≤ 13%) in soya bean seed, processed commodities and aspirated grain fractions. The correlation between the injected amount of substances and the detector response was linear for solvent standards between 0.5 µg/L to 50 µg/L for triflumuron and its metabolites analysed (R^2> 0.99). The summary data is shown in Table 63.

Table 63 Method validation data for Method 00722/M002 with modifications (Brungardt)

Matrix	Analyte	Fortification mg/kg	n	Quantification	
				Recovery range mg/kg (mean)	RSD %
Soya bean seed	Triflumuron	0.01	7	73-96 (85)	10
		0.2	3	93-101 (96)	4
	4-trifluoromethoxyaniline (M07)	0.005	7	82-102 (87)	9
		0.1	3	87-94 (91)	4
	4-trifluoromethoxyphenyl urea (M08)	0.005	7	82-106 (95)	9
		0.1	3	92-120 (105)	13
Soya bean aspirated grain fractions	Triflumuron	0.1	7	102-115 (108)	5
		90	3	88-91 (89)	1
	4-trifluoromethoxyaniline (M07)	0.005	3	72-99 (84)	17
		0.05	3	71-80 (76)	6
	4-trifluoromethoxyphenyl urea (M08)	0.005	3	81-101 (93)	11
		0.05	3	97-108 (105)	6
Soya bean oil, cold pressed	Triflumuron	0.2	3	76-102 (93)	16
	4-trifluoromethoxyaniline (M07)	0.1	3	95-99 (98)	3
	4-trifluoromethoxyphenyl urea (M08)	0.1	3	92-98 (94)	3
Soya bean oil, solvent extracted	Triflumuron	0.01	3	85-103 (92)	11
	4-trifluoromethoxyaniline (M07)	0.005	3	84-100 (91)	9
	4-trifluoromethoxyphenyl urea (M08)	0.005	3	92-96 (94)	2
Soya bean flour	Triflumuron	0.01	3	75-102 (85)	18
	4-trifluoromethoxyaniline (M07)	0.005	3	83-91 (87)	5
	4-trifluoromethoxyphenyl urea (M08)	0.005	3	95-104 (99)	5
Soya bean hulls	Triflumuron	0.01	3	95-100 (98)	3
		0.6	3	93-108 (100)	8

Matrix	Analyte	Fortification mg/kg	n	Quantification	
				Recovery range mg/kg (mean)	RSD %
	4-trifluoromethoxyaniline (M07)	0.005	3	76-79 (77)	2
		0.3	3	88-90 (89)	1
	4-trifluoromethoxyphenyl urea (M08)	0.005	3	74-92 (82)	11
		0.3	3	89-100 (94)	6
Soya bean meal	Triflumuron	0.01	3	90-103 (96)	6
	4-trifluoromethoxyaniline (M07)	0.005	3	78-87 (82)	6
	4-trifluoromethoxyphenyl urea (M08)	0.005	3	78-94 (85)	10
Soya bean milk	Triflumuron	0.01	5	97-110 (104)	6
	4-trifluoromethoxyaniline (M07)	0.005	5	80-89 (84)	4
	4-trifluoromethoxyphenyl urea (M08)	0.005	5	84-101 (92)	6

Method for animal commodities

73295 (Waggoner & Bowman, 1986, 73295, M-030322-01-1)-Milk

Triflumuron residues are extracted from milk (10 g) by shaking with acetone (20 mL), the mixture is centrifuged, and the milk solids discarded. The liquid phase is shaken with dichloromethane (25 mL), and after phase separation the upper aqueous layer is discarded and the lower organic phase is filtered through sodium sulphate. The eluate is concentrated to 3-5 mL by distillation under a Snyder column, and then evaporated to dryness using a water bath. The residue is dissolved in dichloromethane (15 mL) and subjected to clean-up on a silica gel column, eluting with dichloromethane. The eluate is evaporated to 1 mL by distillation under a 3-ball Snyder column, and then to dryness by warming using a steam bath. The residue is redissolved in methanol (0.5 mL) for analysis by HPLC-UV (240 nm) with C18 column (methanol/water 85:15, v/v). The LOQ for triflumuron in milk is 0.01 mg/kg (RSD $\leq$ 4%). Information on the linearity of correlation between the injected amount of substances and the detector response was not available. The summary data is shown in Table 64.

Muscle, liver and kidney

Triflumuron residues are extracted from muscle, liver and kidney (20 g) by Soxhlet extraction for 15 hours with dichloromethane/methanol (9:1 v/v, 130 mL). The extracts are concentrated to 5-10 mL by distillation under a 3-ball Snyder column, and then evaporated to dryness under vacuum (40 mm Hg) using a steam bath. The residue is dissolved in hexane (5 mL), warming to 60 °C if needed, and subjected to partitioning between hexane and acetonitrile. The resulting acetonitrile extract is mixed with 20% aqueous NaCl and partitioned twice against hexane. The hexane extracts are filtered through sodium sulphate, concentrated to near dryness using a Snyder column, and then evaporated to dryness under vacuum using a hot water bath. The residue is dissolved in dichloromethane (15 mL) and subjected to clean-up on a silica gel column, eluting with dichloromethane. The eluate is evaporated to 1 mL by distillation under a 3-ball Snyder column, and then to dryness by warming using a steam bath. The residue is redissolved in methanol (1 mL) for analysis by HPLC-UV (240 nm) with C18 column (methanol/water 85:15, v/v). The LOQ for triflumuron is 0.05 mg/kg (RSD $\leq$ 6%). Information on the linearity of correlation between the injected amount of substances and the detector response was not available. The summary data is shown in Table 64.

Fat

Triflumuron residues are extracted from fat (20 g) by Soxhlet extraction for 15 hours with dichloromethane/methanol (9:1 v/v, 130 mL). The extracts are filtered, the extraction flask is washed with dichloromethane (2 × 10 mL) and the washings filtered. The combined extract is concentrated by distillation under a Snyder column, hexane (25 mL) is added to the flask through the column and the extract concentrated again to approximately 5 mL. The resulting extract is diluted with hexane and partitioned against acetonitrile. The resulting acetonitrile extract is mixed with 20% aqueous NaCl and partitioned twice against hexane. The hexane extracts are filtered through sodium sulphate, and subjected to clean-up on a silica gel column, discarding the hexane eluate after loading the column and eluting with dichloromethane. The dichloromethane eluate is evaporated to 1 mL by distillation under a Snyder column, and then to dryness using a water bath (50 °C). The residue is redissolved in methanol (1 mL) for analysis by HPLC-UV (240 nm) with C18 column (methanol/water 85:15, v/v). The LOQ for triflumuron is 0.1 mg/kg (RSD ≤ 3%). Information on the linearity of correlation between the injected amount of substances and the detector response was not available. The summary data is shown in Table 64.

Table 64 Method validation data for Method 73925

Matrix	Analyte	Fortification mg/kg	n	Quantification	
				Recovery range mg/kg (mean)	RSD %
Bovine fat	Triflumuron	0.1	3	86-91 (88)	3
Bovine kidney	Triflumuron	0.05	4	74-78 (76)	2
Bovine liver	Triflumuron	0.05	4	93-97 (96)	2
Bovine muscle	Triflumuron	0.05	4	80-91 (86)	6
Bovine milk	Triflumuron	0.01	4	74-82 (78)	4

00757 (Heinemann, 2002, MR-118/02, M-066485-01-1)

Triflumuron residues are extracted from 5 g of animal commodities (fat, kidney, liver, meat and milk) by soaking in acetonitrile/n-hexane (3:2 v/v, 50 mL) and then blending. The extracts are filtered and transferred to a separator funnel for phase separation. The hexane layer is discarded, and the acetonitrile phase is evaporated to an aqueous remainder. After dilution with water, the extract is cleaned up using a Chromabond XTR SPE column, eluting with cyclohexane/ethyl acetate (85:15 v/v). The resulting extract is rotary evaporated to dryness and re-dissolved in acetonitrile/water (1:1 v/v). Final determination is by LC-MS/MS using external matrix-matched standards. MRM transitions: m/z 357→154 (quantification) and m/z 357→176 (confirmation). LOQ is 0.005 mg/kg (RSD ≤ 17%). The correlation between the injected amount of substances and the detector response was linear for solvent standards showed R^2> 0.99 between 0.005 mg/kg to 0.5 mg/kg for all matrices. Summary is shown in Table 65.

Table 65 Method validation data for Method 00757

Matrix	Analyte	Fortification mg/kg	N	Quantification		Confirmation	
				Recovery range mg/kg (mean)	RSD %	Recovery range mg/kg (mean)	RSD %
Bovine fat	Triflumuron	0.005	5	76-112 (88)	17	74-107 (86)	15
		0.05	5	77-95 (87)	8	77-96 (87)	9
Bovine	Triflumuron	0.005	5	71-94 (87)	11	69–96 (86)	12

Matrix	Analyte	Fortification mg/kg	N	Quantification		Confirmation	
				Recovery range mg/kg (mean)	RSD %	Recovery range mg/kg (mean)	RSD %
kidney							
		0.05	5	87-111 (96)	10	87-112 (96)	11
Bovine liver	Triflumuron	0.005	5	73-95 (86)	10	71-93 (85)	10
		0.05	5	73-98 (84)	11	72-98 (84)	11
Bovine meat	Triflumuron	0.005	5	81-93 (87)	5	76-91 (86)	7
		0.05	5	76-102 (85)	12	78-104 (87)	12
Bovine milk	Triflumuron	0.005	5	95-101 (98)	3	93-110 (101)	7
		0.05	5	86-93 (90)	3	84-92 (90)	4

Stability of pesticide residues in stored analytical samples

The stability study of triflumuron residues in sunflower seed as a high oil content crop is available (Bomke, 2013, MR-10/021, M-445454-01-1). Individual samples of sunflower seed (5 g) were fortified with triflumuron at a concentration of 0.1 mg/kg and then stored frozen at ≤-18 °C in amber glass bottles. Samples were analysed after nominal storage intervals of 0, 30, 60, 90, 180, 270, 360, 540 and 720 days. On day 0, five fortified samples per analyte and two control samples were analysed. At each subsequent interval, three fortified samples per analyte, two procedural recoveries per analyte and one control were analysed.

Samples were analysed for triflumuron using method 00722/M002 (Bomke) with a LOQ of 0.01 mg/kg. The samples were extracted by blending with acetonitrile/water (3:2 v/v) and then filtered. After phase separation, the acetonitrile phase was concentrated to an aqueous remainder, and then diluted for analysis by LC-MS/MS. The stored recovery of triflumuron from sunflower seed was 90-109% after frozen storage for between 0 and 707 days (Table 66).

Table 66 Storage stability of triflumuron in sunflower seed

Storage interval (days)	Residues (mg/kg)	Mean (%)	Mean procedural recovery (%)
0	0.107, 0.114, 0.107, 0.106, 0.110	109	110
27	0.093, 0.085, 0.091	90	95
57	0.094, 0.094, 0.097	93	98
91	0.099, 0.093, 0.092	98	100
176	0.095, 0.091, 0.096	94	97
344	0.084, 0.086, 0.087	86	86
534	0.094, 0.096, 0.093	94	88
707	0.119, 0.094, 0.092	102	96

A storage stability study for 4-Trifluoromethoxyaniline (M07) and 4-Trifluoromethoxyphenyl urea (M08) in soya bean seed is available (Li & Neeley, 2017, RASIN008, M-600936-01-1). Untreated soya bean seed samples (5 g) were separately fortified with 4-Trifluoromethoxyaniline (M07) and 4-Trifluoromethoxyphenyl urea (M08) at a concentration of 0.1 mg/kg and then stored frozen at ≤-20 °C in glass bottles. Samples were analysed immediately after fortification (0 day) and after storage intervals of 29, 91, 103, 183 and 365 days. On day 0, five fortified samples per analyte and two control samples were analysed. At each subsequent interval, three fortified samples per analyte, two procedural recoveries per analyte and one control were analysed.

Samples were analysed for 4-Trifluoromethoxyaniline (M07) and 4-Trifluoromethoxyphenyl urea (M08) using method 00722/M002 with modifications 1 and 2 (Harbin) with LOQs of 0.005 mg/kg for 4-Trifluoromethoxyaniline (M07) and 4-Trifluoromethoxyphenyl urea (M08). The stored recovery of 4-Trifluoromethoxyaniline (M07) was 63-78% for the storage intervals between 29 and 365 days (Table 67). The stored recovery of 4-Trifluoromethoxyphenyl urea (M08) was 72-104% up to 365 days (Table 68).

Table 67 Storage stability of 4-Trifluoromethoxyaniline (M07) in soya bean seed following storage at ≤-20 °C

Storage interval (days)	Residues (mg/kg)	Mean (%)	Mean procedural recovery (%)
0	0.100, 0.0998, 0.0982, 0.100, 0.101	100	100
29	0.0658, 0.0644, 0.0632	65	96
91	0.0707, 0.0695, 0.0738	71	100
103	0.0761, 0.0776, 0.0790	78	88
183	0.0623, 0.0653, 0.0673	65	92
365	0.0660, 0.0613, 0.0630	63	88

Table 68 Storage stability of 4-Trifluoromethoxyphenyl urea (M08) in soya bean seed following storage at ≤-20 °C

Storage interval (days)	Residues (mg/kg)	Mean (%)	Mean procedural recovery (%)
0	0.0861, 0.0893, 0.0844, 0.0875, 0.0888	87	88
29	0.0730, 0.0742, 0.0701	72	96
91	0.0930, 0.0900, 0.0938	92	90
103	0.0980, 0.0966, 0.0919	96	75
183	0.0950, 0.0948, 0.0973	96	86
365	0.0998, 0.105, 0.107	104	88

Information on a storage stability study for triflumuron on bovine liver, muscle and milk is available (Freitag, 2003, MR-415/02, M-081062-01-1). Untreated bovine liver, muscle and milk samples were fortified with triflumuron at a concentration of 0.1 mg/kg and then stored frozen at≤-18 °C in glass bottles. Samples were analysed immediately after fortification (0 day) and after storage intervals of 61 days (liver and muscle) or 89 days (milk), and 103 days (liver, muscle and milk). On day 0, for each matrix, five fortified samples and two control samples were analysed. At each subsequent interval, three fortified samples, two procedural recoveries at 0.005 mg/kg (LOQ), two procedural recoveries at 0.1 mg/kg, and one control were analysed. The concurrent recovery experiments for liver on day 0 were repeated on day 29 because of poor recovery values.

Samples were analysed for triflumuron using method 00757 with a LOQ of 0.005 mg/kg. The stored recoveries of triflumuron in bovine liver, bovine muscle and milk during the storage interval of 0–103 days were 79–107%, 95–107% and 63–91%, respectively (Table 69).

Table 69 stability of residues of triflumuron in matrices of animal origin following storage at ≤-18 °C

Matrix	Storage interval (days)	Residues (mg/kg)	Mean (%)	Mean procedural recovery (%)
Bovine liver	0	100, 101, 97, 91, 79	94	-
	29	-	-	88
	61	118, 103, 99	107	91
	103	78, 78, 80	79	70

Matrix	Storage interval (days)	Residues (mg/kg)	Mean (%)	Mean procedural recovery (%)
Bovine muscle	0	99, 94,92, 98, 87	94	-
	61	103, 113, 105	107	95
	103	96, 93, 95	95	84
Milk	0	89, 88, 95, 89, 92	91	-
	89	91, 91, 92	91	92
	103	58, 70, 60	63	81

USE PATTERN

The Meeting received the GAP for soya bean as shown in Table 70. The label provided covers a broader spectrum of uses.

Table 70 Registered uses of triflumuron on soya bean considered by the Meeting

Crop	Country	Formulation	Application					PHI (days)
			Method	Rate (kg ai/ha)	Spray conc. (kg ai/hL)	Water volume (L/ha)	Max number of applications	
Soya bean [a]	Colombia	480 SC	Foliar spray	0.048–0.077	-	-	2 (interval: no less than 15 days)	21

[a] GAP for use against Velvetbean caterpillar (*Anticarsia gemmatalis*)

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received nine supervised trials conducted in 2016 on soya beans in Brazil (Harbin, 2017, RASIN006, M-606533-01-1). Five trials included decline data for the treated plot and four were at harvest trials. Treated plots received two applications of 0.48 kg/L SC formulation of triflumuron at 0.073–0.082 kg ai/ha. The first application was made at BBCH 75-79 and the second at BBCH 79–97 with an application interval of 13–15 days. For the decline trials, duplicate samples of soya bean seed were collected from the last application of 6-7, 12-14, 19-21, 26-28 and 33-35 days. For the trials at harvest, duplicate samples were collected from the last application of 19-21 days. Samples were analysed within 10 months for triflumuron and 4-trifluoromethoxyphenyl urea (M08) and 8 months for 4-trifluoromethoxyaniline (M07).

Residues of triflumuron and its metabolites 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) were analysed by LC-MS/MS using method 00722/M002 with modifications 1 and 2 (Harbin). The LOQs for triflumuron, 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) were 0.01 mg/kg, 0.005 mg/kg (0.01 parent equivalent (pe) mg/kg) and 0.005 mg/kg (0.008 pe mg/kg), respectively. The samples were stored frozen (-20 °C) after collection until extraction and analysis. The maximum periods of storage were 10 months for triflumuron and 4-trifluoromethoxyphenyl urea (M08), and 8 months for 4-trifluoromethoxyaniline (M07).

Residues of triflumuron, 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) in soya bean seed were < 0.010–0.055 mg/kg, < 0.010 pe mg/kg and < 0.008–0.012 pe mg/kg at 19-21 DALA, respectively. The summary is shown in Table 71.

Table 71 Residues in soya beans resulting from supervised trials in Brazil (triflumuron 480 SC formulation)

Soya bens Location, year	Application				DALA	Triflumuron (mg/kg)	M07 (mg/kg)	M08 (mg/kg)
(variety)	No	Interval (days)	Rate (kg ai/ha)	kg ai/hL	(days)	Replicate values (mean)	Replicate values (mean)	Replicate values (mean)
GAP, Colombia	2		0.077		21			
Primavera do Leste,[a] Brazil, 2016 (M7739 Ipro)	2	14	0.0738 0.0750	0.038 0.035	7 13 20 28 35	< 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.010, < 0.01 (< 0.01) < 0.010, < 0.01 (< 0.01) < 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008)
Tamarana, Brazil 2016 (Monsoy 5947)	2	15	0.0759 0.0750	0.035 0.035	7 12 20 28 35	0.024, 0.030 (0.027) 0.024, 0.026 (0.025) 0.012, 0.010 (<u>0.011</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008)
Restinga Seca, Brazil 2016 (NA 5909 RR)	2	15	0.0731 0.0756	0.034 0.035	7 14 21 28 35	< 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008)
Montividiu, Brazil 2016 (Anta 82 RR)	2	15	0.0754 0.0739	0.035 0.036	6 12 19	0.021, 0.018 (0.020) 0.012, 0.016 (0.014) 0.015, 0.014 (<u>0.014</u>)	< 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008)

Soya bens Location, year	Application				DALA	Triflumuron (mg/kg)	M07 (mg/kg)	M08 (mg/kg)
(variety)	No	Interval (days)	Rate (kg ai/ha)	kg ai/hL	(days)	Replicate values (mean)	Replicate values (mean)	Replicate values (mean)
					26 33	0.013, 0.012 (0.012) 0.012, 0. 011 (0.012)	< 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008)
Barreiras, Brazil 2016 (M 8808 IPRO)	2	15	0.0778 0.0816	0.036 0.037	6 13 20 26 34	0.10, 0.13 (0.12) 0.12, 0.10 (0.11) 0.052, 0.050 (<u>0.051</u>) 0.042, 0.050 (0.046) 0.027, 0.034 (0.031)	< 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (<u>≤ 0.01</u>) < 0.01, < 0.01 (< 0.01) < 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008) < 0.008, < 0.008 (< 0.008)
Primavera do Leste,[b] Brazil, 2016 (M7739 Ipro)	2	13	0.0768 0.0769	0.035 0.035	21	0.052, 0.058 (<u>0.055</u>)	< 0.01, < 0.01 (< 0.01)	0.012, 0.013 (0.012)
Londrina, Brazil 2016 (Monsoy 5917)	2	15	0.0790 0.0791	0.035 0.030	19	0.045, 0.050 (<u>0.048</u>)	< 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008)
Uberlândia, Brazil 2016 (SYN1163 RR)	2	15	0.0752 0.0760	0.033 0.034	21	0.015, 0.014 (<u>0.014</u>)	< 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008)
Conchal, Brazil 2016 (BMX Potencia RR)	2	14	0.0767 0.0768	0.037 0.036	21	< 0.01, < 0.01 (<u>≤ 0.01</u>)	< 0.01, < 0.01 (< 0.01)	< 0.008, < 0.008 (< 0.008)

[a], [b] Location 1 and Location 2 are in the same city (Primavera do Leste) but away from each other (34 km) and different climate (Total rainfall in Location 1: 438 mm, Location 2: 59 mm).

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received a hydrolysis study of triflumuron using [phenoxy-UL-^{14}C] triflumuron (Figure 1) under conditions relevant to major food processing operations such as pasteurization (20 minutes at 90 °C, pH 4), baking, brewing and boiling (60 minutes at 100 °C, pH 5) and sterilization (20 minutes at 120 °C, pH 6) (Babczinski, 2002, MR-099/02, Bayer Ref: M-066092-01-1).

[phenoxy-UL-^{14}C] triflumuron was dissolved in buffered drinking water at 0.0234 mg/L. The solution was incubated under conditions representative of pasteurization; baking, brewing and boiling; and sterilization. At zero time and at the termination of the test, the samples were analysed by TLC. The content of radioactivity was determined by LSC. Material balances were established at each sampling time.

At the termination of the test, the material balances were in a range of 84.5-98.4%. After incubation under conditions representative of pasteurization, 98.4% of AR were recovered as triflumuron. The amount recovered as triflumuron decreased to 88.9% in baking, brewing and boiling; and to 51.4% in sterilization (Table 72). 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) were found as degradates.

Table 72 Hydrolysis of triflumuron under conditions of processing studies

Condition	Sampling time (minutes)	Radioactivity in solution (% AR)		
		Triflumuron	4-trifluoromethoxyaniline (M07)	4-trifluoromethoxyphenyl urea (M08)
Pasteurization (90 °C, pH 4, 20 min)	0	96.8	nd	nd
	20	98.4	nd	nd
Baking, brewing, boiling (100 °C, pH 5, 60 min)	0	98.4	nd	nd
	60	88.9	3.4	nd
Sterilization (120 °C, pH 6, 20 min)	2	97.4	nd	nd
	20	51.4	17.3	15.8

nd: < 0.05 µg eq/L

Processing study

Two field trials applied as a foliar spray at exaggerated (5×) rate of 2 × 0.384 kg ai/ha (BBCH 78-79 and BBCH 81-84 with an application interval of 14 days) were conducted in the USA in 2016 (Brungardt, 2017, RASIN007, M-599624-01-1). Soya bean seed was harvested at 19-21 DALA and frozen prior to shipment to the processing facility.

The soya beans were processed in such a way as to simulate industrial practice as closely as possible. Samples from one field trial (SI011-16PA) were dried at 43-57 °C to moisture content of 10-13%. Samples from another trial (SI010-16PA) did not require drying before processing. Samples were batch processed in a dust generation room to separate the aspirated grain fractions, and the soya bean samples cleaned by aspiration and screening (Figure 6).

Cleaned whole soya beans were cracked in a roller mill, and the hulls and kernels separated by passing through an aspirator. After adjustment of the moisture content to 13.5% and tempering for 12 hours or more, kernels were heated to 71-79 °C and flaked in a rolling mill. A portion of the flakes was removed for direct solvent extraction. The remaining flakes were extruded and turned into collets by direct steam injection and compression. Collets were ground in a disc mill before being oven-dried at 65-82 °C for 30-40 minutes. Ground collets and flakes were placed in separate stainless-steel batch extractors and submerged in 49-60 °C hexane. After 30 minutes, the miscella (crude oil and hexane) was drained and the solvent extraction cycle repeat two more times. Miscella from ground collets and flakes were combined and separated into crude oil and hexane using a laboratory vacuum evaporator. Crude oil was heated to 90-96 °C to remove residual hexane and filtered. The crude oil was neutralized with 14° Baumé sodium hydroxide, centrifuged and filtered to give refined oil and

soapstock. The refined oil was bleached and deodorized to give refined, bleached, deodorized (RBD) solvent-extracted oil. Solvent was removed from the solvent-extracted flakes with warm air and ground in an Alpine pin mill. The ground material was sieved, and the material passing through the 62-mesh screen was defatted soy flour. Solvent extracted collets were toasted by steam injection up to 103-106 °C followed by heating at 104-116 °C for 30-60 minutes. After cooling and sieving, the material passing through an 8/64" sieve was toasted meal. Cleaned whole soya beans were washed and soaked in water for a minimum of 12 hours. Soaked beans were ground and filtered to separate the liquid (soya milk) and solids. The liquid was heated to 91-96 °C and held at that temperature for 9-11 minutes to give the final soya milk sample (Figure 7).

Cleaned whole soya beans were adjusted to 16.0% moisture and allowed to equilibrate for 45 minutes. The resulting seed was fed into a mechanical screw press to separate crude oil from the presscake. Crude oil was processed in the same way as the solvent extracted oil, to give refined, bleached, deodorised (RBD) cold pressed oil (Figure 8).

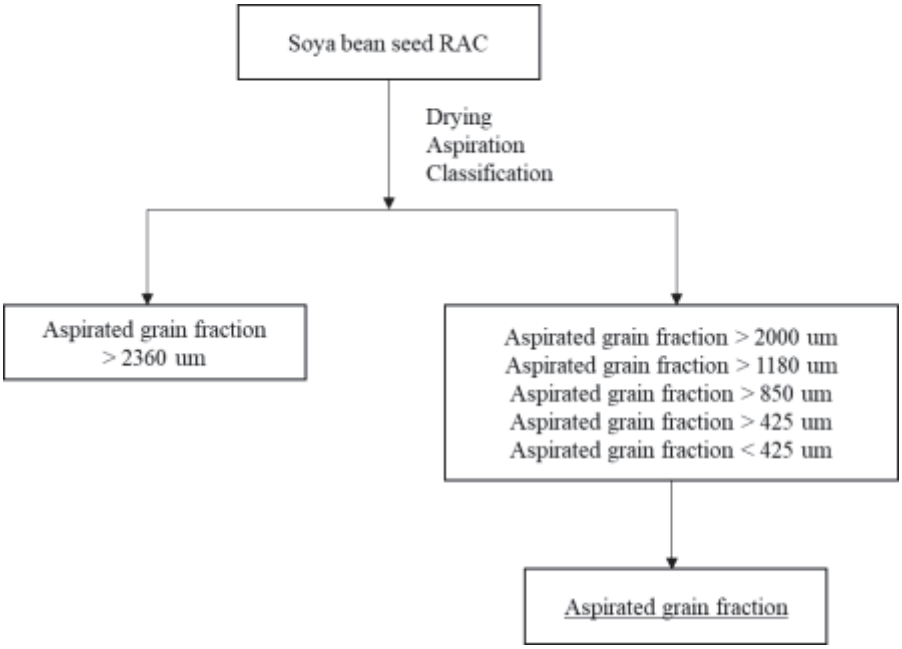


Figure 6 Soya bean processing to aspirated grain fractions

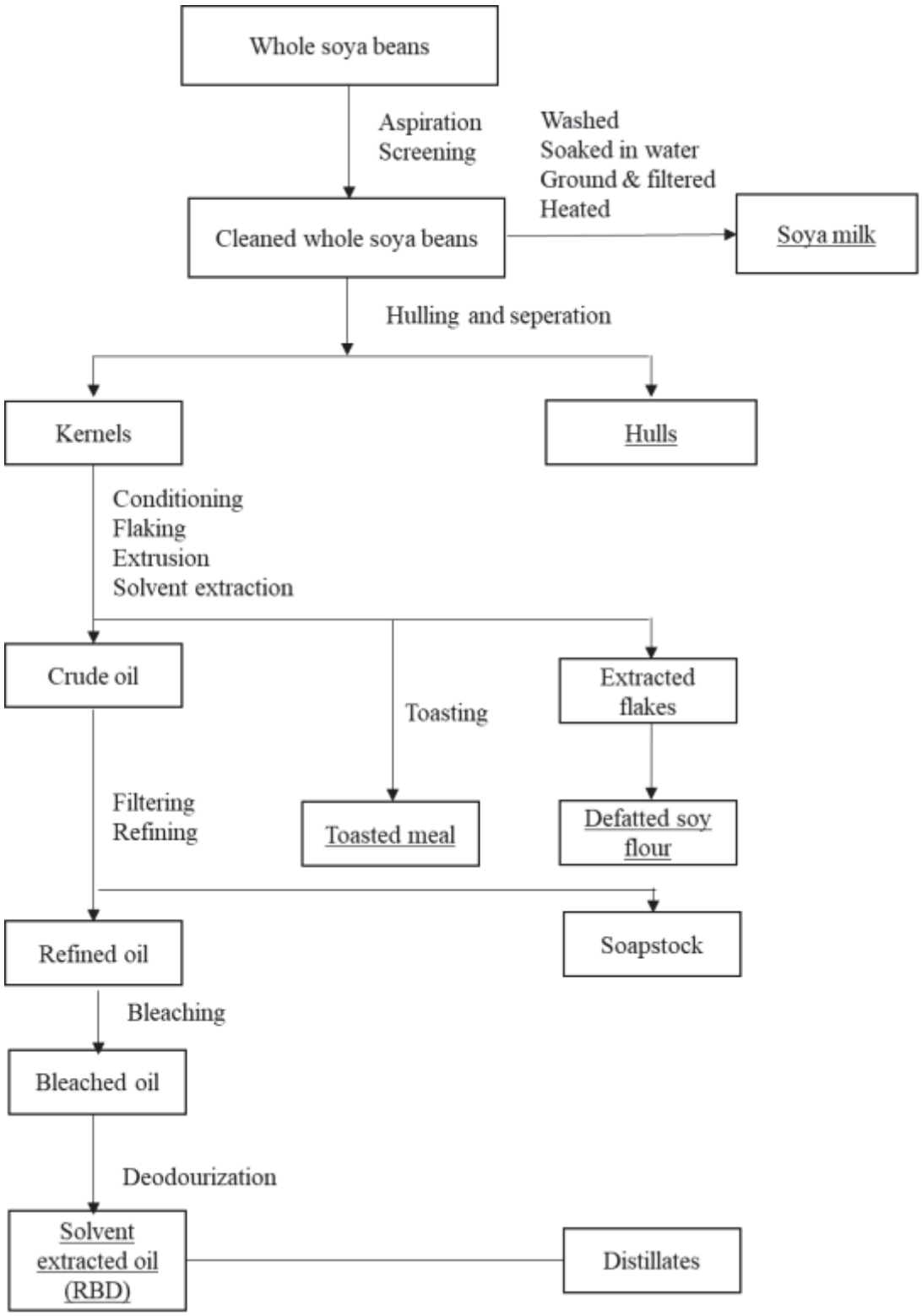


Figure 7 Soya bean processing to hulls, milk, meal, flour and solvent extracted oil

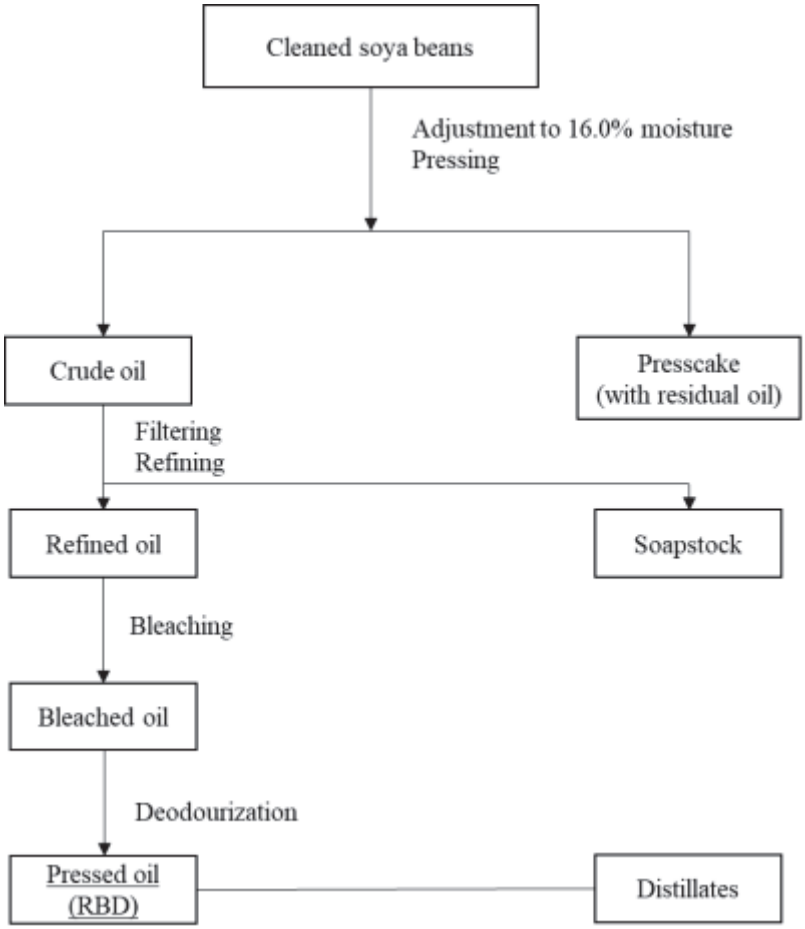


Figure 8 Soya bean processing to cold pressed oil

Residues of triflumuron, 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) in soya bean and its processed commodities were analysed by LC-MS/MS using Method 00722/M002 with modifications (Brungardt). LOQs for triflumuron were 0.01 mg/kg in soya bean, flour, hulls, meal, soya milk and oil, and 0.10 mg/kg in aspirated grain fractions. The LOQs for 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) were 0.005 mg/kg in all commodities.

Procedural recoveries of triflumuron at fortification levels of 0.01–0.20 mg/kg in seed and oil, 0.01–0.60 mg/kg in hulls, 0.01 mg/kg in meal, flour and milk and 0.1-90 mg/kg in aspirated grain fractions were 70-120% with RSD < 20%. Procedural recoveries of 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) at fortification levels of 0.005–0.10 mg/kg (0.01–0.20 mg eq/kg for M07 and 0.01–0.20 mg pe/kg for M08) in seed oil, 0.005 mg/kg (0.01 mg eq/kg for M07 and 0.008 mg/kg for M08) in meal, flour and milk and 0.005–0.05 mg/kg (0.01–0.10 mg eq/kg for M07 and 0.008–0.08 mg eq/kg for M08) in aspirated grain fractions were 70-110% with RSD < 20%.

The samples were frozen after collection and stored frozen (≤-12 ºC) until extraction and analysis. The maximum period of frozen storage was 180 days (6 months). Residue levels of triflumuron were <LOQ (< 0.01 mg/kg) in soya bean meal, flour, milk, solvent extracted oil and cold pressed oil. Concentration of triflumuron residues occurred in hulls with processing factors of 3.3-3.6, and in aspirated grain fractions with processing factors of 370-615. Residues of 4-trifluoromethoxyaniline (M07) and 4-trifluoromethoxyphenyl urea (M08) were <LOQ (< 0.005 mg eq/kg) in soya bean seed and all processed commodities except aspirated grain fractions, where low residues were detected (Table 73).

Table 73 Residues in soya beans and processed products (triflumuron 480 SC formulation)

Soya beans Location, year	Application				Processed products	Triflumuron (mg/kg)		M07 (mg/kg)	M08 (mg/kg)
(variety)	No	Rate (kg ai/ha)	kg ai/hL	DALA (days)		Residue (mg/kg)	Processing factor	Residue [a] (mg/kg)	Residue [a] (mg/kg)
Stewardson, IL USA, 2016 (93Y84)	2 (14)	0.388 0.387	0.18 0.18	19	Soya bean (RAC)	0.13		< 0.005	< 0.005
					Aspirated grain fraction	80	615	0.0060	0.048
					Hulls	0.47	3.6	< 0.005	< 0.005
					Meal	< 0.01	< 0.1	< 0.005	< 0.005
					Flour	< 0.01	< 0.1	< 0.005	< 0.005
					Soya milk	< 0.01	< 0.1	< 0.005	< 0.005
					Solvent extracted oil (RBD)	< 0.01	< 0.1	< 0.005	< 0.005
					Cold pressed oil (RBD)	< 0.01	< 0.1	< 0.005	< 0.005
Atlantic, IA USA, 2016 (21LF32)	2 (14)	0.377 0.377	0.19 0.19	21	Soya bean (RAC)	0.027		< 0.005	< 0.005
					Aspirated grain fraction	10	370	< 0.005	0.034
					Hulls	0.088	3.3	< 0.005	< 0.005
					Meal	< 0.01	< 0.4	< 0.005	< 0.005
					Flour	< 0.01	< 0.4	< 0.005	< 0.005
					Soya milk	< 0.01	< 0.4	< 0.005	< 0.005
					Solvent extracted oil (RBD)	< 0.01	< 0.4	< 0.005	< 0.005
					Cold pressed oil (RBD)	< 0.01	< 0.4	< 0.005	< 0.005

[a] Residue levels in RAC are <LOQ (0.005 mg/kg).

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

A cow feeding study was reported to the Meeting (Waggoner, 1986, 73381, M-074857-01-1). Two groups of three Holstein dairy cows were orally dosed once daily with triflumuron in gelatine capsules for 29 consecutive days. One additional cow was maintained as control and received no test compound. The dose rates were 0.3 mg/kg bw per day (equivalent to 5.93 ppm diet) and 0.6 mg/kg bw per day (equivalent to 11.85 ppm diet). The cows, body weight of 608-807 kg (average of 654 kg), were fed with about 33.1 kg of a mixture of ground alfalfa hay, corn silage, brewer's grains, ground ear corn, cottonseed, soya bean meal and a mineral supplement per day. Milk was collected three times daily during the dosing period. Milk samples were analysed on study days of -1 (the day before first administration), 26, 27 and 28. On study day 29, the animals were sacrificed 4 to 8 hours after the last dose administration. Milk, liver, kidney, composite muscle, and composite fat were collected for residue analysis.

Milk samples for analysis were pooled to give one sample per animal per day. Each sample was analysed for triflumuron using method 73295. All samples were analysed within 41 days. The residues in all commodities were below the LOQ (0.01 mg/kg for milk, 0.05 mg/kg for liver, kidney and muscle and 0.1 mg/kg for fat) in all analysed samples (Tables 74 and 75). As milk collected only on 26, 27 and 28 days was analysed and the analytical values were all < 0.01 mg/kg, no plateau was found. For calculation of STMR and HR, the relevant LOQ was used.

Table 74 Residues of triflumuron in whole milk collected during 29 days oral administration to dairy cows

Commodity	Daily dose level (mg/kg bw/day)	Sampling interval (days)	Triflumuron residues	
			Individual animal data (mg/kg)[a]	Average (mg/kg)
Milk	0	-1[b]	< 0.01	< 0.01
		26	< 0.01	< 0.01
		27	< 0.01	< 0.01
		28	< 0.01	< 0.01
	0.3	-1[b]	< 0.01, < 0.01, < 0.01	< 0.01
		26	< 0.01, < 0.01, < 0.01	< 0.01
		27	< 0.01, < 0.01, < 0.01	< 0.01
		28	< 0.01, < 0.01, < 0.01	< 0.01
	0.6	-1[b]	< 0.01, < 0.01, < 0.01	< 0.01
		26	< 0.01, < 0.01, < 0.01	< 0.01
		27	< 0.01, < 0.01, < 0.01	< 0.01
		28	< 0.01, < 0.01, < 0.01	< 0.01

[a] All samples were analysed in duplicate and the mean value is presented for each sample.

[b] Samples were taken on day before first administration.

Table 75 Residues of triflumuron in tissues collected after 29 days of oral administration to dairy cows

Commodity	Daily dose level (mg/kg bw/day)	Sampling interval (days)	Triflumuron residues	
			Individual animal data (mg/kg)	Average (mg/kg)
Liver	0	29	< 0.05	< 0.05
	0.3	29	< 0.05, < 0.05, < 0.05	< 0.05
	0.6	29	< 0.05, < 0.05, < 0.05	< 0.05
Kidney	0	29	< 0.05	< 0.05
	0.3	29	< 0.05, < 0.05, < 0.05	< 0.05
	0.6	29	< 0.05, < 0.05, < 0.05	< 0.05
Muscle	0	29	< 0.05	< 0.05
	0.3	29	< 0.05, < 0.05, < 0.05	< 0.05
	0.6	29	< 0.05, < 0.05, < 0.05	< 0.05
Fat	0	29	< 0.1	< 0.1
	0.3	29	< 0.1, < 0.1, < 0.1	< 0.1
	0.6	29	< 0.1, < 0.1, < 0.1	< 0.1

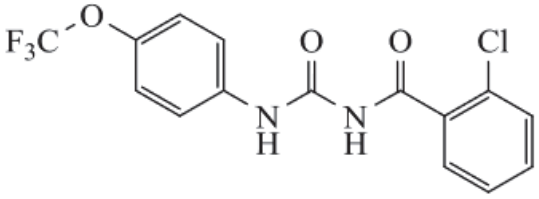
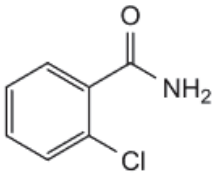
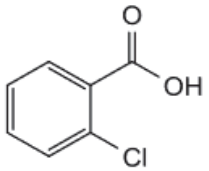
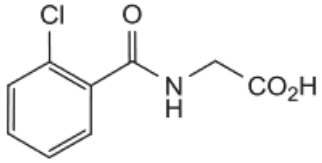
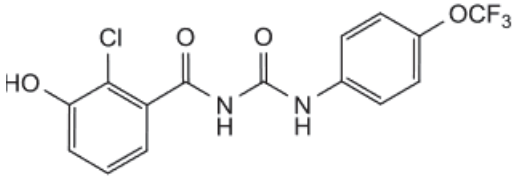
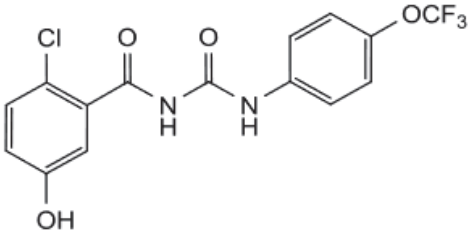
APPRAISAL

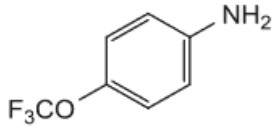
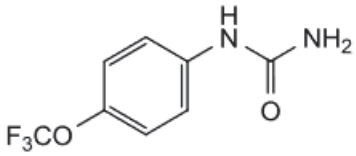
Triflumuron is a benzoylurea insecticide. The mode of action is insect growth regulation by inhibiting the synthesis of chitin in insect larvae that are about to moult and interfering with the moulting hormone system. The IUPAC name for triflumuron is 1-(2-chlorobenzoyl)-3-[4-trifluoromethoxyphenyl]urea.

Triflumuron was scheduled at the Fiftieth Session of CCPR (2018) for evaluation as a new compound by the 2019 JMPR. The Meeting received information on identity, physical and chemical properties, plant and animal metabolism, environmental fate, methods of analysis, use pattern and supervised trials on soya bean, fate of residues in storage and processing and an animal feeding study.

The code numbers, chemical names and chemical structures of the compounds are as follows:

Table 1 Triflumuron and its metabolites referred to in this appraisal

Compound Code number, Chemical name	Structure
Triflumuron	
M01 2-Chlorobenzamide	
M02 2-Chlorobenzoic acid	
M03 2-Chlorohippuric acid	
M04 1-(2-chloro-3-hydroxybenzoyl)-3-[4-trifluoromethoxyphenyl]urea	
M05 1-(6-chloro-3-hydroxybenzoyl)-3-[4-trifluoromethoxyphenyl]urea	

Compound Code number, Chemical name	Structure
M07 4-Trifluoromethoxyaniline	
M08 4-Trifluoromethoxyphenyl urea	

With respect to the physical and chemical properties that may impact on residues in crops, triflumuron is not regarded as volatile and the log P_{ow} is 3.5–3.6.

Plant metabolism

The Meeting received information on the fate of triflumuron in apples after direct treatment or soaking and in tomatoes, soya beans and potatoes after foliar applications. In the studies, triflumuron labelled with ^{14}C at the chlorophenyl group ([2-chlorophenyl-UL-^{14}C] triflumuron) and the 4-trifluoromethoxyaniline group ([4-trifluoromethoxy-phenyl-UL-^{14}C] triflumuron) were used. In the metabolism studies, total radioactive residues (TRR) are expressed in mg triflumuron equivalents/kg (mg eq/kg).

In a translocation study on apples, shoots cut from apple tree were placed into a beaker filled with an aqueous solution of labelled triflumuron. The apple cuttings absorbed 7.5–7.7% of the labelled triflumuron over a 13-day period. In another treatment, ten leaves of apple trees were treated with labelled triflumuron and, 13 days after treatment 0.05% of the applied radioactivity was translocated into upper plant parts.

In a metabolism study on apples conducted over two years, chlorophenyl-label or 4-trifluoromethoxyaniline-label triflumuron was applied topically to three individual apples on a tree under outdoor conditions, at rates equivalent to spray concentrations of 10 g ai/L in the first year and 0.2 or 1.0 g ai/L in the second year. Harvested apples 5–35 DAT were washed with acetone (acetone wash) and peeled. Peel and pulp were extracted with acetone. Surface wash accounted for 90–99% TRR. Most of the radioactivity in apples was present as triflumuron (≥ 98% of TRR at 31 DAT).

In a metabolism study on tomatoes, chlorophenyl-label or 4-trifluoromethoxyaniline-label triflumuron was applied to tomato plants grown in a greenhouse, with two foliar applications (21-day interval) at 0.38–0.39 kg ai/ha. Tomatoes were harvested 7 DALA and thereafter. Ripe tomatoes were washed with dichloromethane and then homogenized and extracted with acetonitrile/water (80:20) and then acetonitrile. Of the total radioactivity for whole tomatoes (1.1–1.4 mg/kg), 97–98% was found in surface wash. Almost 100% TRR in tomato was present as triflumuron.

In a translocation study on soya bean, labelled triflumuron was painted on the leaf surface of soya bean. About 0.02% of the AR was recovered in mature soya beans harvested 101 DAT. When 5 mg of labelled triflumuron was injected into the stem, 0.10–0.11% of the applied radioactivity was recovered in mature soya beans 101 DAT.

In a metabolism study on soya bean, chlorophenyl- or 4-trifluoromethoxyaniline-label triflumuron was applied to soya beans under field conditions at full bloom, with a foliar spray at an application rate of 1.12kg ai/ha. Radioactive residues in foliage decreased over time during the study period (84 mg eq/kg at 0 DAT to 5.8 mg eq/kg at 77 DAT for the chlorophenyl-label; 41 mg eq/kg at 0 DAT to 11 mg eq/kg at 70 DAT for the 4-trifluoromethoxyaniline-label). TRR were higher in foliage (5.8–84 mg eq/kg, 0–77 DAT) and pods (0.4–9.0 mg eq/kg, 0–77 DAT) than in mature beans (0.21–0.31 mg eq/kg, 60–77 DAT).

In mature soya bean seed (60–77 DAT), 29–55% TRR was extracted by methanol, of which 14–15% TRR was partitioned into hexane and 13–41% TRR was in the aqueous phase. The majority of radioactivity was unextracted (45% or 71%). In the hexane phase, triflumuron was predominant (14% TRR, 0.03–0.04 mg eq/kg), followed by M01 (0–0.4% TRR, 0–0.001 mg eq/kg) and M02 (0–0.4% TRR, 0–0.001 mg eq/kg). Acid hydrolysis (6 M HCl, reflux for 16 hours) of PES released further parent triflumuron (2.1% of TRR, 0.004 mg eq/kg) and M02 (30% of TRR, 0.063 mg eq/kg) for the chlorophenyl-label and M07 (33% of TRR, 0.10 mg eq/kg) for the 4-trifluoromethoxyaniline-label.

In foliage and pods of soya bean (0–77 DAT), the major portion of radioactivity was extracted by methanol (73–99% TRR), of which 72–99% TRR and 58–98% TRR, respectively, was partitioned into hexane and 0.4–17% TRR and 2.1–17% TRR, respectively, in the aqueous phase. Triflumuron accounted for the majority of the residue in the hexane phase of foliage and pods at 72–99% TRR and 69–98% TRR, respectively. M01, M02 and M08 were also found (≤ 1.3% TRR). TRR in the water phase was not identified. Acid hydrolysis (6 M HCl, reflux for 16 hours) of the unextracted residue of foliage (60 DAT) released M07 (0.4% TRR).

In a translocation study on potatoes, the leaf surface of four potato plants was treated with chlorophenyl- or 4-trifluoromethoxyaniline-label triflumuron. The potato tubers harvested 42 days post-treatment contained 0.005–0.008% of the AR. Another treatment with chlorophenyl- or 4-trifluoromethoxyaniline-label triflumuron was injected into the stems of two potato plants. Stem injection resulted in low levels of residue in the tubers (up to 0.7% AR, 12 DAT).

In a metabolism study on potatoes, chlorophenyl- or 4-trifluoromethoxyaniline-label triflumuron was applied to potatoes grown outdoors or in the greenhouse, respectively, at bloom as a foliar spray at an application rate of 1.12 kg ai/ha. Radioactive residues in tubers after application of 2-chlorophenyl-label increased from 0.01 mg eq/kg (7 DAT) to 0.08 mg eq/kg (42 DAT) and TRR in foliage or pods (23–98 mg eq/kg between 0–42 DAT) was always higher than that in tubers.

After treatment with 2-chlorophenyl-label triflumuron, in mature potato tubers (42 DAT), acetone and dichloromethane extracted 46–78% TRR, with a further 14% extracted by water. In the organic-solvent extract, triflumuron and M02 accounted for 26% TRR (0.037 mg eq/kg) and 14% TRR (0.011 mg eq/kg), respectively. Hydrolysis of the organic-solvent extract with HCl (1 M, reflux for 6 hours) released further triflumuron (8.3% TRR, 0.007 mg eq/kg). Acid hydrolysis (1 M HCl, reflux for 6 hours) of PES released additional parent triflumuron (15% TRR, 0.012 mg eq/kg), M01 (2.9% TRR, 0.002 mg eq/kg) and M02 (14% TRR, 0.011 mg eq/kg).

After treatment with 4-trifluoromethoxyaniline-label triflumuron, 0.01 mg eq/kg TRR was found in mature potato tubers (42 DAT), from which 78% TRR was extracted with methanol (0.008 mg eq/kg). In the methanol extract, triflumuron (42% TRR, 0.004 mg eq/kg) and M08 (14% TRR, 0.001 mg eq/kg) was identified. Further extraction or hydrolysis was not conducted.

Summary of plant metabolism

Translocation studies on apples, soya bean and potatoes indicated limited translocation of triflumuron within the plant.

When triflumuron was applied to apples and tomatoes, most residues were at the surface of the fruits (>96% TRR in surface wash). When triflumuron was applied to soya beans and potatoes, residues were at lower concentrations in edible parts of the plants than inedible parts. In all of the plants tested, the majority of the triflumuron remained unmetabolized.

Triflumuron is the main residue in apples (>98% TRR, free), tomatoes (>99% TRR, free), soya bean foliage and pods (>69% TRR, free and conjugated), soya bean seeds (16% TRR, 0.03–0.04 mg/kg, free and conjugated), and potato tubers (42 DAT, 42–49% of TRR, free and conjugated).

Major metabolites in soya bean seeds were M02 (30% TRR, 0.063 mg eq/kg) and M07 (33% TRR, 0.10 mg eq/kg), and in potato tubers M08 (14% TRR, 0.001 mg eq/kg) and M02 (14% TRR, 0.011 mg eq/kg) (all metabolites were totals of free and conjugated).

The Meeting concluded that the metabolic profiles between the species were qualitatively similar. All of the plant metabolites identified are also observed in the metabolism in rat.

Environmental fate

Aerobic degradation in soil

Triflumuron is not persistent in soil (DT_{50} 1.7–19 days).

Hydrolysis

Triflumuron is stable to hydrolysis at pH 5 and 7. It is hydrolysed at pH 9 (DT_{50} = 57 days at 25 °C) resulting in M02 at 29% AR after 30 days.

Soil photolysis

Triflumuron is stable to photolysis.

Rotational crop studies (confined)

All the metabolites of triflumuron found in succeeding crops (kale, red beet and wheat) were also found in the plant metabolism studies. At an application rate 7.3 times higher than the maximum seasonal rate in the Colombian GAP, TRR were 0.25–0.66 mg eq/kg at 1-month PBI and decreased to ≤0.08 mg eq/kg at longer PBI (4 or 9 months). At 1-month PBI, M02 (13–20% TRR, 0.03–0.12 mg eq/kg) was predominant, followed by M01 (1.1–20% TRR, 0.01–0.11 mg eq/kg) and triflumuron (1.4–5.7% TRR, < 0.01–0.02 mg eq/kg). The Meeting, however, concluded that it was unlikely that triflumuron used according to the GAP rate would carry over to follow-on crops at longer PBI (4 or 9 months).

Animal metabolism

Information was available on the metabolism of triflumuron in laboratory animals, lactating goats and laying hens. The evaluation of metabolism studies in rats was carried out by the WHO group.

In lactating goats, the metabolic fate of triflumuron was investigated using chlorophenyl- or 4-trifluoromethoxyaniline-label triflumuron.

For the chlorophenyl-label, triflumuron was administered once orally at 3.0 mg/kg bw and then 56 hours later at 25.1 mg/kg bw (equivalent to 170 ppm in the feed, dry matter basis (DM)). The goat was sacrificed 20 hours after the last dose. Following the first dose, approximately 60% of AR was excreted in faeces (54% AR) and urine (6.2% AR) within 56 hours.

The TRR in edible tissues was the highest in liver (3.3 mg eq/kg), followed by fat (1.8 mg eq/kg), kidney (0.87 mg eq/kg) and muscle (0.30 mg eq/kg). In milk, the TRR was 0.43 mg eq/kg.

The tissues and milk were extracted with dichloromethane (milk), hexane (fat) or methanol (liver, kidney and muscle). In milk and fat, 96–99% TRR was extracted. In liver, kidney and muscle, extractability with methanol was 31, 71 and 83% TRR, respectively. Water extracted an additional 4.2, 50, 21 and 7.1% TRR for milk, liver, kidney and muscle.

Parent triflumuron was a major component of the residue representing 75% TRR (0.32 mg eq/kg) in milk, 15% TRR (0.51 mg eq/kg) in liver, 20% TRR (0.18 mg eq/kg) in kidney, 58% TRR (0.18 mg eq/kg) in muscle and 96% TRR (1.8 mg eq/kg) in fat. The following metabolites were identified: M01 (free) in milk, liver, kidney, muscle and fat (0.9–20% TRR, 0.02–0.15 mg eq/kg); M03 (free and conjugated) in milk (6.2% TRR, 0.03 mg eq/kg) and kidney (36% TRR, 0.31 mg eq/kg); M04 (free and conjugated) in milk (4.1% TRR, 0.02 mg eq/kg) and kidney (0.8% TRR, 0.01 mg eq/kg); and M05 (free and conjugated) in liver (4.0% TRR, 0.13 mg eq/kg).

For the 4-trifluoromethoxyaniline-label, triflumuron was administered orally at 18 mg/kg bw (equivalent to 440 ppm in the feed (DM)) for 3 consecutive days with sacrifice 20 hours after the final dose.

The TRR in edible tissues was the highest in liver (6.1 mg eq/kg), followed by fat (4.8 mg eq/kg), kidney (1.6 mg eq/kg) and muscle (0.18 mg eq/kg). In milk collected 20 hours after first application, the TRR was 0.76 mg eq/kg.

The tissues and milk were extracted with dichloromethane (milk), hexane (fat) or methanol (liver, kidney and muscle). In milk, kidney, muscle and fat, 86–98% TRR was extracted. In liver, 52% TRR was extracted. Additional radioactivity was extracted with water (3.0–8.8% TRR in milk, liver, kidney and muscle).

Parent triflumuron was a major component of the residue at 60% TRR (0.45 mg eq/kg) in milk, 20% TRR (1.2 mg eq/kg) in liver, 27% TRR (0.43 mg eq/kg) in kidney, 80% TRR (0.14 mg eq/kg) in muscle and 95% TRR (4.6 mg eq/kg) in fat. The following metabolites were identified: M04 (free and conjugated) in milk (11% TRR, 0.08 mg eq/kg), liver (8.2% TRR, 0.50 mg eq/kg), kidney (28% TRR, 0.45 mg eq/kg), muscle (1.6% TRR, < 0.01 mg eq/kg) and fat (0.9% TRR, 0.04 mg eq/kg); and M08 (free and conjugated) in milk (4.8% TRR, 0.04 mg eq/kg), liver (1.5% TRR, 0.09 mg eq/kg), kidney (2.4% TRR, 0.04 mg eq/kg) and muscle (0.9% TRR, < 0.01 mg eq/kg).

In laying hens, the metabolic fate of triflumuron was investigated using chlorophenyl-label triflumuron. Three hens received daily oral doses of 8.0 mg/kg bw (equivalent to 100 ppm in the feed (DM)) for five consecutive days. Animals were sacrificed 3 hours after the last treatment.

The TRR in edible tissues was the highest in fat (27 mg eq/kg), followed by skin (14 mg eq/kg), liver (7.3 mg eq/kg), kidney (3.1 mg eq/kg) and muscle (0.80 mg eq/kg). In eggs, the TRR consistently increased from 24 to 96 hours after application (0.61–0.98 mg eq/kg) and no plateau was reached. Only eggs collected 96 hours after the last application were used for extraction.

Extractability with the solvents used was >78% TRR; methanol for liver and kidney, acetone and dichloromethane for muscle and eggs, and hexane and acetonitrile for fat and skin.

Parent triflumuron was a major component of the residue and was found at 86% TRR (0.57 mg eq/kg) in eggs, 91% TRR (0.73 mg eq/kg) in muscle, 97% TRR (26 mg eq/kg) in fat, 97% TRR (13 mg eq/kg) in skin, 59% TRR (1.8 mg eq/kg) in kidney and 86% TRR (6.2 mg eq/kg) in liver. The following metabolites, free and conjugated, were identified: M01 in eggs (7.6% TRR, 0.05 mg eq/kg), muscle (6.3% TRR, 0.05 mg eq/kg), fat (0.3% TRR, 0.08 mg eq/kg), skin (0.5% TRR, 0.07 mg eq/kg), kidney (4.4% TRR, 0.14 mg eq/kg) and liver (8.9% TRR, 0.65 mg eq/kg); and M02 (free and conjugated) in muscle (0.3% TRR, < 0.01 mg eq/kg), fat (< 0.1% TRR, < 0.03 mg eq/kg), skin (0.1% TRR, 0.1 mg eq/kg), kidney (26% TRR, 0.81 mg eq/kg) and liver (0.3% TRR, 0.02 mg eq/kg) (absent in eggs).

Five other hens received a single oral dose of the chlorophenyl-label at 2.5 mg/kg bw (equivalent to 33 ppm in the feed (DM)). Eggs were collected within 96 hours after dosing and the TRR in eggs (96 hours after application) was 0.075 mg eq/kg with 92% TRR extracted with acetone and then dichloromethane. Parent triflumuron was found at 85% TRR (0.064 mg eq/kg) and the only identified metabolite was M01 (3.3% TRR, 0.002 mg eq/kg).

Summary

Parent triflumuron is the major component of the residue. Major metabolites were M03 and M04 in kidney of goats and M02 in kidney of hens. The Meeting concluded that the metabolic profiles were qualitatively similar between goats and hens.

Methods of analysis

The Meeting received methods of analysis for supervised field trials and animal feeding studies.

Method 00722/M002 was for analysis of triflumuron, M07 and M08 in sunflower seeds, soya beans and its processed commodities and aspirated grain fractions. In general, triflumuron, M07 and M08 were extracted with acetonitrile or acetonitrile/hexane (3:2, v/v) and filtered. Determination was by LC-MS/MS using external matrix-matched standards. The method was validated for triflumuron in sunflower seeds, soya beans and processed commodities of soya beans (soya bean oil, cold pressed; soya bean oil, solvent extracted; soya bean flour; soya bean hulls; soya bean meal; and soya bean milk) (LOQ=0.01 mg/kg), and aspirated grain fractions (LOQ=0.1 mg/kg) with mean recovery ranges of 83–108%. The method was also validated for M07 and M08 in soya beans and soya bean products with a LOQ of 0.005 mg/kg (equivalent to 0.01 mg/kg triflumuron) with mean recovery ranges of 82–105%.

Method 73295 was for analysis of triflumuron residues in animal commodities. Residues of triflumuron were extracted with acetone (milk) or dichloromethane/methanol (9:1, v/v; muscle, liver, kidney or fat), filtered, and cleaned up. Determination was by HPLC-UV (240 nm). The LOQs for triflumuron in milk, certain tissues (muscle, liver and kidney) and fat were 0.01 mg/kg, 0.05 mg/kg and 0.1 mg/kg, respectively. The mean recoveries (76–96%) were within the acceptable range. The Meeting confirmed that the method is suitable for analysis of triflumuron in milk, muscle, liver, kidney and fat.

Method 00757 was for analysis of triflumuron residues in animal commodities. Residues of triflumuron are extracted with acetonitrile/n-hexane (3:2 v/v) and filtered. Determination is by LC-MS/MS using external matrix-matched standards. The method was validated for determination of triflumuron in animal commodities with LOQs of 0.005 mg/kg in fat, kidney, liver, meat and milk and the mean recoveries (84–101%) were within the acceptable range. The Meeting confirmed that the method is suitable for milk, muscle, liver, kidney and fat.

Stability of residues in stored analytical samples

A stability study on triflumuron residues in fortified sunflower seed (high oil content crop) was available. The Meeting concluded that triflumuron in high oil content commodities stored at ≤ -18 °C was stable for at least 23 months.

Based on a stability study on M07 stored at ≤-20 °C in fortified soya bean seed, the Meeting concluded that M07 in soya bean seed was stable for up to 3.3 months.

M08 stored at ≤-20 °C in fortified soya bean seed was stable for at least 12 months.

In animal commodities, a stability study on triflumuron residues in fortified liver, muscle and milk stored at ≤ -18 °C was available. No significant degradation was observed for at least 3.4 months in liver and muscle and up to 3.0 months for milk. The Meeting concluded that triflumuron residues in animal commodities stored at ≤ -18 °C are stable for at least 3.0 months.

Definition of the residue

Plant commodities

In the plant metabolism studies on apple, tomato, soya bean and potato, the predominant residue in solvent extracts was parent triflumuron (>98% TRR in apple and tomato fruits; 16% TRR in soya bean seeds and 42–49% TRR in potatoes). Triflumuron was found in all primary crop commodities tested. The Meeting noted that suitable analytical methods exist to measure triflumuron in plant commodities. The Meeting considered that parent triflumuron was suitable marker for enforcement.

In deciding which compounds should be included in the residue definition for dietary risk assessment, the Meeting noted that no metabolites exceeded 10% TRR or 0.01 mg eq/kg in the metabolism studies on apples and tomatoes. In the metabolism study on soya bean, M02 and M07 exceeded 10% TRR and 0.01 mg eq/kg after acid hydrolysis of the unextracted residue. In the plant metabolism study on potatoes, M02 exceeded 10% TRR and 0.01 mg/kg after acid hydrolysis of the unextracted residue.

For M02, similar toxicity to parent triflumuron was assumed and the ADI for triflumuron (0–0.008 mg/kg bw) should apply to M02. The Meeting noted that the level of M02 in the soya bean metabolism study (DAT 77, free: 0.001 mg eq/kg, released by HCl hydrolysis: 0.063 mg eq/kg) was significant (total: 0.064 mg eq/kg), but that M02 (free and conjugated) was not analysed in the supervised trials. Considering the residue of the parent compound was 0.030 mg eq/kg in the same plant metabolism study at the same DAT, it was likely that the residue of M02 would be higher than that of the parent compound and it was necessary to estimate the residue level of M02 for dietary risk assessment. The Meeting concluded that the concentration of M02 in soya bean could be estimated to be 2.1 times (0.064 / 0.030) higher than parent triflumuron.

The Meeting established a separate ADI of 0–0.02 mg/kg bw and ARfD of 0.02 mg/kg bw to be applicable to M07 and M08 (expressed as M07). According to the plant metabolism study (DAT 60), free M07 was not found but the level of conjugated M07 (0.10 mg eq/kg) was higher than the parent compound (0.04 mg eq/kg). In the supervised field trials, free M07 was analysed, but conjugated M07 (released by HCl hydrolysis) was not. Considering the residue of the parent compound in the same plant metabolism study at the same DAT was 0.040 mg eq/kg, it was likely that the residue of M07 would be higher than that of the parent compound. Thus, it was necessary to estimate the residue level of conjugated M07 for dietary risk assessment. The Meeting concluded that the concentration of conjugated M07 in soya bean could be estimated to be 2.5 times (0.10 / 0.040) higher than parent.

In the plant metabolism study, triflumuron was released by HCl from the unextracted residue at a low concentration (0.004 mg eq/kg) where the application rate was 7.2 times higher than that of the maximum seasonal application rate in the Columbian GAP. The Meeting decided not to include conjugated triflumuron in the residue definition.

In the hydrolysis study simulating conditions of pasteurisation, baking/brewing/boiling, and sterilization, M07 and M08 were identified. In the processing study on soya bean (hulls, meal, flour, soya milk, solvent extracted oil and cold pressed oil), M07 and M08 were <LOQ (0.005 mg/kg). The Meeting concluded that further consideration of M07 and M08 in processed commodities was not necessary.

Animal commodities

In the animal metabolism studies on lactating goat and laying hen, the predominant residue was parent triflumuron. Triflumuron is found in all animal commodities tested. The Meeting noted that suitable analytical methods exist to measure triflumuron in animal commodities. The Meeting considered parent triflumuron to be a suitable marker compound for enforcement.

In the animal metabolism study, the triflumuron residues were much higher in fat than in muscle (6–27 times higher in lactating goat and 27–85 times higher in laying hen). While no information was available on the partition of residues in milk or eggs, the Meeting considered triflumuron to be fat-soluble.

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting noted that among the animal commodities tested, the residues that exceeded 10% TRR and 0.01 mg eq/kg were: in lactating goat, M03 and M04 (free and conjugated) in kidneys, and in laying hens, M02 in kidneys.

For M02, significant levels were only found in kidneys of hens and not found in lactating goats. The Meeting decided that it was not necessary to include M02 in the residue definition for animal commodities.

For M03, similar toxicity to parent triflumuron was assumed. M03 was found only in milk and kidney from lactating goat in the metabolism study. The estimated levels of M03 in animal commodities based on the calculated animal dietary burden were very low (0.012 mg/kg in kidney and 0.001 mg/kg in milk). The Meeting decided not to include M03 in the residue definition for dietary risk assessment.

No toxicity data were available for M04. Based on its structure, the Meeting concluded that it was appropriate to apply the TTC approach for a potentially genotoxic compound. The Meeting estimated that long-term dietary exposure to M04 from animal commodities (0.0041 µg/kg bw per day) was higher than the threshold of toxicological concern for potential genotoxic compounds (0.0025 µg/kg bw per day).

TTC consideration of M01

Metabolite M01 was found in soya bean (seed and forage) in the metabolism study, tissues and milk from lactating goats, eggs and kidney from hens in the animal metabolism studies and kale, beet (tops and roots) and wheat (forage, heads and straw) in the confined rotational crop study. As no toxicity information was available for M01, based on its structure, the Meeting concluded that it was appropriate to apply the TTC approach for a potentially genotoxic compound.

The Meeting noted that the estimated long-term dietary exposure to M01 from soya bean, rotational crops (leafy vegetables, root and tuber vegetables and cereal grains) and animal commodities were higher (0.046 µg/kg bw per day) than the threshold of toxicological concern for potential genotoxic compounds (0.0025 µg/kg bw per day).

Conclusion

The Meeting decided that the residue definition for compliance with the MRL for plant and animal commodities was triflumuron. The residue is fat-soluble.

The Meeting was unable to conclude on residue definitions for dietary risk assessment for plant and animal commodities.

Results of supervised residue trials on crops

Soya beans

The critical GAP for triflumuron on soya bean in Colombia is two applications at 0.077 kg ai/ha with a minimum interval between sprays of 15 days and a PHI of 21 days. In trials matching the Colombian GAP, residues of triflumuron in soya beans were (n = 9): < 0.01 (3), 0.011, 0.014 (2), 0.048, 0.051 and 0.055 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg.

As the residue definitions for dietary risk assessment were not established, the Meeting could not estimate an STMR for soya bean or complete a dietary risk assessment. .

Fates of residues during processing

High temperature hydrolysis

Triflumuron was shown to be hydrolytically stable for the simulated conditions of pasteurisation (> 97% of AR, 90 °C, pH 4, 20 min). Under simulated baking/brewing/boiling conditions (100 °C, pH 5, 60 min), 89% of the AR remained as triflumuron and M07 was formed (3.4% AR). Under the simulated sterilization condition (120 °C, pH 6, 20 min), 51% of triflumuron remained and compounds derived from hydrolysis, M07 (17% of AR) and M08 (16% of AR), were formed. Further characterization was not conducted.

Processing

The Meeting received information on the fate of triflumuron residues during the processing of soya beans. The Meeting estimated processing factors for parent triflumuron of 3.4 for soya bean hulls and 0.1 for soya bean meal, flour, soya milk, solvent extracted oil (RBD) and cold pressed oil (RBD).

Residues in animal commodities

Farm animal feeding studies

The Meeting received a dairy cow feeding study. Triflumuron in gelatine capsules was administered orally once daily to two groups of dairy cows (three animals in each group) for 29 days at levels equivalent to 5.9 ppm or 12 ppm in the feed (DM; 0.3 or 0.6 mg/kg bw). The residue levels of triflumuron in milk, liver, kidney, muscle and fat were < 0.01, < 0.05, < 0.05, < 0.05 and < 0.1 mg/kg, respectively, at both dose levels.

Farm animal dietary burden

The OECD diets include soya bean, hulls, meal, soya bean hay and fodder. In the supervised trials for soya bean, only seed was analysed. The levels of triflumuron in soya bean hay and forage were estimated using plant metabolism study.

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the Meeting. The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 and summarized below.

Table 2 Animal dietary burden for triflumuron

	Animal dietary burden of triflumuron, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	max	mean	max	mean	max	mean
Beef cattle	0.009	0.009	0.008	0.008	13❶	13❸	0.012	0.012
Dairy cattle	3.1	3.1	0.009	0.09	6.1❷	6.1❹	0.011	0.011
Poultry – broiler	0.007	0.007	0.013	0.013	0.008	0.008	0.005	0.005
Poultry – layer	0.007	0.007	1.5❺❼	1.5❻❽	0.008	0.008	0.005	0.005

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

❸Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

❻Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

❼Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

❽Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

Animal commodity maximum residue levels

Cattle

The Meeting noted that no residues were detected in milk at 2× the dietary burden for dairy cattle or in tissues at the approximate dietary burden for beef cattle. The Meeting estimated maximum residue levels of 0.01(*) mg/kg for milks, 0.05(*) mg/kg for mammalian offal, 0.1(*)(fat) for meat, mammalian and 0.1(*) mg/kg for mammalian fat.

Table 3 Maximum residue levels of triflumuron in poultry commodities

	Feed Level (ppm) for eggs residues	Triflumuron (mg/kg) in eggs	Feed Level (ppm) for tissue residues	Triflumuron (mg /kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (broiler or laying hen)							
Feeding Study	100	0.57	100	0.73	6.2	1.8	26

	Feed Level (ppm) for eggs residues	Triflumuron (mg/kg) in eggs	Feed Level (ppm) for tissue residues	Triflumuron (mg /kg)			
				Muscle	Liver	Kidney	Fat
Dietary burden and estimate of highest residue	1.5	0.0085	1.5	0.011	0.093	0.027	0.39

The Meeting noted that no feeding study for laying hen was available. The Meeting considered the metabolism study where hens were administered triflumuron for 5 days at rates 67× the estimated dietary burdens. In the absence of a poultry feeding study no maximum residue levels were estimated for poultry.

RECOMMENDATIONS

Definition of the residue for compliance with the MRL for animal and plant commodities: triflumuron

Definition of the residue for dietary risk assessment for animal and plant commodities: a conclusion could not be reached.

DIETARY RISK ASSESSMENT

No maximum residue levels are recommended, nor are levels estimated for use in long-term and acute dietary exposure assessments as the Meeting could not reach a conclusion on the residue definitions for dietary risk assessment.

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