

MESOTRIONE (277)

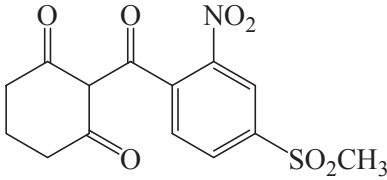
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EXPLANATION

Mesotrione is a systemic pre-emergence and post-emergence herbicide for the selective contact and residual control of broadleaf weeds. It is rapidly absorbed by green plant tissue or taken up through the soil during emergence. It acts by inhibiting the enzyme 4-hydroxyphenolpyruvate dioxygenase (HPPD), leading to a reduction of carotenoids causing bleaching symptoms in the plant. Mesotrione was scheduled for the evaluation as a new compound by the 2014 JMPR at the 45th Session of the CCPR (2013). Metabolism studies on animal and plants, environmental fate studies, analytical methods and residue trials on berries, okra, sweet corn, soya bean and tolerant soya bean, asparagus, rhubarb maize, millet, oat, rice, sorghum, sugarcane and linseed were submitted for evaluation.

IDENTITY

ISO Common Name	Mesotrione
Chemical name	IUPAC: 2-(4-mesyl-2-nitrobenzoyl)cyclohexane-1,3-dione CAS (104206-8): 2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione
Structural formula	
Molecular formula:	C ₁₄ H ₁₃ NO ₇ S
Molecular mass:	339.3

PHYSICAL AND CHEMICAL PROPERTIES

Mesotrione pure material (> 99%) was used for colour, physical state, odour, melting point, pH and spectroscopic characterisation, and also for density, aqueous solubility, dissociation constant, octanol/water partition coefficient and vapour pressure determinations. Colour, odour, solubility in organic solvents, density, pH and surface tension determinations for mesotrione technical material were conducted with material containing 96.7% mesotrione.

Properties of pure mesotrione

Property	Results	Reference; Report
Melting point	165.3 °C with decomposition	Goodman, 1996; RR95-077B
Density at 20 °C	1.49 g/cm ³	
Vapour pressure at 20 °C	< 5.7 × 10 ⁻⁶ Pa	
Volatility at 20 °C	Henry's law constant: < 5.1 × 10 ⁻⁷ Pa/m ³ /mol.	
Physical state, colour, odour	Odourless pale yellow solid (room temperature)	
Solubility in water at 20 °C	0.16 g/L (unbuffered water) 2.2 g/L at pH 4.8 (buffered water) 15 g/L at pH 6.9 (buffered water) 22 g/L at pH 9 (buffered water)	
Partition coefficient n-octanol/ water at 20 °C	log P _{OW} : 0.11 in unbuffered water 0.90 at pH 5 < -1.0 at pH 7 and 9	
pH at 20 °C	3.38 at 25.4 °C in 1% aqueous solution	

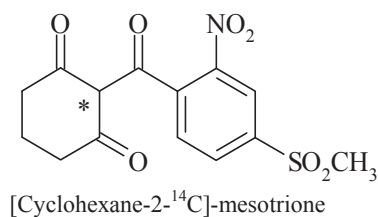
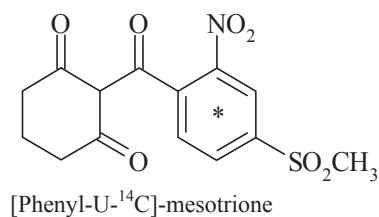
Property	Results	Reference; Report
Hydrolysis in water at 25 °C	Less than 10% degradation of mesotrione (1 µg/ml) occurred during the test period of 30 days in the pH of 4–9 at both 25 and 50 °C.	Miles & Powell, 1995; RJ1776B
Photochemical stability in water	The photolysis half-life and rate constant in sterile aqueous buffer solutions at pH 7 at 25 °C were 83.7 days and 8.40×10^{-3} /day, respectively at 37°56' latitude local sunlight, or 92 days at 50°N. No degradates exceeding 10% of the applied radioactivity were observed during the photolysis of [14 C-phenyl] mesotrione. The major degradation product was carbon dioxide (14 CO ₂), the others were present at levels < 5% of the applied radioactivity. Recovery ranged from 92.2 to 101.8%. Recovery of 92.2% was only observed prior to trapping volatiles formed during photolysis of [14 C-cyclohexane] mesotrione. The average recovery of applied radioactivity after trapping volatiles was 95.4%.	Eya, 1995; RR94-071B
Dissociation constant at 20 °C	pK _a =3.12	Goodman, 1996; RR95-077B

Technical grade material (Goodman, 1996; RR96-004B)

Property	Results
Physical state, colour, odour	Light tan or sand coloured opaque solid with slight odour. sweet (not pungent)
Solubility in organic solvents at 20 °C	Acetone 76.4 g/L Acetonitrile 96.1 g/L 1,2-Dichloroethane 82.7 g/L Ethyl acetate 16.6 g/L Heptane < 0.3 g/L Methanol 3.6 g/L Toluene 2.7 g/L Xylenes 1.4 g/L
Density	The density at 20 °C: 1.46 g/ml; bulk density at 23.3 °C: 0.56 g/mL
pH	The pH of a 1% solution: 3.42 at 24.8 °C
Surface tension at 20 °C	Aqueous solution, 90% saturated: 72.5 mN/m (without indication of surface activity)

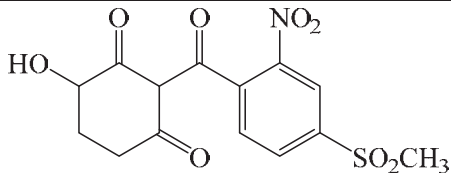
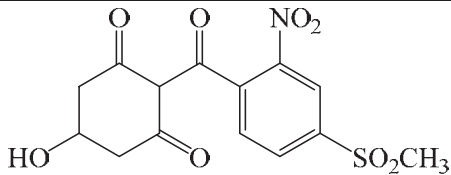
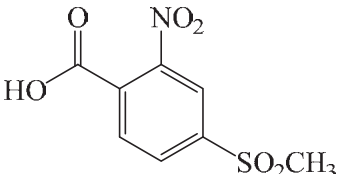
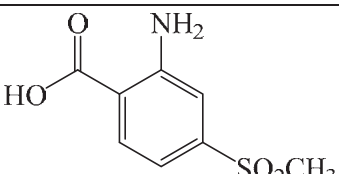
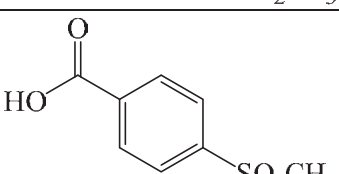
METABOLISM AND ENVIRONMENTAL FATE

The fate and behaviour of mesotrione in animals, plants, and soils were investigated using the following [14 C] labelled test materials:



Concentrations of radioactivity are expressed as mg mesotrione equivalents/kg throughout. The chemical structures of the major degradation compounds arising from the metabolism of mesotrione are presented in the following table.

Degradation compounds from metabolism of mesotrione in plants and animals

Compound Name	Structure	Found in:
4-Hydroxy-mesotrione 4-hydroxy-2-[4-(methylsulphonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione		Livestock, plant
5-Hydroxy-mesotrione 5-hydroxy-2-[4-(methylsulphonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione		Livestock, plant
MNBA 4-(methylsulphonyl)-2-nitrobenzoic acid		Livestock, plant
AMBA (2-amino-4-(methylsulphonyl)-benzoic acid)		Livestock, plant
MBA 4-(methylsulphonyl) benzoic acid		Livestock, plant

Animal metabolism

Cow

Two metabolism studies were conducted in lactating cows, one with [phenyl- ^{14}C]-mesotrione (Powell & Marples, 1996; RJ 1890B) and one with [cyclohexane-2- ^{14}C]-mesotrione (Hand, 1995; RJ 1830B). The studies used similar dosing regimens, sampling protocol and analysis. In each study, one lactating cow was dosed orally with [^{14}C]mesotrione for 7 consecutive days at a nominal rate of 10 ppm in the diet (450–650 kg cow, 20 kg feed intake per day). Milk and excreta were collected daily; the cows sacrificed 16 hours after the final dose and tissues taken for analysis. Milk production was around 20 L per day.

Tissue samples were homogenised in sequence in the presence of solvents such as hexane, acetonitrile/hexane, dichloromethane, acetonitrile, acetonitrile/water (1:9) and 2% sodium dodecyl sulphate (SDS). Each of the extracts was radioassayed. Extracts were partitioned in diethyl ether and water, ethyl acetate and water, hexane and water or hexane and acetonitrile. Solubilised protein was denatured with acetone. Hydrolyses of proteins and extracts were conducted to characterise the bound and/or conjugated residues, using a variety of enzymes, including papain (to hydrolyse ester and peptide bonds) and β -glucuronidase (to hydrolyse glucuronides). Subsamples of the milk extract were submitted to chemical hydrolysis using either 1 M HCl or 1 M NaOH. Protein hydrolysate samples

were derivatized with isobutyl chloroformate to facilitate the chromatography. Residues in tissues were determined by combustion analysis. Normal or reserve phase TLC and HPLC radiochromatograms were used to characterise the components.

Table 1 shows the total radioactive residues found in tissues, milk and excreta (Hand, 1995, Powell & Marples, 1996). Over 90% of the administered dose was found in excreta, mostly in faeces. Liver and kidney contained the highest residues (about 0.1 mg/kg eq.). Residues in muscle were below 0.01 mg/kg eq and reached 0.08 mg/kg eq in milk, with a plateau at day 5 in the cyclohexane experiment (Table 2).

Table 1 Radioactive residues in lactating cow tissues, milk and excreta after oral administration of [¹⁴C]mesotrione

Matrix	[phenyl-U- ¹⁴ C]-mesotrione		[Cyclohexane-2- ¹⁴ C]-mesotrione	
	mg/kg eq.	% Total administered	mg/kg eq.	% Total administered
Liver	0.077	—	0.110	—
Kidney	0.067	—	0.110	—
Muscle (fore)	0.002	—	0.007	—
Muscle (hind)	0.002	—	0.007	—
Peritoneal Fat	≤ 0.004	—	0.008	—
Perirenal Fat	0.007	—	0.005	—
Subcutaneous Fat	≤ 0.004	—	0.013	—
Milk	0.01 to 0.04 (day 5)	0.29	0.06 to 0.08 (day 5–8)	—
Urine		9.65d		13.1
Faeces		80.9		79.4
Total in excreta		90.6		92.5

Table 2 Mean daily radioactive residues in milk (mg/kg mesotrione equivalents)

Study	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8 ^a
[phenyl-U- ¹⁴ C]	0.006	0.052	0.065	0.067	0.074	0.078	0.079	0.072
[Cyclohexane-2- ¹⁴ C]-	0.01	0.03	0.03	0.03	0.04	0.03	0.03	0.03

^a AM collection only

Table 3 (Powell & Marples, 1996) and Table 4 (Hand, 1995) show the results of the characterization of radioactive residues in liver and kidney. Mesotrione accounted for up to 12.3%TRR in liver and 18%TRR in kidney (< 0.02 mg/kg eq.), while AMBA was only identified in kidney.

Table 3 Summary of the identification and characterisation of radioactive residues of mesotrione in liver and kidney of cows treated with [phenyl-U-¹⁴C]-mesotrione

Commodity		Liver		Kidney	
TRR mg/kg		0.085		0.065	
Origin of component	Component	%TRR	mg/kg	%TRR	mg/kg
Solvent extracts ^a	Mesotrione	10.3	0.009	18.0	0.012
	AMBA	ND	ND	15.0	0.010
	Unknowns	24.1 ^b	0.020	35.8 ^c	0.023
	Remainder ^d	6.9	0.006	5.7	0.004
Protein associated fractions	Acetone precipitate	8.7	0.007	1.7	0.001
	Acetone precipitate/Papain hydrolysis debris	9.9	0.008	2.9	0.002
Uncharacterised organosoluble fractions	Acetone ^e	3.0	0.002	4.5	0.003
	Methanol ^f	8.3	0.007	4.0	0.003
	Acidified methanol ^f	13.2	0.011	—	—

Commodity		Liver		Kidney	
Uncharacterised aqueous soluble fractions	H ₂ O extract	3.7	0.003	2.5	0.002
Unextracted		1.7	0.001	4.1	0.003
Losses/gains		10.2	0.009	5.8	0.004
Total		100	0.083 ^g	100	0.066 ^g

^a Values are based on the mean values obtained from three different TLC systems

^b Containing at least eight individual components

^c Components, none of which were > 7.9% TRR, 0.007 mg/kg

^d Containing at least seven individual components, none of which were > 6.4% TRR, 0.004 mg/kg

^e Area corresponding to quantifiable radioactivity containing no discrete bands

^f Originates from the de-proteination of a subsample of the SDS tissue fraction

^g Originates from the C₁₈ fractionation of the solubilised material from the papain hydrolysis

Table 4 Summary of the identification and characterisation of radioactive residues of mesotrione in liver and kidney of cows treated with [cyclohexane-2-¹⁴C]-mesotrione

Matrix	Liver		Kidney	
TRR ^a	0.101 mg/kg		0.107 mg/kg	
Component/Fraction	%TRR ^a	mg/kg	%TRR ^a	mg/kg
Mesotrione	12.5	0.013	14.4	0.015
Neutral organosoluble unknowns ^b	4.2	0.004	6.0	0.006
Aqueous unknowns ^c	1.8	0.002	—	—
Acidic organosoluble unknowns ^d	—	—	2.5	0.003
Acidic unknowns from debris ^e	—	—	0.7	0.01
Remainder ^f	21.3	0.022	13.6	0.015
Uncharacterised soluble fractions. organosoluble ^g	3.4	0.003	8.2	0.009
Aqueous soluble ^h	12.1	0.012	11.9	0.013
Aqueous soluble (eluted from anion ex. resin) ⁱ	—	—	19.0	0.020
Acid soluble ^j	10.0	0.010	—	—
Base soluble	7.5	0.008	—	—
Proteinaceous solids ^k	29.3	0.030	7.1	0.008
Unextracted	5.4	0.005	—	—
Ion exchange resin-bound	—	—	10.1	0.011
Losses During Fractionation	4.9	0.005	6.5	0.005
Total	100.0	0.101	100	0.106 ^l

^a TRRs was calculated from the summation of fractions obtained from various complex extraction schemes required to determine the nature of this type of residue

^b Consists of three components, the largest represents 2% (0.002 mg/kg) liver and 2.6% (0.003 mg/kg) kidney

^c Consists of two components, largest represents 0.9% (< 0.001 mg/kg)

^d Consists of two components, largest represents 1.4% (0.001 mg/kg)

^e Consists of five components resulting from 6 N HCl and subsequent derivatization (largest 0.2%, < 0.001 mg/kg)

^f Derives from chromatography of multiple extracts. Includes baseline material and non-discrete areas of ¹⁴C

^g Acetonitrile wash of debris (liver), SDS extraction of debris (kidney), which consists of two extracts (max 6.2% TRR, 0.007 mg/kg)

^h Consists of two extracts, largest represents 7.4% (0.007 mg/kg) in liver and 7.1% (0.008 mg/kg) in kidney

ⁱ Consists of three extracts, largest representing 11.0% TRR (0.012 mg/kg) of the which could not be chromatographed due to the high concentration of acid present. These extracts were considered to chemical distinct in terms of their ionic nature

^j Consists of two extracts, largest represents 6.3% TRR (0.006 mg/kg) liver

^k Consists of two precipitates, largest represents 22.7% (0.023 mg/kg) liver |TRR. This was hydrolysed with 6 N HCl and derivatized with isobutyl chloroformate. The resulting acidic organosoluble fraction consisted of five components, the largest representing 4.0% (0.004 mg/kg)

^l The slight discrepancy is due to rounding of the calculated values.

Tables 5 shows the results of the characterization study of residues in milk collected from the phenyl labelled experiment (Powell & Marples, 1996). Most of the radioactivity was found in the skimmed milk (97.3% TRR), which was deproteinated using acetone. The resultant aqueous acetone fraction (78.2% TRR) showed to contain at least three polar components, not hydrolysed with enzymes but degraded to up to seven components under harsh acid and basic conditions.

Table 5 Summary of the identification and characterisation of radioactive residues of mesotrione in milk [phenyl-U-¹⁴C]-mesotrione

Total ^a			0.036 mg/kg	
Component/Fraction			%TRR	mg/kg
Whole milk	Centrifugation	Butterfat	0.6	0.000
		Skim milk ^b	1.8	0.001
Skimmed milk (following centrifugation)	Partition	Dichloromethane soluble ^c	3.6	0.001
	SDS precipitation	Protein	4.6	0.002
		Aqueous acetone fraction ^d	3.9	0.001
	Chromatography of aqueous fraction (following SDS precipitation)	Unknown 1	50.0	0.018
		Unknown 2	2.2	< 0.001
		Unknown 3	21.9	0.008
Remainder ^e		4.1	0.001	
Losses During Fractionation			7.3	0.005
Total			100.0	0.036

Based on the extraction of the Day 6 (initial) milk sample. Further characterisation of additional samples was carried out following derivatization or acid/base hydrolysis but as this did not lead to the identification of discrete components the data is not reported in the table above

^a Total concentration taken from sum of extractions and combustion analyses

^b Consists of one sample, skim milk removed from the butterfat samples

^c Consists of one sample from the partition of skimmed milk with dichloromethane

^d Consists of two samples obtained from the aqueous wash of the protein fraction

^e Area corresponding to quantifiable radioactivity containing no discrete bands

TRR in milk from the cyclohexanedione experiment (Hand, 1995) was 0.07 mg/kg eq., 90% of which was associated with the skimmed milk fraction. Lactose accounted for 35.1% TRR in whole milk (Table 6), with the remainder of the extracted radioactivity comprised several unknown components, the largest of which was aqueous soluble and represented 14.7% TRR (0.010 mg/kg). Low levels of radioactivity were found to be associated with the proteinaceous solids and the butterfat.

The radioactive residues in subcutaneous fat were 0.012 mg/kg, with 37% extracted with acetonitrile, leaving 63.3% associated with the debris, from which 46.7% was not extracted (Hand, 1995).

Table 6 Summary of the identification and characterisation of radioactive residues of mesotrione in milk [cyclohexane-2-¹⁴C]-mesotrione

Total ^a	0.070 mg/kg	
Component/Fraction	%TRR ^a	mg/kg
Lactose	35.1	0.025
Aqueous Fractions ^b	24.4	0.017
Organosoluble Fractions ^c	10.5	0.007
Remainder ^d	10.5	0.007
Proteinaceous material	7.6	0.005
Butterfat	10.2	0.007
Losses During Fractionation	1.7	0.001
Total	100.0	0.069 ^e

^a Total concentration taken from sum of skimmed milk and butterfat

^b Consists of five unknowns, largest of which represents 14.7% TRR (0.01 mg/kg)

^c Consists of nine unknowns, largest of which represents 5.3% TRR (0.004 mg/kg)

^d Remainder derives from the chromatography of four extracts. No discrete peaks

^e Slight difference due to rounding.

Swine

One female Hampshire swine (30 kg) was dosed orally with [phenyl-U-¹⁴C]-mesotrione for 5 consecutive days at 6 ppm (Brown, 2006; T020371-04). Excreta were collected daily, the animal sacrificed 23 hours after the final dose, and tissues taken for analysis. Tissue samples were extracted twice with acetonitrile/water (80:20), and further extraction with water was performed on the solids

obtained from the kidney and liver sub-samples. Liquid-liquid partitions were carried out with ethyl acetate after adjustment to pH 2 with acetic acid. C₁₈ SPE columns were used to purify the isolated metabolites. Protein was precipitated with either ammonium sulphate or acetone. To extract ionic material and membrane-bound proteins from cell walls, liver PES was extracted with 2% SDS solution, and the solids obtained were submitted to enzymatic hydrolysis. Analytes were separated using 1 and 2-D TLC on silica gel plates. The metabolites were characterised and isolated using HPLC coupled to a UV detector, radioisotope flow monitor and a fraction collector. Additional confirmation and identification was undertaken using tandem mass spectrometry and ¹H-NMR.

The radioactivity detected in the excreta accounted for 89.4% of the administered dose. In tissues, highest residues were found in liver (1.75 mg/kg eq) and kidney (0.12 mg/kg eq); residues in muscle reached 0.01 mg/kg eq. (Table 7). Residues in the tissues were readily extracted with solvents, and showed to be mainly mesotrione (up to 90% TRR in liver; Table 8). Residues AMBA accounted for up to 2% TRR in kidney. MNBA was only found in liver (0.3% TRR). The major identified residue in urine collected on day 5 was AMBA which accounted for approximately 36.5% TRR.

Table 7 Radioactive residues in swine tissues and excreta after oral administration of [phenyl-U-¹⁴C]-mesotrione

Matrix	Radioactive residue phenyl-U- ¹⁴ C mesotrione equivalents		
	% of the applied dose	mg/kg eq. ^a	mg/kg eq. ^b
Liver	4.2	1.748	1.630
Kidney	0.05	0.117	0.118
Muscle	0.26	0.0096	0.011
Fat	0.06	0.006	n/d
Urine	36.5	–	–
Faeces	51.1	–	–
Total in excreta	89.4 (includes cage washings)	–	–
Total recovered	94.2	–	–

^a Based on the initial combustion values

^b Based on dpm from extracted sample. These values were used for calculation in the study. Fat and the remaining tissues were not analysed further

Table 8 Summary of the identification and characterisation of radioactive residues of mesotrione in tissues of swine dosed with [phenyl-U-¹⁴C]-mesotrione

Matrix	Liver		Kidney		Muscle	
TRR (mg/kg eq.):	1.630		0.118		0.011	
Extracted (%)	98.7 ^b		90.2		90.7	
Component/Fraction	%TRR ^a	mg/kg eq. ^a	%TRR ^a	mg/kg eq. ^a	%TRR ^a	mg/kg eq. ^a
Mesotrione	89.9	1.465	72.8	0.086	77.9	0.009
AMBA	1.8 ^d	0.029 ^d	2.0	0.002	0.7	< 0.001
MNBA	0.3	0.005	–	–	–	–
Unknown 1	1.0	0.016	–	–	–	–
Unknown 2	–	–	0.3	< 0.001	–	–
Unknown 4	0.2	0.003	0.3	< 0.001	–	–
Unknown 5	< 0.2 ^c	0.003	0.5	0.001	–	–
Unknown 6	1.2	0.020	0.2	< 0.001	–	–
Unknown 7	0.9	0.015	0.3	< 0.001	–	–
Unknown 8	0.7	0.011	1.1	0.001	–	–
Unknown 9	0.2	0.004	0.9	0.001	–	–
Baseline	9.3	0.152	11.5	0.014	6.9	0.001
PES	2.8 ^c	0.046 ^c	9.7	0.011	9.3	0.001
Total ^e	108	1.760 ^f	99.6	0.118 ^f	94.8	0.010 ^f

^a These calculations include values obtained from HPLC fraction collecting. Components in the HPLC fractograms, as % of the chromatogram, were calculated from the total radioactivity in specific fractions divided by the total radioactivity injected. The total radioactivity recovered from the HPLC fractions ranged from 97–112%, compared to the amount injected. These recoveries were not taken into account in the calculation and thus contributed to the slight losses or gains in the total accountability compared to the initial residues. Small discrepancies between the % and mg/kg values are due to rounding

^b Includes 2% SDS and protease extraction values

^c < Value not included in summation

^d Portions not identified (0.2% TRR AMBA)

^e Solids remaining after protease and 2% SDS extractions of original PES (16.5% TRR, 0.269 mg/kg)

^f Calculated by % TRR total × total residue mg/kg

Poultry

Two metabolism studies were conducted in poultry, one with [phenyl-U-¹⁴C]-mesotrione (Young & Skidmore, 1995; RJ 177778B) and one with [cyclohexane-2-¹⁴C]-mesotrione (Grout, 1996; RJ 12071B). The studies used the same dosing regimen, sampling protocol and analysis. In each study, hens were dosed by gelatine capsule with [¹⁴C]-mesotrione for 10 consecutive days at 11 ppm, assuming a daily dietary intake of 150 g and average body weight of 2 kg. Each experiment used 10 hens (*Gallus gallus domesticus*). Excreta and eggs were collected daily.

Eggs were separated into yolks and whites and stored frozen for analysis. The hens were sacrificed approximately 16 hours after the final dose, and tissues stored frozen until analysis. Once thawed, liver, kidney and peritoneal fat samples were chopped, muscle samples were minced whilst partly frozen using a food processor. The skin and subcutaneous fat samples were ground with solid CO₂.

The amount of radioactivity in excreta, edible tissues and eggs was determined in 2–3 hens and samples from the remaining hens were retained for analysis if required. TRR were determined using LSC/combustion or tissue solubilisation/LSC. Tissue samples and egg yolk were homogenised and sequentially extracted with methylene chloride, acetonitrile and acetonitrile/water. Fat samples from the cyclohexane experiment were first extracted with hexane. Between each extraction, the organic phase was separated from the solids (post extraction solids, PES) by centrifugation and analysed for radioactivity. Liquid phases were concentrated and extracted with either ethyl acetate and water or hexane and acetonitrile. SDS was used to extract ionic material and membrane-bound proteins from cell walls; proteins were precipitated out with acetone. Samples of edible tissues and eggs that contained a radioactive residue greater than 0.01 mg/kg were analysed further for residue characterisation. Components were separated using 2-D normal and reverse phase TLC, HPLC-UV or a radioisotope flow monitor and a fraction collector.

The radioactivity in excreta accounted for 90 to 98.7% of the administered dose in both experiments. TRR in edible tissues and egg samples are summarised in Table 9. Highest residues were found in liver and kidney (0.06–1.2 mg/kg eq.) Residues in muscle were below 0.02 mg/kg eq in eggs, residues were higher in the yolk compared to whites, reaching 0.094 mg/kg eq. in yolk in the cyclohexanedione label experiment.

Table 9 Radioactive residues (mg/kg eq.) in eggs and tissues following oral administration of [¹⁴C]mesotrione to hens for 10 consecutive days at 11 ppm

	[Phenyl-U- ¹⁴ C]-mesotrione	[¹⁴ C-cyclohexanedione]-mesotrione
Matrix	Mean TRR (n=3)	Mean TRR (n=2)
Liver	1.121	1.245
Kidney	0.063	0.068
Thigh muscle	< 0.004	0.011
Breast muscle	0.004	0.012
Skin/subcutaneous fat	0.042	0.048
Peritoneal fat	< 0.003	0.010
Egg white	< 0.004 at all days	0.012 (day 1) to 0.025 (day 4); mean=0.019
Egg yolk	< 0.003 (day 1) to 0.024 (day 10); mean=0.015	0.002 (day 1) to 0.094 (day 9/10), mean=0.056

Summaries of the radioactivity extracted sequentially using a range of solvents in each study are present in Tables 10 and 11. In the phenyl label experiment (Table 10), most of radioactivity was extracted from liver and egg yolk with ACN/water, and about 70% TRR extracted from fat with methylene chloride (Young & Skidmore, 1995). Radioactivity in PES ranged from 0.6 to 4.5% TRR in egg yolk.

Table 10 Radioactivity levels extracted from hen tissues and egg samples after oral administration of [phenyl-U-¹⁴C]-mesotrione using a range of different solvents

Matrix (TRR, mg/kg eq.)	Extraction solvent (% TRR)					PES (%)
	Methylene chloride	ACN:water (9:1)	ACN:water (1:1)	Water	SDS solution	
Liver (1.234)	–	52.8	22.0	10.3	14.3	0.6
Skin/subc. fat (0.037)	69.2	23.1	1.4	2.7	–	3.6
Egg yolk (0.021)	37.2	53.1	5.3	0	–	4.5

PES Post extraction solids

– Not applicable

In the ¹⁴C-cyclohexanedione experiment (Table 11), the majority of the radioactive residue in skin and subcutaneous fat and egg white was extracted with acetonitrile and acetonitrile/water, while peritoneal fat residues were mostly extracted with hexane (Grout, 1996). Half of residues present in muscle were extracted with acetonitrile, with the remaining staying in PES. Residues from egg yolk were mostly extracted with methylene chloride.

Table 11 Radioactivity levels extracted from hen tissues and egg samples after oral administration of [¹⁴C-cyclohexanedione]-mesotrione using a range of different solvents

Matrix	Extract (% TRR)						PES (%)
	Hexane	Methylene chloride	ACN	ACN/water (1:1)	Water	SDS	
Liver	–	–	19.2	23.5	36.6	18.9	1.8
Skin/subc. fat (n=2)	4.4	–	72.6	11.4	0.0	–	11.6
Peritoneal fat	64.0	–	–	–	–	–	36.0
Thigh muscle	–	–	49.9	–	–	–	50.1
Breast muscle (n=2)	–	–	50	–	–	–	50
Egg white	–	–	32.3	38.5	5.5	–	23.7
Egg yolk	8.1	59.1	12.4	–	7.1	11.0	2.4

DCM: dichloromethane

ACN= acetonitrile

PES: Post extraction solids

– Not applicable

Mesotrione was detected in both experiments, accounting for over 85% TRR (1.1 mg/kg) in liver (Table 12). Mesotrione was not detected in muscle. The lipid fraction present in the combined yolk sample from the [¹⁴C-cyclohexanedione] experiment was extracted with acetone which released 51.7% TRR (0.041 mg/kg), and found to contain mesotrione (0.019 mg/kg). The second acetone fraction was saponified, fractionated and showed to contain palmitic/oleic acid (Table 12). Excreta were shown to contain mesotrione (19.7–55%TRR) and AMBA (18% TRR).

Table 12 Mesotrione residues extracted from hen liver, skin, fat and egg yolk after oral administration

	Liver				Subcutaneous Fat and Skin				Egg Yolk			
	[¹⁴ C-phenyl]		[¹⁴ C-cyclo]		[¹⁴ C-phenyl]		[¹⁴ C-cyclo]		[¹⁴ C-phenyl]		[¹⁴ C-cyclo]	
	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR
Mesotrione	1.051	85.2	1.097	90.7	0.032	85.3	0.033	59.0-71.2	0.017	80.9	0.019	19.5
Palmitic/oleic acid											0.015	15.0

Metabolism of AMBA in cow

AMBA is a metabolite found in maize forage and fodder when the plant is treated with mesotrione. A lactating cow was orally administered [phenyl-U-¹⁴C]-AMBA using gelatine capsule for 7 consecutive days at a rate of 12.2 ppm in the diet, equivalent to 200 mg ai/day (Hand, 1997; RJ

2309B). Milk and excreta were collected daily. The cow was sacrificed approximately 23 hours after the final dose and tissues were taken for analysis.

Samples were sequentially homogenised in the presence of a variety of solvents including hexane, ethyl acetate, acetonitrile/water (1:1) and water. Liquid-liquid partitions were carried out between ethyl acetate and water. Metabolites were separated using normal or reserve phase TLC with visualisation of the radiochromatograms undertaken by phosphor image analysis.

The total recovery was found to be 88.7% of the administered dose, with the majority of the radioactivity recovered in the excreta (Table 13). Highest residues were found in kidney (0.053 mg/kg eq.) and fat (0.018 mg/kg eq.), with no detected residues in muscle or in omental fat. Residues in milk reached a maximum of 0.009 mg/kg eq. (Day 6), decreasing to 0.003 mg/kg eq. on Day 8 after the initial dose.

Table 13 Radioactive residues in lactating cow after oral administration of [^{14}C]AMBA

Matrix	Radioactive residue	
	[^{14}C]AMBA equivalents (mg/kg)	% Total administered
Liver	0.005	
Kidney	0.053	
Fat (perirenal)	0.018	
Fat (omental)	0.000	
Fat (subcutaneous)	0.003	
Milk (peak daily concentration)	0.009 (day 6)	0.06
Urine		32.0
Faeces		56.7
Total in excreta		88.7

Residues in the kidney and perirenal fat were readily extracted with solvents. AMBA represented 79.0 and 61.6% TRR, equivalent to 0.038 and 0.013 mg/kg in the kidney and perirenal fat respectively. Unknown components did not account for more than 0.001 mg/kg (Table 14).

Table 14 Nature of the residues of AMBA in kidney and perirenal fat

	Kidney		Perirenal fat	
TRR, mg/kg (% chromatographed)	0.048 (87.9)		0.021 (81.7)	
Component/Fraction	%TRR	mg/kg	%TRR	mg/kg
AMBA	79.0 ^a	0.038	61.6	0.013
Unknown	0.5	0.000	3.2	0.001
Unidentified	8.4	0.004	16.9	0.004
Aqueous fractions ^b	5.9	0.003	21.3	0.004
Organic fractions	2.5	0.001	—	—
Unextracted	3.1	0.001	9.7	0.002
Gains/Losses	0.6	0.000	−12.7	0.003
Total	100.0	0.047	100.0	0.021

^a Summation of the % AMBA in two major fractions

^b Two fractions, the largest of which represents 12.6% TRR (0.003 mg/kg) in the perirenal fat and 3.0% TRR (0.001 mg/kg) in the kidney;

Based on the metabolism studies conducted with [cyclohexane-2- ^{14}C]-mesotrione, [phenyl- ^{14}C]-mesotrione and [^{14}C]AMBA with poultry, cow and swine, the proposed biotransformation of mesotrione in livestock involves the oxidative cleavage of the parent molecule to yield MNBA and the reduction of the nitro group in MNBA giving AMBA (Figure 1).

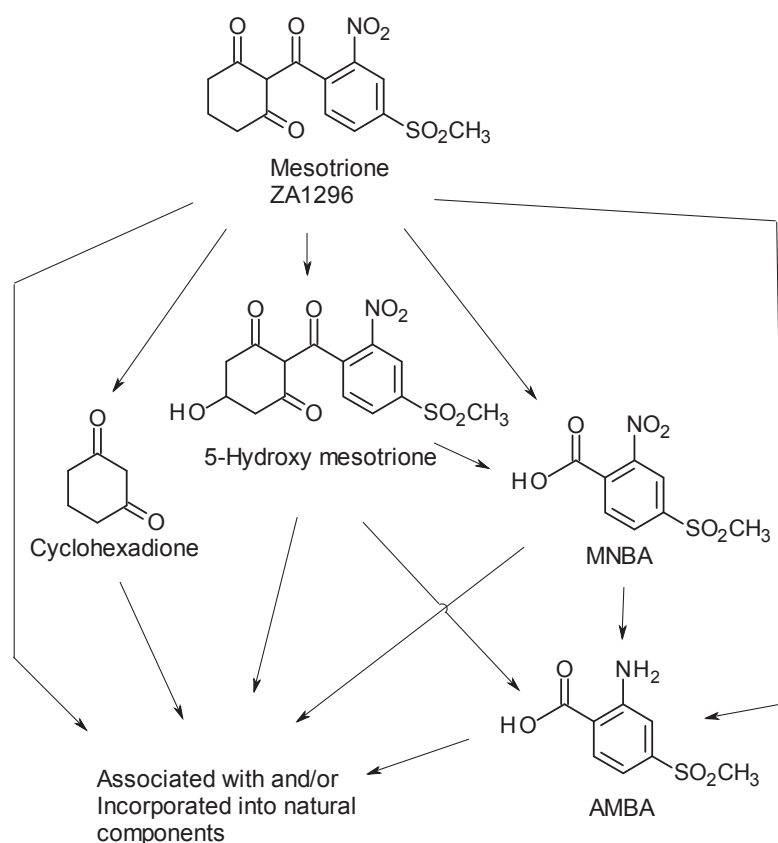


Figure 1 Proposed metabolism of mesotrione in livestock

Plant metabolism

Cranberries

[Phenyl- ^{14}C]-mesotrione was applied (diluted with aqueous formulation) twice to pollinated cranberry plants (*Vaccinium spp.* var. Howe), 4 to 5 years in age, in the greenhouse, under two dose regimes (Ray 2005). The first application at 0.331 or 0.919 kg ai/ha, and the second application, 14 days later, at 0.242 or 0.642 kg ai/ha. Total application at 1 \times rate was 0.573 kg ai/ha and at 3 \times rate 1.561 kg ai/ha. The radioactive residues in samples of mature cranberry foliage and fruit, harvested 46 days after the second foliar application, were quantified and characterized. A subsample of each commodity type was homogenised in dry ice and the radioactive residue determined by combustion/LSC. Mature fruit samples that were found to have a radioactive residue greater than 0.01 mg/kg were subject to additional analysis. Samples were extracted with acetonitrile/water and fractionated. Further treatment comprised of hydrolysis with dilute acid under reflux conditions. Extracted radioactivity was analysed by 2D TLC and reverse-phase HPLC. Additional confirmation and identification was undertaken using tandem mass spectrometry and ^1H -NMR.

About 100% of the total residues were extracted from cranberry fruits from both treatments, with about 2.2 to 2.5% corresponding to PES. TRR in the mature foliage were 16.8 mg/kg and 31.8 mg/kg, for the 1 \times and 3 \times treatments, respectively. The extracted TRRs in the mature cranberry fruit were 2.64 mg/kg eq. and 4.94 mg/kg eq., respectively. Only the fruits samples were used for the characterisation and identification of metabolites (Table 15). The extracted residues identified in cranberry fruits were mostly mesotrione and AMBA, accounting for 60.2% TRR and 34.8% at the 1 \times rate, respectively, with MBNA accounting for 3.0% TRR.

Table 15 Summary of radioactive residues in mature cranberry fruit samples following treatment with [phenyl-U-¹⁴C]-mesotrione

Application Rate:	0.573 kg ai/ha		1.561 kg ai/ha	
Total extracted:	2.637 mg/kg eq.		4.936 mg/kg eq.	
Component	mg/kg eq.	%TRR	mg/kg eq.	%TRR
Mesotrione	1.548	60.2	3.257	67.1
AMBA	0.895	34.8	1.178	24.3
MBNA	0.076	3.0	0.078	1.6
Unknown baseline	0.042	1.6	0.057	1.2
Unextracted (PES)	0.064	2.5	0.107	2.2
Losses/gains	0.075	2.9	0.364	7.5
Total recovered	2.701	105.0	5.043	103.9

Mesotrione tolerant (HT) soya beans

HT soya bean seeds (*Glycine max* var. Jack) were grown under greenhouse conditions in containers filled with a sandy loam soil (Dohn & Chu, 2012). [Phenyl-U-¹⁴C]- and [cyclohexane-2-¹⁴C]-mesotrione were applied separately using three separate treatment regimes. The first application was a single pre-emergence treatment at a nominal rate of 0.225 kg ai/ha, the second was a combined pre-emergence treatment at 0.225 kg ai/ha followed by a post-emergence treatment at 0.125 kg ai/ha and the third was a single post-emergence treatment at a nominal 0.225 kg ai/ha. [¹⁴C]Mesotrione was applied to the plots as a suspension concentrate using a plastic hand held sprayer.

The radioactive residues in the soya bean foliage and hay samples were extracted twice with a mixture of acetonitrile and water (1:1). Soya bean seed samples were sequentially extracted with acetone/hexane (1:4), acetonitrile/water (1:1) and acetonitrile. Characterisation of the PES samples with residues $\geq 10\%$ TRR or ≥ 0.05 mg/kg was attempted using with hydrolytic enzymes, 1 N hydrochloric acid at room temperature and/or 60 °C. One and 2D normal phase TLC, reverse-phase HPLC-UV, radioisotope flow monitor and a fraction collector was used for sample analyses. Additional identification was undertaken using LS-MS.

The TRR (extracted plus PES) for soy commodities from each application regime and labels are summarised in the Table 16. In the phenyl label experiment, PES accounted for 41%TRR in pre-emergence forage to 57.7%TRR in post-emergence seed. In the cyclohexane label experiment, the values ranged from 21.9%TRR in pre-/post-emergence hay to 53.3%TRR in post-emergence seed.

Table 16 Total radioactive residues in soya bean RACs

Soya bean RAC	Application Stage	DAA (days)	TRR (mg/kg) Phenyl label	TRR (mg/kg) Cyclohexane label
Forage	Pre-emergence	28	0.212	0.077
	Pre- and post-emergence	28	0.162	0.055
	Post-emergence	22	0.499	0.260
Hay	Pre-emergence	42	0.142	0.076
	Pre- and post-emergence	42 (1 st app.); 9 (2 nd app.)	2.015	1.632
	Post-emergence	40	0.370	0.082
Seed	Pre-emergence	123	0.063	0.039
	Pre- and post-emergence	123 (1 st app.); 90 (2 nd app.)	0.104	0.093
	Post-emergence	110/118 ^a	0.052	0.015

^a [Phenyl-U-¹⁴C]-mesotrione: 110 days. [Cyclohexane-2-¹⁴C]-mesotrione: 118 days. DAA=days after application

A summary of radioactive residues from [phenyl-U-¹⁴C]-mesotrione treated soya bean forage extract is detailed in Table 17. MNBA was the most abundant residue present in the single pre-emergence and combined pre- and post-emergence forage samples and 4 or 5-hydroxy-mesotrione the most abundant residues in the post-emergence sample (0.073 mg/kg). Mesotrione residues were in the range of 0.021–0.03 mg/kg eq.). Extraction and acid hydrolysis released about 30% TRR found in pre-emergence forage PES, and lignin extraction released about 50% TRR in post-emergence forage

PES. Results from the [cyclohexane-2-¹⁴C]-mesotrione experiment showed mesotrione and 4 or 5-hydroxy-mesotrione as the major residues in forage (8.5 to 19.2% TRR), while MNBA and AMBA were not detected. Most of the radioactivity found in PES was released by enzyme, acid extraction and lignin extraction.

Table 17 Summary of radioactive residues in soya bean forage samples following pre- and post-emergence application of [¹⁴C]mesotrione

Treatment regime:	Pre-emergence ^a		Pre-+ post-emergence		Post-emergence	
Experiment	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-
TRR, mg/kg eq.	0.212	0.077	0.162	0.055	0.499	0.260
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
MNBA	0.052 (24.5)		0.039 (24.1)		0.065 (13.0)	
AMBA	0.003 (1.4)		0.001 (0.6)		0.004 (0.8)	
4/5-Hydroxy-mesotrione	0.017 (8.0)	0.011 (14.3)	0.011 ^b (6.8)	0.007 (12.7)	0.073 (14.6)	0.050 (19.2)
Mesotrione	0.030 (14.2)	0.013 (16.9)	0.021 (13.0)	0.010 (18.2)	0.028 (5.6)	0.022 (8.5)
Polar unknowns ^c	0.005 (2.4)	0.009 (11.7)	0.006 (3.7)	0.005 (9.1)	0.008 (1.6)	0.034 (13.1)
Unassigned (< 10% TRR)	–		–		0.032 (6.4)	
Unassigned (each < 0.01 mg/kg)	0.018 (8.5)	0.010 (13.0)	0.010 (6.2)	0.08 (14.5)	0.044 (8.8)	0.057 (21.9)
Total characterised	0.125 (59.0)	0.043 (55.8)	0.088 (54.3)	0.030 (54.5)	0.254 (50.9)	0.163 (62.7)
PES	0.087 (41.0)	0.034 (44.2)	0.074 (45.7)	0.025 (45.5)	0.245 (49.1)	0.097 (37.3)
Total	0.212 (100)	0.077 (100)	0.162 (100)	0.055 (100)	0.499 (100)	0.260 (100)

^a Forage harvested prior to second application

^b Confirmed by TLC

^c Acetonitrile/water combined extracts

–=Not applicable

The metabolites MNBA and 4/5 hydroxy-mesotrione were the major residues found in hay of soya-treated with [phenyl-U-¹⁴C]-mesotrione in the three application regimes ranging from 9 to 20%TRR (Table 18). Mesotrione accounted for 6.2 to 8.8%TRR (up to 0.178 mg/kg eq.). Treatment of PES with enzymes, 1 N HCl and lignin digestion released 15.5%TRR in plants from the pre-emergence trial and 13.2% TRR in post-emergence. 4/5-hydroxyl-mesotrione was the major identified residue in hay of soya treated with [cyclohexane-2-¹⁴C] mesotrione, accounting for 15 to 25% TRR (0.409 mg/kg eq). Mesotrione accounted for 6.6 to 7.3%TRR, and the metabolites AMBA and MNBA were not detected. PES treatment released a total of 16.8% TRR in the pre-and post-emergence plants.

Table 18 Summary of radioactive residues in soya bean hay samples following pre- and post-emergence application of [phenyl-U-¹⁴C]-mesotrione

Treatment regime:	Pre-emergence		Pre- and post-emergence		Post-emergence	
Experiment	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-
TRR, mg/kg	0.142	0.076	2.015	1.632	0.370	0.082
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Mesotrione	0.009 (6.3)	0.005 (6.6)	0.178 (8.8)	0.134 (8.2)	0.023 (6.2)	0.006 (7.3)
MNBA	0.015 (10.6)	ND	0.410 (20.3)	ND	0.042 (11.4)	ND
AMBA	ND	ND	0.055 (2.7)	ND	ND	ND
4/5-Hydroxy-mesotrione	0.013 (9.2)	0.012 (15.8)	0.331 (16.4)	0.407 (24.9)	0.054 (14.6)	0.016 (19.5)
Polar unknowns	0.006 (4.2)	0.012 (15.8)	0.026 (1.3)	0.243 (14.9)	0.006 (1.6)	0.012 (14.6)
Unassigned (each < 6%TRR)	0.012 (8.5)	–	0.335 (16.6)	0.491 (30.1)	–	–
Non-defined (each < 0.01 mg/kg)	0.013 (9.2)	0.010 (13.2)	ND	ND	0.05 (13.5)	0.015 (18.3)
Total characterised	0.068 (47.9)	0.039 (51.3)	1.34 (66.3)	1.275 (78.1)	0.175 (47.3)	0.049 (59.8)
PES	0.074 (52.1)	0.037 (48.7)	0.680 (33.7)	0.357 (21.9)	0.195 (52.7)	0.033 (40.2)
Total	0.142 (100)	0.073 (100)	2.015 (100)	1.632 (100)	0.370 (100)	0.082 (100)

None of the identified residues in soya bean seeds from the experiment with [phenyl-U-¹⁴C]-mesotrione accounted for more than 10% TRR in any treatment or at levels higher than 0.007 mg/kg eq. (Table 19). Mesotrione accounted for up to 9.5% TRR%, or 0.006 mg/kg eq. AMBA was detected only in the pre- and post-emergence regime (1.9%TRR). Enzyme extracts of PES contained multiple components up to ≤ 0.007 mg/kg. Only mesotrione and 4/5-hydroxy-mesotrione were detected in soya bean seeds from the [cyclohexane-2-¹⁴C] mesotrione experiment. The PES Viscozyme extracts from the combined pre- and post-emergence sample mainly contained unknown polar peak components (0.011 mg/kg, 11.8% TRR).

Table 19 Summary of radioactive residues in soya bean seed samples following use of [phenyl-U-¹⁴C]-mesotrione

Treatment regime:	Pre-emergence		Pre- and post-emergence		Post-emergence	
Experiment	[phenyl- ¹⁴ C]	[Cyclo - ¹⁴ C]	[phenyl- ¹⁴ C]	[Cyclo - ¹⁴ C]	[phenyl- ¹⁴ C]	[Cyclo - ¹⁴ C]
TRR, mg/kg	0.063	0.039	0.104	0.093	0.052	0.015
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Mesotrione	0.006 (9.5)	0.02 (5.1)	0.003 (2.9)	0.003 (3.2)	0.002 (3.8)	
MNBA	0.001 (1.6)	ND	0.005 (4.8)	ND	ND	
AMBA	ND	ND	0.002 (1.9)	ND	ND	
4/5-Hydroxy-mesotrione	0.003 (4.8)	0.01 (2.6)	0.007 (6.7)	0.003 (3.2)	0.004 (7.7)	
Polar unknowns	0.004 (6.3)	0.07 (17.9)	0.010 (9.6)	0.029 (31.2)	0.008 (15.4)	
Unassigned peaks	–		0.010 (9.6)		–	
Non-defined (each < 0.01 mg/kg)	0.008 (12.8)	0.02 (5.1)	0.007 (6.7)	0.007 (7.5)	0.003 (5.8)	
Total characterised	0.022 (34.9)	0.012 (30.8)	0.044 (42.3)	0.042 (45.2)	0.017 (32.7)	
PES	0.035 (55.6)	0.020 (51.3)	0.052 (50.0)	0.039 (41.9)	0.030 (57.7)	0.008 (53.3)
Acetone/Hexane extractions	0.006 (9.5)	0.007 (17.9)	0.008 (7.7)	0.012 (12.9)	0.005 (9.6)	0.002 (13.3)
Totals	0.063 (100)	0.039 (100)	0.104 (100)	0.093 (100)	0.052 (100)	0.015 (110)

Maize

[Phenyl-U-¹⁴C]-mesotrione was applied to the soil surface after planting the seeds of *maize* (*Zea mays*) (pre-emergence) at a rate of 0.28 kg ai/ha and post-emergence by application to surfaces of plants and soil 28 days after planting at a rate of 0.164 kg ai/ha (Tarr & van Neste, 1997; RR 96-007B). The radioactive residues in plant samples of forage (55 days after planting) and of grain and straw from mature crops (125 days after post-emergence treatment) were characterised.

Samples were extracted with water and acetonitrile (1:1) and fractionated by partition with ethyl acetate and/or with Amberlite XAD7 resin. Acid hydrolysis (micro-wave or reflux) followed by base hydrolysis or treatment with enzymes (porcine carboxylic acid esterase, papain, pancreatin, pectolyase and driselase) of the extracted residues and PES was attempted for characterisation and further solubilisation of residues. The PES from post-emergence fodder was also treated with a solution of potassium permanganate to solubilise bound residues by oxidation. Residue characterization was performed using normal and reversed-phase TLC and/or reversed-phase HPLC with radio-detector or fraction collecting, and LSC.

Table 20 summarises the TRRs found in maize matrices after pre- or post-emergence application. Total residues were higher in fodder and forage, and similar in both treatments in seed (0.013–0.014 mg/kg eq.). Using acetonitrile/water (1:1 v/v), most of the residues were extracted from forage and seed, but unextracted residues accounted for over 60% TRR in fodder. No additional analysis was conducted in seed due to the low radioactivity found.

Table 20 Total radioactive residues (TRRs) in maize after pre- and post-emergence application of [phenyl- ^{14}C]-mesotrione

Sample. DAT	TRR by initial combustion	Extracted residues (acetonitrile/water 1:1 v/v)		Unextracted residues		Sum of extracted and unextracted residues	
	mg/kg eq.	% TRR ^a	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
Pre-emergence application							
Forage. 27	0.356	84.6	0.301	26.2	0.093	110.8	0.394
Fodder. 153	0.795	43.9	0.349	61.8	0.491	105.7	0.840
Grain. 153	0.013	69.6	0.009	26.8	0.004	96.7	0.013
Post-emergence application							
Forage. 28	0.244	72.7	0.177	23.9	0.058	96.6	0.235
Fodder. 125	1.066	48.1	0.513	67.4	0.719	115.5	1.232
Grain. 125	0.014	61.5	0.008	23.2	0.003	84.7	0.011

DAT: Days after treatment

^a Based on residue values derived from initial combustion

Table 21 shows the characterization of the residues found in forage and fodder from both treatments. Mesotrione was a minor component of the residue, present at a higher level in pre-emergence forage samples (2.2% TRR, 0.008 mg/kg eq.). MNBA was the major identified compound in forage from the pre-emergence treatment (19.7%TRR) and a minor component in post-emergence and in fodder. AMBA was a major compound mostly presented in conjugated form accounting for 12–28%TRR in forage and fodder. The predominant AMBA conjugates isolated were α - and β -anomers of the 2-acylglucosides. Acid or base hydrolysis and enzyme treatment did not release additional AMBA from fodder extracts.

Table 21 Summary of radioactive residues in maize forage and fodder samples following pre- and post-emergence application of ^{14}C -phenyl labelled mesotrione

Component	Pre-emergence application		Post-emergence application	
	Forage	Fodder	Forage	Fodder
	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
TRR by combustion	0.356 (100)	0.795 (100)	0.244 (100)	1.066 (100)
Mesotrione	0.008 (2.2)	< 0.003 (< 0.4)	0.001 (0.4)	< 0.003 (< 0.3)
4-OH-mesotrione (total/glucose Conjugate)	0.027/0.013 (7.6/3.8)	0.007/< 0.01 (0.9/< 1.2)	0.016/0.009 (6.6/3.6)	0.007/< 0.01 (0.7/< 0.1)
MNBA	0.070 (19.7)	0.008 (1.0)	0.008 (3.4)	0.019 (1.9)
AMBA (total/conjugates)	0.043/0.031 (12.2/8.9)	0.108/0.104 (13.6/13.0)	0.032/0.029 (13.2/11.5)	0.301/0.295 (28.2/27.6)
Minor components extracted with water/acetonitrile	0.164 (46.0)	0.280 (35.2)	0.128 (52.4) (each < 0.03 mg/kg)	0.320 (30.0) (each < 0.03 mg/kg)
Minor components extracted with 0.1 M HCl /microwave heating	0.049 (13.6)	0.229 (28.8)	0.033 (13.7) (each < 0.03 mg/kg)	0.378 (35.4) (each < 0.03 mg/kg)
Minor components extracted with 1 M NaOH/40 °C		0.030 (3.8)		0.044 (4.1)
Not extracted	0.028 (7.9)	0.033 (4.2)	0.017 (7.0)	0.063 (5.9)

In another study conducted in maize, [phenyl- ^{14}C]-mesotrione was applied to the bare sandy loam soil surface one day after planting the seeds (pre-emergence) at a rate of 0.302 kg ai/ha and post-emergence (31 days after planting) by application to surfaces of plants at the true eight leaf stage at a rate of 0.179 kg ai/ha (Vispetto & Smith, 1999; RR 99-006B). Samples of forage (78 days after first pre-emergence treatment) and of stover and grain from mature crops, 121 days after pre-emergence treatment were taken for analysis.

Samples were extracted with a mixture of water and acetonitrile (1:1), extracts fractionated by hydrolysis with HCl or partitioned with ethyl acetate. PES which exceeded 10% TRR or 0.05 mg/kg were hydrolysed by microwave extraction with HCl, and the hydrolysates cleaned up by C_{18} SPE. HPLC radiochromatograms were obtained by fraction collecting and LSC. TLC was conducted using silica plates.

Highest residues were detected in the stover (0.57 mg/kg), with lower residues in the forage (0.27 mg/kg) and grain (0.03 mg/kg) (Table 22). Over 60% of the radioactivity was extracted and contained a large number of components, all at ≤ 0.01 mg/kg and predominantly water soluble. Mesotrione was not detected in any sample. AMBA, MNBA and their conjugates, and 4-hydroxy-mesotrione were the major compounds identified in forage. 4-hydroxy mesotrione was not detected in grain and stover.

Table 22 Total radioactive residues (TRRs) in maize after pre- and post-emergence application of [phenyl- ^{14}C]-mesotrione

		Forage		Stover		Grain	
		% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
Total Residue (by combustion)		100	0.27	100	0.57	100	0.03
Acetonitrile/water		69.6	0.19	70.3	0.40	60.6	0.02
2 M/4 M HCl Hydrolysis of extract	Organosoluble	—	—	9.5	0.05	—	—
	Aqueous soluble	—	—	45.8	0.26	—	—
	Precipitate	—	—	10.7	0.06	—	—
Non-extracted (PES)		29.9	0.08	32.9	0.19	46.9	0.01
2 M/4 M HCl PES Hydrolysis		9.8	0.03	7.0	0.04	—	—
Post-hydrolysed solids (PHS)		22.6	0.06	23.5	0.13	—	—
AMBA/ conjugate		2.4/4.6	0.01/0.01	1.7/2.3	0.01/0.01	—	—
MNBA/ conjugate		3.3/2.2	0.01/< 0.01	2.2/1.0	0.01/0.01	—	—
4-hydroxy-mesotrione		5.4	0.01	—	—	—	—
Unidentified		5.7	0.14	7.7	0.34	6.9	0.02

^a Fraction comprises a number of small components all < 4.6% TRR (≤ 0.02 mg/kg)

In the third study conducted in maize, [cyclohexane-2- ^{14}C]-mesotrione was applied to the soil surface after planting the seeds (pre-emergence) at a rate of 0.307 kg ai/ha and post-emergence 28 days after planting at a rate of 0.161 kg ai/ha (Wei & Dohn, 1997; RR 96-026B). Samples of forage (27 or 28 days after planting) and of grain and straw from mature crops (125 days after post-emergence treatment) were extracted three times with either a mixture of water and acetonitrile (1:1). Acid hydrolysis of the extracted residues and PES from post-emergence fodder samples was attempted for characterization of residues and enzyme digestion was used in pre- and post-emergence forage samples. HPLC radiochromatograms were obtained using a flow through radio-detector or by fraction collecting and LSC. All critical analyses were confirmed by use of at least two TLC and or HPLC methods.

Total radioactive residues in maize were 0.067, 0.001 and 0.015 mg/kg in pre-emergence forage, grain and fodder respectively. Corresponding values for post-emergence treatments were 0.098, 0.011 and 0.330 mg/kg (Table 23). Much of the radioactivity in forage was readily extracted (78–84% TRR). Remaining solids after extraction were not analysed further. About 60% TRR in pre-emergence fodder and post-emergence grain were not extracted with solvents.

Table 23 Total radioactive residues (TRRs) in maize after pre- and post-emergence application of [cyclohexane-2- ^{14}C]-mesotrione

Sample	TRR by initial combustion	Extracted residues		Unextracted residues	
	mg/kg	% TRR ^a	mg/kg eq.	% TRR	mg/kg eq.
Pre-emergence application					
Forage	0.067	78.3	0.053	20.9	0.014
Fodder ^b	0.015 (0.025 in leaves)	38.1.	0.006 (0.010 in leaves)	60.8	0.010 (0.015 in leaves)
Grain	0.001	—	—	—	—
Post-emergence application					
Forage ^b	0.098 (0.190 in leaves)	84.0	0.082 (0.159 in leaves)	19.6	0.018 (0.037 in leaves)
Fodder	0.330 ^b (0.649 in leaves)	67.5 ^c	0.223	9.4	0.031
Grain	0.011	35.1	0.004	60.7	0.007

^a % TRR's are based on residue values derived from initial combustion

^b The extractions were performed on the leaf sample, but the mg/kg values are corrected for the total (leaves + stalks) sample mass

^c Includes material released by acid hydrolysis and lignin and cellulose fractions

The major identified residue in forage was 4-dihydroxy-mesotrione, accounting for up to 10.4%TRR in pre-emergence samples (0.007 mg/kg) (Table 24). Mesotrione was found at low levels (up to 0.002 mg/kg). In fodder, mesotrione was detected only in trace amounts (< 0.0005 mg/kg) and no other compound was identified (Table 24). The solubilised material from PES accounted for 13.6% TRR (0.045 mg/kg) and showed to contain glucose. Treatment of remaining solids gave evidence for the incorporation of ¹⁴C into lignin and cellulose.

Table 24 Summary of radioactive residues in maize forage and fodder following pre- and post-emergence application of [cyclohexane-2-¹⁴C]-mesotrione

Component	Forage		Fodder (leaves and stalks)
	Pre-emergence mg/kg (% TRR)	Post-emergence ^a % of TRR ^b	post-emergence mg/kg eq.
TRR combustion value	0.067 (100)	0.098 (100)	0.330 (100)
Mesotrione	0.002 (3.0)	0.001 (1.0)	< 0.0005
4-Hydroxy-mesotrione	0.007 (10.4)	0.006 (6.1)	
Components extractable with water/acetonitrile (including neutral carbohydrates formed by acid hydrolysis of the extract) ^c			0.123 (37.3) (each < 0.012 mg/kg)
Components solubilised by refluxing 6 M HCl. majority were neutral carbohydrates, including glucose ^c			0.045 (13.6) (each < 0.029)
Intractable solids from neutralisation of 6 M HCl reflux			0.016 (4.8)
Lignin ^c			0.044 (13.3)
Cellulose ^c	0.006 (9.0)	0.002 (2.0)	0.011 (3.4)
Minor components including carbohydrates	0.038 (56.7)	0.067 (each ≤ 0.012 mg/kg)	
Post-extracted solids (PES)	0.014 (20.9)	0.018 (18.4)	0.031 (9.4)
Not analysed ^b	—	0.007 (7.1)	0.024 (7.3)

^a Leaves and the woody stalks, the leaves contained over 90% of the residue

^b The characterisation experiments were performed on the leaf sample, but the values are corrected for the total (leaves + stalks) sample

^c The order of the extractions was water/acetonitrile. 6 M HCl, hot dimethylsulphoxide (for lignin), and Schweizer's reagent (for cellulose)

Rice

Rice plants (*Oryza sativa* spp. *japonica* cv Kirana 397) at the 2–3 leaf stage in a flooded paddy greenhouse system were treated with [phenyl-U-¹⁴C]-mesotrione added directly to the paddy water at either 0.090 kg ai/ha or 0.225 kg ai/ha (Humphries & Evans. 2005; RJ 3738B). The day 14 and 27 days after treatment (DAT) samples were whole tops, the 40 DAT samples were separated into ears and stalks, the 109 DAT samples were separated into grain, husk and straw. The paddy was maintained flooded to a depth of 3–5 cm until 7 days before final harvest, when irrigation was stopped allowing the paddy system to dry. Natural daytime sunlight was supplemented with artificial light with a fixed 13 hour day period. Samples were extracted with acetonitrile/water mixture and water and PES were further extracted with microwave assisted extractions and acid hydrolysis. Liquid partitions were carried out between ethyl acetate/water (1:1). Extracted radioactivity was analysed by normal and reversed-phase TLC and reverse-phase HPLC.

Extracted radioactivity in the whole tops up to 27 DAT accounted for at least 60%TRR (Table 25). Total residues in grain were 0.01 and 0.02 mg/kg eq. at 0.09 and 0.225 kg ai/ha, respectively, with extracted residues accounting for up to 0.003 mg/kg eq., which were not further characterized. Residues in straw were higher (0.032 and 0.066 mg/kg eq.), from which 43–45%TRR was extracted.

Table 25 Total radioactive residues (TRRs) in rice after treatment of paddy water with [phenyl-U-¹⁴C]-mesotrione

Treatment. kg ai/ha	DAA		Acetonitrile/water		Non-extracted residues		Total Residue mg/kg eq.
		Commodity	% TRR ^a	mg/kg	% TRR	mg/kg	
0.090	14	Whole tops ^{b)}	70.7	0.0461	29.3	0.0191	0.0652
	27	Whole tops	59.9	0.0198	40.1	0.0133	0.0331
	40	Ears	n/a	n/a	n/a	n/a	0.0057
		Stalks	48.1	0.0092	51.9	0.0099	0.0191
	109	Grain	12.4	0.0011	87.7	0.0087	0.0099
		Husk	22.7	0.0022	77.3	0.0077	0.0099
		Straw	43.1	0.0139	56.9	0.0183	0.0321
0.225	14	Whole tops	76.2	0.1934	23.8	0.0604	0.2538
	27	Whole tops	60.7	0.0416	39.3	0.0270	0.0686
	40	Ears	27.0	0.0032	73.0	0.0085	0.0117
		Stalks	46.4	0.0175	53.6	0.0202	0.0377
	109	Grain	17.2	0.0033	82.8	0.0158	0.0191
		Husk	33.1	0.0108	66.8	0.0217	0.0325
		Straw	45.3	0.0298	54.7	0.0360	0.0659

^a %TRR's are based on residue values derived initial combustion

^b Immature plants harvested by cutting the plant 2 cm above the paddy rice water

Tables 26 shows the residues identified in immature whole tops (14 and 27 DAT) and stalks (40 DAT). All commodities analysed contained parent mesotrione, 5-hydroxy-mesotrione, AMBA and MNBA. At early stage, mesotrione comprised the majority of the residues, accounting for 28% TRR for the 0.225 g ai/ha treatment.

Table 26 Summary of radioactive residues in immature rice samples following treatment of paddy water with phenyl-U-¹⁴C labelled mesotrione

Component	0.090 kg ai/ha						0.225 kg ai/ha	
	14 Whole Tops ^a		27 Whole Tops ^a		40 Stalks		14 Whole Tops	
	% TRR	mg/kg	% TRR	mg/kg eq.	% TRR	mg/kg	% TRR	mg/kg
Mesotrione	15.0	0.0098	5.9	0.0020	5.0	0.0010	27.9	0.0708
5-HO-mesotrione ^b	11.4	0.0074	7.5	0.0025	11.1	0.0021	14.1	0.0359
MNBA	4.6	0.0030	4.8	0.0016				
AMBA	2.0	0.0013	3.0	0.0010	1.7	0.0003	2.2	0.0055
Total	33.0	0.0215	21.2	0.0071	17.8	0.0034	44.2	0.1122
Unknowns	4.9	0.0032						
Baseline	16.2	0.0105	30.1	0.0100	16.5	0.0032	16.0	0.0406
Remainder	18.5	0.0120	6.8	0.0021	15.4	0.0030	15.2	0.0386
Unextracted ^c	29.3	0.0191	40.1	0.0133	51.9	0.0099	23.8	0.0604
Loss/gain on fractionation ^c	-1.8	-0.0012	1.8	0.0006	-1.7	-0.0003	0.8	0.0020
Total	100.1	0.0651	100.0	0.0331	99.9	0.0192	100.0	0.2538

Unknowns: unidentified discrete components; Baseline: Polar residue components, which are retained on the baseline after elution

Remainder: diffuse unassigned radioactivity comprising minor indiscrete components and areas of streaking of radioactivity

^a Immature plants harvested by cutting the plant 2 cm above the paddy rice water

^b 5-Hydroxy-mesotrione was assigned based on TLC data from later samples where reference standards were available

^c Not chromatographed

Residues in straw extracts showed the similar pattern as seen in whole tops and stalk samples (Table 27). Baseline polar extract was acidified to 0.1 M with HCl, heated to 80 °C for two hours to released 5-hydroxy-mesotrione (3.2% TRR, 0.001 mg/kg) and MNBA (2.4% TRR, 0.0008 mg/kg), mesotrione (1.5% TRR, 0.0005 mg/kg) and AMBA (1.1% TRR, 0.0004 mg/kg). The 56.9% TRR

(0.0183 mg/kg) in the PES was further extracted showing mesotrione and AMBA present at < 1% TRR (0.0002 mg/kg), MNBA and 5-hydroxy-mesotrione (0.4% TRR).

Table 27 Summary of radioactive residues in mature rice straw (109 DAT) following treatment of paddy water with phenyl- ^{14}C labelled mesotrione at 90 g ai/ha

Component	Solvent extraction		Acid hydrolysis of solvent extract		Acid hydrolysis of debris sample	
	% TRR	Residue	% TRR	Residue	% TRR	Residue
Mesotrione	1.8	0.0006	1.5	0.0005	0.7	0.0002
5-HO-mesotrione ^a	5.0	0.0016	3.2	0.0010	0.4	0.0001
MNBA			2.4	0.0008		
AMBA	1.2	0.0004	1.1	0.0004	0.6	0.0002
Baseline	24.6	0.0079	13.8	0.0044	13.3	0.0042
Remainder	7.6	0.0024	9.0	0.0029	3.5	0.0012
Extracted	n/a	n/a	n/a	n/a	53.7	0.0173
Unextracted ^b	56.9	0.0183	56.9	0.0183	26.1	0.0084
Loss/gain on fractionation ^b	2.8	0.0009	12.0	0.0038	1.6	0.0005
Total	99.9	0.0321	99.9	0.0321	99.9	0.0321

Unknowns: unidentified discrete components; Baseline: Polar residue components which are retained on the baseline after elution; Remainder: diffuse unassigned radioactivity comprising minor indiscrete components and areas of streaking of radioactivity

^a 5-hydroxy-mesotrione was assigned on basis of comparison with TLC data from later samples where reference standards were available

^b Not chromatographed

Peanuts

[Phenyl- ^{14}C]-mesotrione was solubilised in acetonitrile, diluted with aqueous blank formulation and applied to peanuts (*Arachis hypogaea* var. NCV 11) planted in silt loam soil in three outdoor subplots (Brown, 2003; Report 1286-01). [^{14}C]mesotrione was applied to the soil surface after planting the seeds (pre-emergence) at 0.305 and 0.796 kg ai/ha. Peanut foliage was harvested 90 DAT (50% maturity), mature peanuts and peanut hay were harvested 153 DAT.

Samples were extracted four times with water:acetonitrile (8:2) and nutmeat was extracted twice with hexane and twice with acetonitrile:water (8:2). Aliquots of the extracts were partitioned with hexane, dichloromethane or chloroform. Cellulase and amyloglucosidase enzymes were added to sodium acetate buffered subsamples of aqueous and hexane fractions (pH 4.6). Esterase, lipase, and/or pancreatin were added to TRIS-hydrochloric acid buffered subsamples of aqueous and hexane fractions (pH 7.8–8). Samples were incubated for approximately 24 hours at 42 or 47 °C. Aqueous and hexane fractions and PES were hydrolysed with 6 N hydrochloric acid under reflux followed by XAD-7 separation and/or dichloromethane partitioning. Hexane fractions were hydrolysed with 1 N potassium hydroxide in water under reflux for approximately 6 or 24 hours. Aminolysis using diethylamine was attempted. PES was refluxed over night with water to extract various complex sugars. Plant extracts cleaned up with C_{18} or NH_2 SPE. Components were separated using 2-D normal phase TLC. Reversed-phase HPLC coupled to UV detector, radioisotope flow monitor and a fraction collector was also used. Additional separation was performed by ion exchange chromatography where necessary.

Table 28 shows the residues found in peanut foliage and hay after both treatments. From 30 to 42% TRR was extracted from foliage and hay, with MNBA being the major residue. AMBA residues were released from PES after basic hydrolysis. MBA was found in trace amount only in hay treated at the highest rate.

Table 28 Summary of radioactive residues in peanut foliage and hay samples following pre-emergence treatment with [phenyl-U-¹⁴C]-mesotrione

Application Rate:		0.305 kg ai/ha		0.796 kg ai/ha	
Commodity		Foliage	Hay	Foliage	Hay
Total Radioactive Residue (%TRR):		0.028 (100)	0.012 (100)	0.064 (100)	0.028 (100)
Extracted residues (%TRR) ^a		0.012 (42.2)	0.004 (31.5)	0.026 (40.5)	0.010 (34.2)
Component		mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Aqueous following partition (%TRR)		0.007 (23.6)	0.002 (16.0)	0.012 (19.2)	0.007 (24.8)
MNBA		0.003 (10.9)	0.001 (4.0)	0.007 (10.7)	0.002 (5.8)
AMBA		0.001 (2.1)	–	< 0.001 (0.5)	< 0.001 (1.1)
MBA					< 0.001 (1.1)
Unknown 1		0.001 (4.5)	0.001 (4.0)	0.003 (4.4)	0.002 (7.6)
Unknown 2		0.001 (1.9)	< 0.001 (1.0)	–	0.001 (3.4)
Unknown 4			–		0.001 (3.3)
Others		^b	^d	^c	
Organic following partition (%TRR):		0.004 (15.8)	0.002 (13.3)	0.010 (15.6)	0.003 (8.8)
Organic after 6 N HCl hydrolysis		0.003 (12.3)	0.001 (10.3)	0.009 (14.1)	0.002 (6.7)
AMBA		0.001 (4.9)	< 0.001 (2.2)	0.003 (4.3)	< 0.001 (1.5)
Unextracted (PES)		0.014 (48.7)	0.008 (62.8)	0.038 (59.6)	0.019 (67.7)
PES treated with NaOH	MNBA	< 0.001 (1.4)	< 0.001 (1.1)	–	< 0.001 (0.6)
	MBA	–	< 0.001 (3.2)	< 0.001 (0.6)	0.001 (1.7)
	AMBA	0.003 (9.7)	< 0.001 (4.7)	0.002 (2.3)	0.001 (2.0)

^a Extract was separated into aqueous and organic fraction^b At least seven components ranging from 0.2% to 1.4% TRR^c At least eight components ranging from 0.1% to 1.2% TRR^d At least five components ranging from 0.3% to 0.7% TRR

The TRRs in hulls and nutmeat for plots treated with the higher rate were 0.025 and 0.037 mg/kg, respectively (Table 29). Extracted residues were higher from nutmeat samples (35–40%TRR) than hulls (20–23% TRR). Low levels of MNBA residues were detected in hull samples treated at both application rates (≤ 0.002 mg/kg) including residues released from PES. Traces of AMBA, 4-hydroxy-mesotrione and MBA were detected only after hydrolysis of hull PES.

Table 29 Summary of radioactive residues in peanut hulls and nutmeat samples following treatment with [phenyl-U-¹⁴C]-mesotrione

Application Rate:		0.305 kg ai/ha		0.796 kg ai/ha	
Commodity		Hulls	Nutmeat	Hulls	Nutmeat
Total Radioactive Residue, mg/kg eq.		0.011	0.013	0.025	0.037
Extracted residues		0.003 (23.0) ^a	0.005 (40.0)	0.005 (19.6) ^a	0.013 (35.9)
Aqueous following partition (%TRR):		0.002 (15.5)		0.005 (18.2)	
Acetonitrile fraction from nutmeat extract ^b			< 0.001 (3.8)		0.001 (3.9)
Hexane fraction from nutmeat extract ^c			0.005 (36.2)		0.013 (35.9)
Chromatography of aqueous partition for hulls	Component	mg/kg	%TRR	mg/kg	%TRR
	MNBA	< 0.001 (2.4)		< 0.001	1.7
	Unknown 1	< 0.001 (5.4)		0.002	6.7
	Unknown 2	< 0.001 (3.0)			
	Unknown 3	–	–	< 0.001	1.7
	Others	^d		^e	
Organic following partition of hull extract (%TRR):		< 0.001 (5.3)		< 0.001	2.2
Unextracted (PES)		0.007 (60.1)	0.008 (60.7)	0.017 (68.5)	0.023 (61.5)
PES treated using NaOH	MNBA	< 0.001 (1.2)		0.002 (7.9)	< 0.001 (2.4)
	MBA	0.001 (6.7)		–	–

Application Rate:		0.305 kg ai/ha		0.796 kg ai/ha	
	AMBA	< 0.001 (1.6)	0.002 (15.0)	< 0.001 (1.4)	< 0.001 (1.4)
	4-hydroxy-mesotrione	0.001 (6.9)		–	–

^a Extract was separated into aqueous and organic fraction

^b Low residues in the aqueous fraction and its concentrated matrices prevented TLC and HPLC analyses of samples

^c The hexane fraction was further characterised by treatment with base partition treatment with acid and further partition or with a mixture of enzymes which afforded several unknowns none greater than 0.002 mg/kg

^d At least three components ranging from 0.2% to 1.2% TRR

^e At least four components ranging from 0.2% to 1.4% TRR

In another study conducted with peanuts, [cyclohexane-2-¹⁴C]-mesotrione was applied to the soil surface after planting the seeds (pre-emergence) at rates of 0.327 kg ai/ha and 0.836 kg ai/ha (Brumback, 2003; Report 1287-01). Peanut foliage was harvested 90 DAT (50% maturity), and mature peanuts and peanut hay harvested 154 DAT. Auxiliary in vitro experiments with peanut cell culture and excised peanut shoots were performed to generate metabolites for identification. Peanut cells were dosed at 50 mg/L [cyclohexane-2-¹⁴C]-mesotrione, diluted in dimethyl sulfoxide (DMSO) and harvested 7 and 14 days after dosing. Peanut shoots were dosed at 100 mg/L and harvested after 2 days. Plant samples having radioactive residues ≥ 0.010 mg/kg were extracted with water:acetonitrile (8:2). Nutmeat was sequentially extracted with hexane, hexane/ethyl acetate and acetonitrile:water (8:2). Aliquots of the extracts were partitioned between aqueous and organic phases. An aqueous buffer mixture of 0.1 M sodium acetate buffer and 0.1 M acetic acid (1:1) was added to subsample extracts, following addition of cellulase and amyloglucosidase enzymes and incubation for 16 hours at 37 to 47 °C. Nutmeat extracts were acidified to pH 1 after microwave treatment and partitioning between water and hexane. Components in extracts were separated using 2-D normal phase TLC and/or reversed-phase or anion exchange. The components were detected with HPLC-UV detector; radioisotope flow monitor and a fraction collector were also used. Additional confirmation was undertaken using tandem mass spectrometry and ¹H-NMR for the metabolites isolated from the in vitro experiments.

Samples of peanuts treated at 0.327 kg ai/ha resulted in TRR < 0.01 mg/kg eq and no further analysis in the samples was performed. Table 30 shows the radioactive residues found in peanut samples from the higher rate plots. TRR was higher in foliage and nutmeat (0.02 mg/kg eq.), with about 40–50% being extracted. Characterisation of extracts from foliage, hay and hull samples from the 0.836 mg/kg rate showed only one significant metabolite, identified as 4-hydroxy-mesotrione, which was also generated in vitro. The peanut oil fraction was shown to be composed primarily of ¹⁴C-labelled neutral lipids resulting from metabolism of mesotrione to single carbon units that entered the carbon pool.

Table 30 Summary of radioactive residues in peanut samples following treatment with [cyclohexane-2-¹⁴C]-mesotrione at 0.836 kg ai/ha

Crop and Commodity:	Peanut foliage	Peanut hay	Peanut hulls	Peanut nutmeat
TRR, mg/kg eq.:	0.020	0.011	0.015	0.022
Initial extraction (%TRR):	0.008 (38.9%)	0.003 (28.2%)		
Aqueous acetonitrile extract (%TRR):			0.003 (19.9%)	0.001 (4.7%)
Non polar solubles (%TRR)			–	0.011 (51.4%)
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Neutral/base components	0.003 (11.7)	0.001 (6.7)	< 0.001 (4.4)	–
Acidic components	0.003 ^a (17.3)	0.002 ^{c, d} (16.0)	0.003 ^{a, c} (12.6)	–
Organic wash	< 0.001 (1.2)	< 0.001 (1.4)	< 0.001 (1.8)	–
Lipids				0.008 (37.8)
Fatty acids				0.001 (5.7)
Phospholipids				0.001 (4.1)
Unextracted (PES)	0.013 (65.9)	0.008 (71.6)	0.013 (84.5)	0.011 (50.8)

^a Three separate regions

^b Two separate regions

^c Contained a component characterised as 4-hydroxy-mesotrione (1.4% TRR)

Based on the metabolism studies conducted in cranberries, soya bean, maize, rice and peanut with labelled mesotrione, a metabolism pathway of mesotrione in plants is proposed in Figure 2. The process involves hydroxylation, hydrolysis to form MNBA, which is reduced to AMBA followed by de-amination to form MBA.

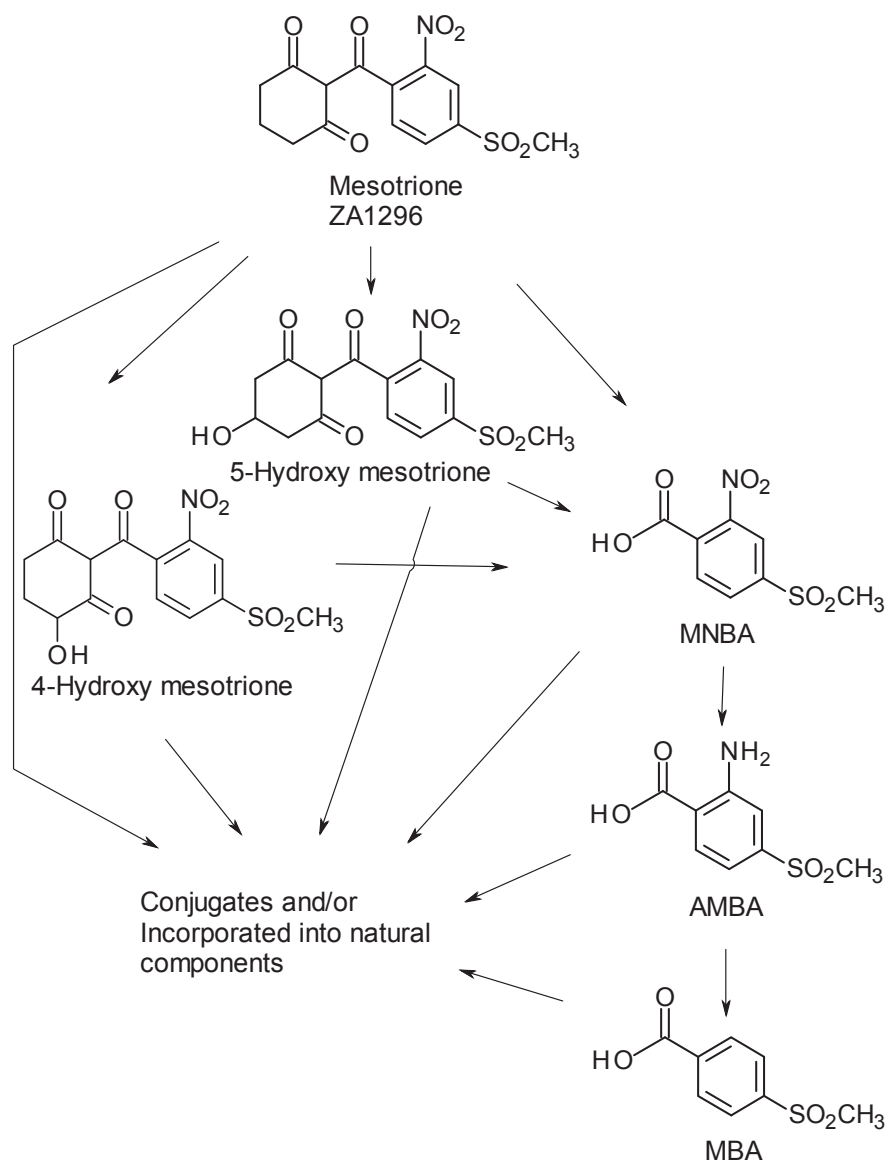


Figure 2 Proposed metabolism of mesotrione in plants

Environmental Fate in Soil

Two soil photolysis studies and five aerobic and anaerobic degradation studies of mesotrione in soil were conducted, using soils collected in 1994 and 1995 in Richmond, Wisconsin (USA). The soil characteristics are shown in Table 31.

Table 31 Silt loam soils used to investigate the metabolism of mesotrione under aerobic conditions

Soil properties	Studies RR95-082B, RR95-047B, RR96-033B, RR96-060B	Studies RR97-033B
% Sand	17.1	16.4

	Studies	Studies
Soil properties	RR95-082B, RR95-047B, RR96-033B, RR96-060B	RR97-033B
% Silt	57.7	58.9
% Clay	25.2	24.8
% Organic Matter	2.72	2.16
pH	6.2	6.1
Cation exchange capacity (meq/100 g)	12.0	10.5
% Moisture holding capacity (saturation)	—	47.2
% Moisture holding capacity (1/3 Bar)	27.4	—
Moisture holding capacity (15 Bar)	7.90	—
Microbial biomass, mg C/kg (zero to end of incubation)	^a	230 to 182–191

^a Biological activity of soil was monitored by measuring the soil respiration for 11 days soil pre-incubation. The soil was active as indicated by the formation of 0.6 mg of CO₂ per gram of soil during 11 days of incubation.

Soil photolysis

The photolysis of [phenyl-¹⁴C] mesotrione and [cyclohexane-¹⁴C] mesotrione was studied silt loam soil (Table 31) (Van Nest *et al.*, 1996; RR 96-060B). Mesotrione was applied at a nominal rate of 0.30 kg ai/ha, the soil incubated for 31 to 36 days in local sunlight (latitude 37° 56') at 20 to 24 °C. Dark control plates were maintained at 20 to 24 °C. Photolysis of mesotrione was studied under experimental conditions specified by EPA and EEC guidelines. In the EPA protocol, the soil disks were air-dried overnight before the treatment, whereas in the EEC conditions, they were further oven-dried at 35 °C for 7 hours prior to treatment. One day of xenon irradiation corresponded to 2.42 days of irradiation by sunlight in the phenyl EPA study, 2.17 days in the phenyl EEC study and 2.20 days in the cyclohexane study.

The percentage of mesotrione recovered at each sampling interval is shown in Tables 32 and 43. Over 90% AR was recovered immediately after application from the photolysed samples and 88.6% from the control samples. In general, CO₂ was the major photodegradation product from the irradiated samples of [phenyl-¹⁴C]- and [cyclohexane-¹⁴C] mesotrione. The total ¹⁴CO₂ produced after 31–36 sunlight equivalent days of photolysis was approximately 12% AR for the [phenyl-¹⁴C] mesotrione and 32% AR for the [cyclohexane-¹⁴C] mesotrione. MNBA and AMBA residues accounted for less than 12% AR each. The calculated half-lives of [phenyl-¹⁴C] mesotrione (EEC study), [cyclohexane-¹⁴C] mesotrione and [phenyl-¹⁴C] mesotrione (EPA study) were 15, 19 and 21 days, respectively, with a mean of 18.5 ± 3.6 days (equivalent to 20.3 days at 50°N; Eya, 1997).

Table 32 Mesotrione soil photolysis treated with [phenyl-¹⁴C] and [cyclohexyl-¹⁴C] mesotrione (EEC guideline; Van Nest *et al.*, 1996; RR 96-060B), in % of applied ¹⁴C in soil extracts

		Equivalent days of summer sunlight at latitude 37° 56' North [phenyl- ¹⁴ C] / [cyclohexyl- ¹⁴ C]						
		0 / 0	4.7 / 4.5	13 / 13	17 / 18	21 / 22	28 / 29	33 / 31
% of applied ¹⁴ C, [phenyl]/[cyclohexyl]	Mesotrione ^a	94.4 / 95.5	71.5 / 68.8	46.5 / 47.6	42.1 / 41.2	39.2 / 72.8	33.3 / 46.8	32.4 / 54.3
	MNBA	0.4	7.6	11.5	10	8.7	9	8.5
	AMBA	0.4	3.4	6.6	7.5	6.7	7.8	7.3
	¹⁴ CO ₂	—	1.78 / 14.9	6.23 / 25.4	8.33 / 28.3	9.74 / 29.9	11.2 / 31.4	11.8 / 31.8

^a Mean of replicate analysis

Table 33 Mesotrione silt loam soil photolysis treated with [phenyl-¹⁴C] mesotrione (EPA protocol, Van Nest *et al.*, 1996; RR 96-060B), in % of applied ¹⁴C

	Equivalent days of summer sunlight at latitude 37° 56' North									
	0.0	4.9	12	19	36	0.0 D	2.0 D	8.0 D	14 D	30 D
Mesotrione ^a	92.4	70.8	52.2	44.3	41.0	88.6	90.1	88.7	87.0	84.5
MNBA	0.1	5.4	6.8	7.6	6.5					

	Equivalent days of summer sunlight at latitude 37° 56' North									
	0.0	4.9	12	19	36	0.0 D	2.0 D	8.0 D	14 D	30 D
AMBA	0.3	2.4	5.2	6.0	5.5					
¹⁴ CO ₂			12.4	14.4	14.4					

^a Mean of replicate analysis

D=dark control. Sunlight equivalent days calculated for dark control assuming 24 h of darkness equivalent to 2 days of photolysis

Aerobic and anaerobic degradation in soil

The metabolism of [phenyl-2-¹⁴C]-mesotrione (Subba-Rao, 1996; RR 95-082B) and [cyclohexane-2-¹⁴C]-mesotrione (Vispetto & Tovshteyn, 1997; RR 95-047B) was investigated in a silt loam soil under aerobic conditions at a rate equivalent to 0.313 and 0.348 kg ai/ha, respectively. The treated soil was incubated in the dark at 25 ± 1 °C and the moisture content was maintained at 20.6% for 58–63 days under conditions which allowed ¹⁴CO₂ and any other volatilised degradates to be trapped. In addition, the metabolism of [phenyl-¹⁴C] mesotrione was further investigated at a rate of 0.233 kg ai/ha in a soil incubated in the dark at 20 ± 2 °C and 23.6% moisture content (Miller, 1997; RR 97-033B). ¹⁴CO₂ was collected in NaOH traps over the course of the study. Polyurethane foam plugs inserted in the sidearm of the flasks were analysed for any other volatile radioactive degradates. Soil was extracted with 0.05 M NH₄OH and acetone or acetonitrile. Unextracted radioactivity was further extracted from soil with 0.5 N NaOH or 0.1 N NaOH and microwave extraction.

Average recoveries ranged from 89.8 to 111% AR in the three studies. The percentages of mesotrione and resultant degradates recovered at each sampling interval are presented in Tables 34–36. Levels of radioactivity bound to the soil (unextracted) in the cyclohexane study ranged from 1.5 to 15.2% AR, with no correlation with incubation time; CO₂ accounted for 75% AR by the end of the study (Table 34). In both phenyl- labelled studies (Table 35 and 36), bound residues in soil (unextracted with 0.5 M NaOH) increased throughout the incubation period, when accounted for 34.1 to 37.0% AR. DT₅₀ and DT₉₀ values for the three studies calculated using first order kinetic ranged from 12 to 14 days and from 45 to 54 days, respectively.

Table 34 Mesotrione and degradates under aerobic conditions in silt loam soil treated with [cyclohexane-¹⁴C] mesotrione at 0.313 kg/ai/ha, in % AR

Distribution of residues	Incubation period, days									
	0	1	3	6	9	13	15	21	30	58
Mesotrione	91.0	86.7	73.6	61.5	55.7	47.7	39.5	29.8	19.6	5.67
Reminder—NH ₄ OH/acetone ^a	2.40	4.30	4.10	4.40	1.10	0.00	6.40	0.70	1.6	1.17
Unidentified ^b	— ^e	— ^e	9.07	— ^e	— ^e	9.84	— ^e	— ^e	11.4	8.59
Reminder—0.1 N NaOH ^c	— ^e	— ^e	0.83	— ^e	— ^e	1.63	— ^e	— ^e	— ^e	0.71
¹⁴ CO ₂	0.04	2.08	7.83	17.1	25.4	34.6	38.9	50.1	60.3	75.2
Trapped volatiles ^d	0.00	0.00	0.27	0.05	0.45	0.00	0.00	0.00	0.00	0.02
Unextracted	2.70 ^d	3.00 ^d	1.50	6.80 ^d	11.2 ^d	4.43	12.5 ^d	15.2 ^d	3.63	3.10
Total Recovered	96.1	96.1	96.4	89.8	93.9	96.6	97.3	95.8	96.5	93.8

^a Radioactivity remaining in the NH₄OH/acetone extracts after accounting for parent from the total via reverse-phase HPLC quantification of the residue

^b Unidentified; represents two metabolites extracted from 0.1 N NaOH microwave extractions of soil which had been previously extracted with NH₄OH, neither metabolite exceeded 10% of the applied radioactivity

^c Radioactivity remaining in the 0.1 N NaOH extract after accounting for the unidentified metabolites

^d Average of two flasks at each interval

^e NaOH extractions were not completed on these sampling points

MNBA reached a maximum level of 7.6% of the [phenyl-¹⁴C] applied radioactivity after six days and declined rapidly, with less than 1% remaining after 63 days (Table 35). The calculated DT₅₀ for MNBA in the soil was 1.1 days (Subba-Rao, 1996, RR 95-082B).

Table 35 Mesotrione degradates under aerobic conditions silt loam soil treated with [phenyl- ^{14}C]-mesotrione at 0.348 kg ai/ha, in % AR

Distribution of residues	Incubation period. Days									
	0	1	3	6	9	13	16	23	30	63
Mesotrione	97.9	96.4	89.1	74.4	57.4	46.3	35.5	27.2	19.1	6.5
MNBA	0.0	4.1	6.8	7.6	5.7	5.5	4.1	1.8	1.8	0.7
AMBA	0.0	2.2	3.7	5.5	7.4	8.7	9.4	9.7	7.3	4.5
$^{14}\text{CO}_2$	0.0	0.1	0.8	0.2	5.0	7.4	10.9	15.7	17.8	27.5
NaOH extract ^a	6.1	5.8	6.5	8.9	14.6	13.7	15.2	16.2	23.7	21.1
Unextracted	1.6	2.3	4.4	9.8	8.5	16.4	22.8	24.8	26.9	34.1
Total Recovered	106	111	111	106	98	98	98	95	97	94

^a The radioactivity not extracted by ammonium hydroxide and acetone was further extracted from soils with 0.5 N NaOH. The alkali extracts humic and fulvic acid components of soil organic matter. This is the overall total for the NaOH fraction before additional clean-up and analysis steps

In the study conducted by Miller (1997; RR 97-033B).MNBA reached a maximum level of 5.8% of the [phenyl- ^{14}C] applied radioactivity after 10 days and declined rapidly to non-detectable levels at 56 days (Table 36). In this study the DT₅₀ of MNBA, calculated using a first order metabolite model was 0.7 day.

Table 36 Mesotrione degradates under aerobic conditions silt loam soil treated with [phenyl- ^{14}C]-mesotrione at 0.233 kg ai/ha, in % AR

Distribution of residues	Incubation period. Days							
	0	3	7	10	14	21	28	56
Mesotrione	94.6	82.4	71.2	53.1	48.5	33.4	24.2	11.9
MNBA	1.4	4.5	5.0	5.8	5.1	1.0	0.9	nd
AMBA	nd	2.3	1.8	3.2	3.0	2.3	4.9	7.9
$^{14}\text{CO}_2$	na	1.1	3.1	4.9	6.9	10.9	14.7	24.5
Unidentified	nd	nd	nd	7.0	3.2	7.5	2.5	8.1
Water soluble radioactivity	na	1.7	2.9	3.9	4.0	3.5	4.0	6.1
Reminder ^a	3.0	0.7	1.0	1.1	1.3	1.7	1.8	2.0
Unextracted	0.8	5.9	11.7	16.0	22.7	33.0	37.6	37.0
Total Recovered	99.8	98.6	96.8	95	94.6	93.1	90.6	97.5

nd=not detected

na=not applicable

^a Radioactivity associated with the flocculate which precipitated after acidification of the 0.05 M NH_4OH extract

In two studies to investigate the degradation of mesotrione under anaerobic conditions, [cyclohexane-2- ^{14}C] (Vispetto & Tovshteyn, 1996; RR 95-048B) and [phenyl- ^{14}C]-mesotrione (Carley, 1996; RR 96-033B) were applied to a silt loam soil at 0.280 and 0.340 kg ai/ha, respectively. The soil was incubated in the dark at $25 \pm 2^\circ\text{C}$ and anaerobic conditions were produced by flooding the soil under nitrogen for 30 days prior to treatment.

Average total recoveries ranged from 86.2 to 105% AR throughout the incubation period, with the exception of day 59 of the phenyl-labelled experiment (Tables 36 and 37). Using the [cyclohexane-2- ^{14}C]-mesotrione, the amount of unextracted radioactivity increased to a maximum of 23.4% AR after 30 days of incubation (Table 36), while it reached 17% AR after 59 days of incubation using [phenyl- ^{14}C]-mesotrione (Table 37). AMBA was found to be the only degradation product (Table 37).

Table 36 Mesotrione and degradates under anaerobic conditions in silt loam soil treated with [cyclohexyl-2- ^{14}C]-mesotrione, in % AR

Distribution of residues	Incubation period. Days					
	0	1	3	7	14	30
Mesotrione (s + w)	102	77.6	74.5	39.4	9.32	< 0.01
$^{14}\text{CO}_2$	0.05	0.09	0.48	1.96	4.83	16.1
Trapped volatiles	0.01	0.00	0.00	0.04	0.93	0.00
Unidentified ^a (s + w)	—	—	4.48	8.19	9.18	10.4

Distribution of residues	Incubation period. Days					
	0	1	3	7	14	30
Remaining ^{14}C from microwave hydrolysis	1.13	3.21	3.97	3.91	9.12	8.0
0.1 N NaOH precipitate (fulvic and humic acids)	0.47	1.57	3.95	7.90	11.4	12.2
Reminder (s + w)	0.21	11.4	4.95	14.4	28.5	16.2
Unextracted (soil after base and microwave)	1.13	4.43	8.70	17.7	14.3	23.4
Total Recovered	105	98.3	101	93.5	87.6	86.3

(s)=Detected in soil extracts

(w)=Detected in flood water

^a Up to two compounds detected at any sampling interval, with no individual compound exceeding 9.7%Table 37 Mesotrione and degradates under anaerobic conditions in silt loam soil treated with [phenyl- ^{14}C]-mesotrione, in % AR

Distribution of residues	Incubation period. Days						
	0	1	3	7	14	30	59
Mesotrione (w)	90	80	47	18	2.7	0	0
Mesotrione (s)	0	3.2	16	18	3.5	0	0
AMBA (w)	0	0	1.5	1.8	9.2	2.7	7.9
AMBA (s)	0	0	1.8	15	28	38	14
$^{14}\text{CO}_2$	< 0.1	< 0.5	< 0.1	< 0.1	< 0.1	< 0.3	< 0.5
Reminder ^a (w)	2.5	6.4	5.1	7.9	8.1	5.9	4.9
Reminder ^a (s)	0	2.6	3.9	18	32.9	24.8	12.4
Unextracted	1.5	5.1	11.7	7.4	8.4	14.5	17
Total Recovered	93.7	97.1	87.6	86.3	92.8	86.2	56

(s)=Detected in soil extracts

(w)=Detected in flood water

^a Unresolved and background radioactivity in chromatograms

Table 38 summarizes the degradation rates of mesotrione in silt loam soil estimated under aerobic and anaerobic conditions.

Table 38 Mesotrione DT₅₀ and DT₉₀ in silt loam soil under aerobic and anaerobic conditions

Labels (ref)	Conditions	DT ₅₀ (days)	DT ₉₀ (days)
[Cyclohexane- ^{14}C] mesotrione (Vispetto & Tovshteyn, 1995)	Aerobic	14	45
[Phenyl- ^{14}C] mesotrione (Subba-Rao, 1996)	Aerobic	12	54
[Phenyl- ^{14}C] mesotrione (Miller, 1997)	Aerobic	14	47
[Cyclohexane- ^{14}C] mesotrione (Vispetto & Tovshteyn, 1996)	Anaerobic	4.1	14
[Phenyl- ^{14}C] mesotrione (Carley, 1996)	Anaerobic	3.6	12

The rate of aerobic degradation of mesotrione was studied in sandy loam, loam and clay loam soils with the characteristics summarised in Table 39 (Miller & Wilson, 1997; RR 96-090B). with [phenyl- ^{14}C]-mesotrione applied at a nominal rate of 0.165 kg ai/ha and the soils incubated for up to 56 days in the dark at 20 ± 2 °C. Moisture content was 50% of the maximum holding capacity and under conditions which allowed $^{14}\text{CO}_2$ to be trapped.

Table 39 Soils used to investigate mesotrione rate of degradation under aerobic laboratory conditions

Soil Property	ERTC ^a	GARRONNE ^b	Pickett Piece ^c
% Sand	73.2	43.5	41.3
% Silt	19.2	34.9	25.5
% Clay	7.6	21.6	33.2
% Organic Matter	0.98	1.46	5.70
pH	6.4	7.7	7.1
Cation exchange capacity (meq/100 g)	2.44	8.60	22.90
% Moisture holding capacity (zero saturation)	23.2	44.9	60.5
% Moisture holding capacity (1/3 Bar)	13.1	23.6	32.6
Moisture holding capacity (15 Bar)	—	—	—

Soil Property	ERTC ^a	GARRONNE ^b	Pickett Piece ^c
Microbial biomass. mg C/kg (zero time)	160	180	604
Microbial biomass. mg C/kg (end of incubation)	109	171	667
Soil classification	silt loam	Loam	clay loam

^a Zeneca Eastern Research Technical Centre (ERTC), North Carolina, USA

^b Zeneca Toulouse Field Station, Garomie field, Grisolles, France

^c Pickett Piece, Oxfordshire, England

Degradation of mesotrione in sandy soils was slower, with a DT₅₀ of 12 days. In loam and clay soils the DT₅₀ were 5.9 and 4.5 days, respectively Table 40.

Table 40 Mesotrione degradation rate (Miller & Wilson, 1997) in % AR

Sampling Interval (days)	Residue of mesotrione detected (% of applied ¹⁴ C)		
	Sandy loam	Loam	Clay loam
0	99.9	96.8	102
3	80.0	63.3	73.7
7	42.1	22.3	35.5
10	50.1	16.6	31.8
14	28.4	16.1	18.0
21	39.8	17.2	3.7
28	29.9	1.8	na
56	2.5	na	na
DT ₅₀ (days) mesotrione	12	5.9	4.5
DT ₉₀ (days) mesotrione	39	20	15
DT ₅₀ (days) MNBA	1.3	1.7	-

Na: not analysed as DT₉₀ already achieved

[Phenyl-U-¹⁴C]-mesotrione was applied at rates ranging from 0.60 to 0.85 mg/kg soil, equivalent to approximately 0.600 to 0.850 kg ai/ha, to 13 soils (characteristics summarised in Table 41) (Tarr, 1997, RR 93-092B). The soils were incubated in the dark, under aerobic conditions at 25 ± 2 °C, maintaining a moisture content of 100% of 1/3 bar moisture capacity and evolved ¹⁴CO₂ was trapped. The soils were incubated for up to 28 days, and samples were extracted with 0.05 M NH₄OH and acetone, acidified and portioned with ethyl acetate.

Table 41 Soils used to investigate mesotrione rate of aerobic degradation

	Clay loam	Silt loam			Loam	Loamy sand	loam sandy	Clay loam		Silty clay loam				
Soil Property	721	729	723	725	724	728	727	730	722	731	732	741	742	
% Sand	26.2	18.9	7.2	35.0	86.6	52.2	29.0	19.3	5.8	10.8	8.2	13.8	12.0	
% Silt	36.8	58.7	69.5	37.7	9.2	29.5	42.8	48.5	63.0	57.0	57.4	52.6	48.8	
% Clay	36.9	22.4	23.3	27.3	4.2	18.3	28.2	32.2	31.2	32.3	34.3	33.7	39.2	
% Organic Matter	5.4	1.9	2.1	2.3	1.0	1.6	2.5	1.7	3.2	2.0	5.2	3.2	4.5	
pH	5.8	5.6	5.8	6.2	5.0	6.4	5.6	6.1	6.0	7.2	5.7	5.5	§	
Cation exchange capacity (meq/100 g)	26.2	9.4	8.3	11.9	4.0	6.2	13.9	10.9	16.4	18.2	20.5	16.1	25.5	
% Moisture holding capacity (1/3 Bar/15 Bar)	35.0/16.9	28.0/7.3	31.2/6.4	24.2/8.8	5.2/2.4	19.5/5.4	27.0/10.2	27.4/10.4	34.8/12.6	31.9/13.2	36.8/13.9	28.6/13.2	34.4/13.9	

Average total recoveries immediately after treatment ranged from 86.4 to 92.7% of the applied radioactivity and decreased below 90% for six of the soils after 14 days of incubation (Tarr, 1997). Mesotrione degraded rapidly with DT₅₀ and DT₉₀ values ranging from 8–32 days and 27–105 days, respectively (Table 42). In most soils (10 out of 13), levels of MNBA represented less than 4% AR at any sampling day (DT₅₀ < 2 days).

Table 42 Mesotrione degradation rate (aerobic), in % AR

	721	729	723	725	724	728	727	730	722	731	732	741	742
DT ₅₀ (days)	22	13	17	8	26	8.5	24	19	11	14	16	32	8.2
DT ₉₀ (days)	73	43	55	27	86	28	80	63	35	48	53	105	27

The aerobic degradation of [phenyl-U-¹⁴C]-mesotrione was studied in subsamples of two soils from Michigan (Table 43) treated at a rate of about 1/10 the rate of application to surface soils, or 0.03 mg/kg (Tarr & Tovshteyn, 1997; RR 97-053B). The soils were incubated for up to 42 days in the dark at 20 ± 1 °C, maintaining moisture contents of 100% to 600% of 1/3 bar moisture capacity (about 5 to 10% of the dry soil weight). The higher percentage moisture content was necessary because the soil was very sandy and appeared too dry at < 100% of 1/3 bar. Mesotrione was degraded in soils collected from all depths, at a slower rate in subsoil collected from 1.8–4.8 m below the surface (Table 43).

Table 43 Properties of soils from two locations in Michigan, the USA

	Three Rivers				White Pigeon			
Soil	Loamy sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand
Depth (meters)	0–0.15	1.2–1.8	1.8–2.4	4.2–4.8	0–0.15	1.2–1.8	1.8–2.4	4.2–4.8
Acidity (pH)	6.8	6.6	7.5	8.6	6.4	6.6	7.4	8.4
WHC at Saturation (%)	12.8	12.2	22.2	14.0	14.2	28.8	22.1	18.3
WHC at 1/3 bar (%)	8.1	4.2	3.3	1.5	5.0	4.0	2.6	1.7
WHC at 15 bars (%)	3.6	2.5	1.8	0.8	3.4	2.2	1.3	0.7
Organic Matter (%)	1.4	0.4	0.2	0.1	1.3	0.5	0.2	0.2
CEC (meq/100 g)	6.9	3.0	2.2	1.3	3.6	2.5	2.1	1.2
Sand (%)	79.1	91.9	93.7	96.3	89.5	93.3	96.3	96.8
Silt (%)	12.1	3.4	3.6	1.0	3.8	2.0	0.9	1.3
Clay (%)	8.8	4.8	2.8	2.8	6.8	4.8	2.8	2.0
Dry Bulk Density (g/cm ³)	1.35	1.34	1.43	1.66	1.45	1.46	1.52	1.65
DT ₅₀ (aerobic)	6	24	105	63	13	12	68	98

Aerobic soil degradation of MNBA

One study was conducted with the purpose to determine the rate of degradation of MNBA in four soils collected from agricultural fields in England and the United States (Lay & Peyton, 2000; RR 99-098B). MNBA was applied to the incubated soil at 0.22 µg/g soil (equivalent to 0.220 kg ai/ha). Immediately after treatment of the soil, eight soil samples were taken for analysis and the remaining soil samples were incubated in the dark under aerobic conditions at 20 °C±2 °C. Samples were collected 0.3, 1, 2, 3, 4, 7, 14, 21, 45 and 60 days after treatment, and extracted with 0.05 N NH₄OH. The MNBA in the extract was chemically reduced to AMBA, which was analysed by a reversed-phase HPLC system using fluorescence detection. DT₅₀s ranged from 0.6 days to 10.6 days (Table 44).

Table 44 DT₅₀ and DT₉₀ values for MNBA in soils under laboratory aerobic conditions

Soil Origin	Spinks Soil (Three Rivers, Michigan, USA)	Delavan Soil (Wisconsin, USA)	Wisborough Green Soil (West Sussex, England)	East Anglia Soil (Suffolk, England)
	Loamy Sand	Silt Loam	Loam	Sand
pH	6.7	6	5.5	7.9
% Organic Matter	1.4	2.4	5.1	2.2
DT ₅₀ . days	8.6	10.1	0.6	10.6
DT ₉₀ . days	28.5	33.6	2.0	35.1

Aerobic soil degradation of AMBA

One study was conducted with the purpose to determine the rate of degradation of MNBA in four soils collected from agricultural fields in England and the United States (Lay & Peyton, 2000; RR 99-

098B). MNBA was applied to the incubated soil at 0.22 µg/g soil (equivalent to 0.220 kg ai/ha). Immediately after treatment of the soil, 8 soil samples were taken for analysis and the remaining soil samples were incubated in the dark under aerobic conditions at 20 °C±2°C. Samples were collected 0.3, 1, 2, 3, 4, 7, 14, 21, 45 and 60 days after treatment, and extracted with 0.05 N NH₄OH. The MNBA in the extract was chemically reduced to AMBA, which was analysed by a reversed-phase HPLC system using fluorescence detection. DT₅₀'s ranged from 0.6 days to 10.6 days (Table 45).

Table 45 DT₅₀ and DT₉₀ values for MNBA in soils under laboratory aerobic conditions

Soil Origin	Spinks Soil (Three Rivers, Michigan, USA)	Delavan Soil (Wisconsin, USA)	Wisborough Green Soil (West Sussex, England)	East Anglia Soil (Suffolk, England)
	Loamy Sand	Silt Loam	Loam	Sand
pH	6.7	6	5.5	7.9
% Organic Matter	1.4	2.4	5.1	2.2
DT ₅₀ . days	8.6	10.1	0.6	10.6
DT ₉₀ . days	28.5	33.6	2.0	35.1

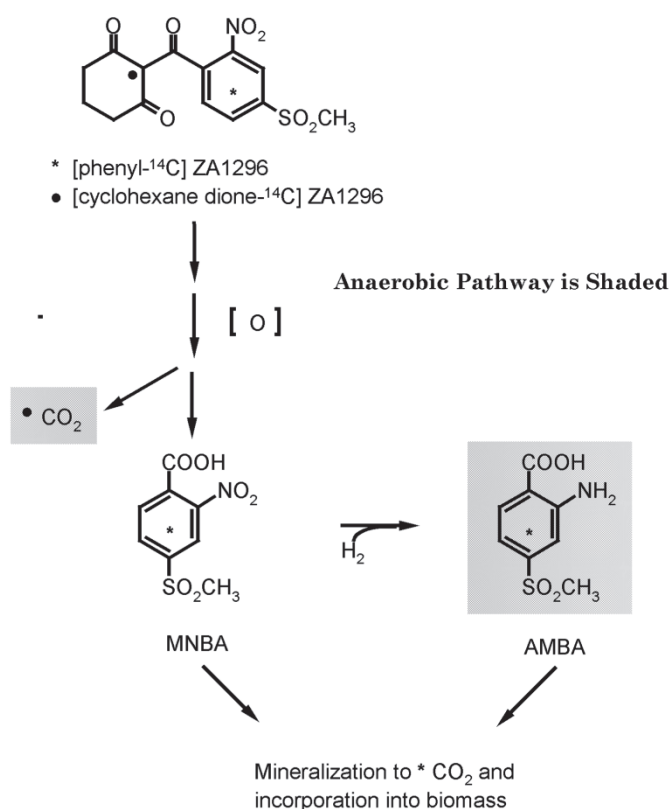


Figure 3 Proposed metabolism of mesotrione in soil

Water sediment study

Mesotrione labelled in either the cyclohexane or phenyl rings was applied to two water sediment systems at a rate equivalent to 0.200 kg ai/ha, evenly distributed throughout a 30 cm deep water body (Cary, 1999; RR 96 033B). The physico-chemical characteristics of the systems are shown in Table 48. The systems were incubated up to 101 days in the dark at 20 °C.

Radioactivity recovered from the water/sediment systems immediately after application ranged from 93% and 102% of the applied dose. The mean recovery from all the samples taken throughout the remainder of the 101 days incubation period was 100% AR. DT₅₀ were 3–6 days (Table 46). The level of mesotrione in the sediment did not exceed 4% AR at any time point, and DT₅₀ and DT₉₀ values in the water-sediment were equivalent to the ones in the water phase.

Table 46 Physico-chemical characteristics of the sediments and water prior to the study start

Sediment			Water		
Property	Old Basing	Virginia Water	Properties	Old Basing	Virginia Water
Soil classification	Sandy loam	Sand	Dissolved oxygen	82	77
Sand (%)	57	96	pH (at collection)	7.2	6.8
Silt (%)	26	2	Suspended solids mg/L	72.0	122
Clay (%)	18	3	Organic carbon, mg/L	64.9	37.1
Organic Matter (%)	7.5	0.5	Total oxidised nitrogen mg/L	5.5	2.0
pH	7.8	7.5	Nitrite mg N/L	< 0.1	< 0.1
Cation exchange capacity, eq/100 g	16.3	1.3	Nitrate mg N/L	5.5	2.0
Phosphorus, mg/kg	19.8	16.9	Ammonia mg N/L	< 0.1	< 0.1
Total nitrogen (%)	0.708	0.033	Orthophosphate mg P/L	< 0.50	< 0.50
			Calcium mg/kg Ca/L	115	37.2
			Magnesium mg/L	1.8	11.4
			Total hardness mg CaCO ₃ /L	294	140
			Conductivity µS/cm	503	379
			Alkalinity	310	52.7
Old Basing	DT ₅₀ : 3 days; DT ₉₀ : 10 days				
Virginia Water	DT ₅₀ : 6 days; DT ₉₀ : 30 days				

[Phenyl-¹⁴C] mesotrione was degraded to MNBA and AMBA (Tables 49 and 50). MNBA was only detected in the Virginia Water system. It reached a maximum of 8% AR on Day 3 and was almost entirely present in the water phase. AMBA was distributed in both the water and the sediment and reached up to 18% at Day 28 in the ‘Old Basing’ system and up to 19% in ‘Virginia Water’ system at Day 14.

Table 47 Distribution of radioactivity in ‘Old Basing’ sediment treated with [Phenyl-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
	¹⁴ CO ₂	nd	0.0	0.0	0.0	0.0	0.4	1.0	1.8	2.9	5.5
Surface Water	Mesotrione	73.2	39.2	16.6	9.9	1.4	0.0	0.0	0.0	0.0	0.0
	MNBA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	0.0	2.6	5.0	7.2	7.1	9.6	9.2	6.6	8.1	7.1
	Baseline	8.3	7.9	5.9	7.4	6.5	3.6	4.5	3.7	3.6	3.9
	Others	6.0	6.2	11.3	12.8	7.1	3.2	4.4	3.3	2.1	2.3
Sediment	Mesotrione	nd	0.5	0.9	0.9	0.3	0.0	0.0	0.0	0.0	0.0
	MNBA	nd	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	nd	2.0	4.7	5.2	5.8	7.9	6.1	7.9	7.0	6.6
	Baseline	nd	1.9	3.2	2.8	4.0	3.2	3.4	2.9	3.6	4.0
	Others	2.9	6.6	6.9	7.6	8.0	7.4	5.2	6.4	6.8	6.0
	Unextracted	2.3	21.3	48.1	55.5	64.7	70.8	67.3	73.8	75.0	73.7
Surface Water + Sediment	Mesotrione	73.2	39.6	17.6	10.8	1.7	0.0	0.0	0.0	0.0	0.0
	MNBA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	0.0	4.5	9.7	12.4	12.9	17.5	15.3	14.5	15.1	13.8
	Unidentified ^a	17.3	22.6	27.3	30.6	25.7	17.4	17.5	16.3	16.1	16.1
	Unextracted	2.3	21.3	48.1	55.5	64.7	70.8	67.3	73.8	75.0	73.7
Total		92.7	88.1	102.7	109.3	105.0	106.1	101.1	106.4	109.1	109.1

^a Unidentified is Baseline+ Others (from TLC of extracts). 0= < 0.05%

nd=Not determined

Table 48 Distribution of radioactivity in 'Virginia Water' sediment treated with [Phenyl-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
¹⁴ CO ₂		nd	0.0	0.0	0.0	0.1	2.6	6.6	9.9	12.1	15.6
Surface Water	Mesotrione	71.2	53.5	41.0	34.8	10.7	3.0	0.5	0.0	Nd	nd
	MNBA	0.0	7.4	4.9	0.8	1.3	2.5	0.9	0.0	Nd	nd
	AMBA	0.0	2.5	4.8	3.0	11.5	9.0	2.9	0.8	Nd	nd
	Baseline	11.4	12.3	7.7	8.5	8.8	4.9	2.9	2.4	Nd	nd
	Others	7.8	6.2	11.6	8.1	8.3	6.4	3.8	2.2	4.3	2.2
Sediment	Mesotrione	nd	3.8	1.3	1.9	0.3	0.0	0.0	0.0	0.0	0.0
	MNBA	nd	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	nd	1.2	2.7	6.4	7.7	6.1	6.2	3.0	4.3	3.1
	Baseline	nd	2.5	4.6	9.2	11.5	10.4	11.3	12.5	12.0	12.1
	Others	5.3	4.4	7.7	9.0	12.6	12.6	11.5	14.7	13.0	9.9
	Unextracted	0.2	3.7	12.4	20.0	27.5	42.1	47.7	56.5	54.8	64.5
Surface Water.+ Sediment	Mesotrione	71.2	57.3	42.2	36.6	10.9	3.0	0.5	0.0	0.0	0.0
	MNBA	0.0	7.9	4.9	0.8	1.3	2.5	0.9	0.0	0.0	0.0
	AMBA	0.0	3.7	7.5	9.4	19.2	15.1	9.1	3.8	4.3	3.1
	Unidentified ^a	24.4	25.4	31.6	34.8	41.2	34.3	29.6	31.8	29.4	24.2
	Unextracted	0.2	3.7	12.4	20.0	27.5	42.1	47.7	56.5	54.8	64.5
Total		95.8	98.0	98.6	101.7	100.3	99.6	94.3	102.0	100.6	107.4

^a Unidentified is Baseline+ Others (from TLC of extracts)

0.0= < 0.05%

nd=Not determined

[Cyclohexane-¹⁴C] mesotrione degraded into CO₂, which accounted for approximately 27% of the applied radioactivity at Day 101 in both the 'Virginia Water' and 'Old Basing' systems respectively (Tables 49–50).

Table 49 Distribution of radioactivity in 'Old Basing' sediment treated with [Cyclohexane-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
¹⁴ CO ₂		nd	0.1	0.2	0.9	3.0	12.5	18.5	22.1	24.7	27.8
Surface Water	Mesotrione	86.0	74.7	31.7	16.3	12.7	0.0	0.4	nd	Nd	nd
	Baseline	8.7	10.4	8.1	8.7	8.2	4.6	2.6	nd	Nd	nd
	Others	5.6	8.2	17.6	14.0	12.4	5.1	1.6	3.8	3.0	2.5
Sediment	Mesotrione	nd	nd	0.0	0.0	0.0	0.0	0.0	nd	Nd	nd
	Baseline	nd	nd	2.5	2.9	3.6	2.8	2.7	2.4	Nd	nd
	Others	0.8	1.8	8.2	9.5	11.2	8.8	3.4	7.0	8.8	7.6
	Unextracted	0.9	3.2	28.8	39.2	43.8	57.6	58.4	60.6	58.0	63.8
Surface water + Sediment	Mesotrione	86.0	74.7	31.7	16.3	12.7	0.0	0.4	nd	Nd	nd
	Unidentified ^b	15.1	20.4	36.4	35.1	35.5	21.3	10.3	13.2	11.8	10.1
	Unextracted	0.9	3.2	28.8	39.2	43.8	57.6	58.4	60.6	58.0	63.8
Total		102.0	98.4	97.1	91.5	95.0	91.4	87.6	95.9	94.5	101.7

^a Any summation differences in values within the table result from rounding of numbers within individual calculations^b Unidentified is Baseline + Others (from TLC of extracts)

0.0= < 0.05%

nd=Not determined

Table 50 Distribution of radioactivity in 'Virginia Water' sediment treated with [Cyclohexane-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
¹⁴ CO ₂		nd	0.3	0.7	2.1	3.6	8.6	16.5	21.0	23.6	26.8
Surface	Mesotrione	80.0	na	59.7	26.4	33.0	4.8	0.8	0.0	nd	nd

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
Water	Baseline	9.5	na	9.1	6.0	7.0	2.1	2.4	0.0	nd	nd
	Others	6.8	na	18.4	10.5	7.6	4.1	3.6	4.4	4.2	4.8
Sediment	Mesotrione	nd	nd	1.4	0.7	1.9	0.0	0.0	0.0	0.0	0.0
	Baseline	nd	nd	3.3	11.5	9.0	9.6	14.9	12.0	8.7	8.2
	Others	3.1	1.1	5.4	17.1	18.3	20.3	20.4	17.2	16.9	14.7
	Unextracted	0.1	0.4	5.7	24.6	19.8	48.4	42.9	48.0	43.9	44.7
Surface water + Sediment	Mesotrione	80.0	na	61.2	27.1	34.9	4.8	0.8	0.0	0.0	0.0
	Unidentified ^a	19.5	na	36.2	45.1	41.8	36.1	41.3	33.6	29.8	27.7
	Unextracted	0.1	na	5.7	24.6	19.8	48.4	42.4	48.0	43.9	44.7
Total		99.6	103.0	103.8	98.9	100.1	97.4	101.0	102.6	97.3	99.2

^a Unidentified is Baseline+ Others (from TLC of extracts)

0.0= < 0.05%

nd=Not determined

na=Not available

Degradation in soil-field studies

Six field studies were conducted to evaluate the rate of degradation of mesotrione in soil. A single application of a 0.100 kg ai/L suspension concentrate of mesotrione was applied to bare soil at rates of 0.150 and 0.200 g ai/ha in 1995/96 and 1996/97 seasons, respectively (Table 51). Soil samples were collected up to 30 cm depth, prepared in 10 cm horizons and analysed for mesotrione, MNBA and AMBA. Calculated DT₅₀ and DT₉₀ for mesotrione are also shown in Table 51.

In the 1996-1997 Italian trial, MNBA was detected at up to 0.031 mg/kg in 0–10 cm horizon, six days after application and declined to < 0.005 mg/kg by 89 days after treatment. AMBA was detected only in the 0-10 cm horizon six days after application (0.006 mg/kg). In one 1996–1997 German trial, MNBA reached a maximum of 0.016 mg/kg in the 0–10 cm horizon eight days after application and decreased to <0.005 mg/kg 89 days after treatment. No measurable residues of mesotrione or metabolites were detected in the soil below 10 cm.

Table 51 Field dissipation of mesotrione in Europe

Year	Location	Application Rate (kg ai/ha)	Soil Type	Soil Property		DT ₅₀	DT ₉₀	Reference
				Organic Carbon (%)	pH			
1995-1996	France (south)	0.150	0 - 10 cm (clay loam)	2.1	6.0	7	73	Graham <i>et al.</i> , 1997a
			10 - 20 cm (clay loam)	1.7	6.3			
			20 - 30 cm (clay)	0.4	7.1			
	Italy	0.150	0 - 10 cm (clay loam)	2.7	6.1	7	36	Graham <i>et al.</i> , 1997b
			10 - 20 cm (silt loam)	1.9	6.6			
			20 - 30 cm (silt clay)	0.4	7.1			
1996-1997	Germany	0.150	0 - 10 cm (sandy clay loam)	2.6	6.2	3	59	Graham <i>et al.</i> , 1997c
			10 - 20 cm (sandy clay)	0.7	6.5			
			20 - 30 cm (sandy loam)	0.4	7.2			
	Italy	0.200	0 - 10 cm (sandy loam)	0.8	8.0	8	26	Graham <i>et al.</i> , 1998a
			10 - 20 cm (sandy loam)	0.6	8.3			
			20 - 30 cm (loamy sand)	0.2	8.5			
	Germany 1	0.200	0 - 10 cm (loam)	2.5	7.0	8	26	Graham <i>et al.</i> , 1997b
			10 - 20 cm (silt loam)	1.6	7.4			
			20 - 30 cm (silt loam)	1.0	7.6			
	Germany 2	0.200	0 - 10 cm (sandy clay loam)	2.8	6.9	2	21	Wiebe, 1999
			10 - 20 cm (sandy loam)	1.6	7.1			
			20 - 30 cm (sandy loam)	0.5	7.4			

Aerobic soil degradation of AMBA

In another study, [phenyl-U-14C]-AMBA was applied at 0.213 kg ai/ha (Marth, 1997; RR 97-032B) or 0.225 kg ai/ha (Lay, 2000; RR 99-096B). Soil characteristics are shown in Table 52. The soils were incubated for up to 60 days in the dark under aerobic conditions at a temperature of 20 ± 2°C,

maintaining a moisture content of 40% of water holding capacity. Average total recoveries ranged from 92.7–98.6% AR at Day 0. AMBA DT₅₀ ranged from 2 to 6 days and no AMBA degradates exceeded 10% AR in any of the three soils (Table 52).

Table 52 AMBA rate of degradation in various soils under aerobic conditions

Soil Property	Marth, 1997			Lay, 2000	
	Wisborough Green, England	Delevan, USA	East Anglia, England	Spinks, USA	
	Clay	Silt loam	Sandy loam	Loamy sand	
% organic matter	3.1	2.4	2.46	1.4	
pH	4.9	6.4	7.9	6.7	
Cation exchange capacity, meq/100 g	10.52	12.11	8.47	6.1	
Sampling days	% of AR			Sampling days	% of AR
0	98.6	98.6	98.1	0	92.7
0.1	72.2	82.8	90.8	0.3	84.1
3	40.1	55.2	45.6	1	71.4
7	30.3	45.7	28.4	2	54.1
10	25.4	41.5	19.3	3	43.6
14	19.2	37.4	18.1	4	39.1
21	13.4	26.6	17.1	7	24.5
28	7.8	23.7	15.1	14	13.2
56	7.8	20.2	11.1	21	11.4
				45	14.1
				60	11.8
DT ₅₀ (days)	3	6	2	1.8	
DT ₉₀ (days)	39	>56	63	56	

Confined rotational crops

This study was designed to provide information on the uptake and metabolism of [phenyl-U-¹⁴C]-mesotrione in rotational crops following a single application onto the bare surface of a sandy loam soil at a rate of 0.165 kg ai/ha (Gorder *et al.*, 1997; RR 96-084B). At intervals of 120 and 300 days after treatment (DAT), three representative rotational crops (endive, radish and wheat) were sown into the treated soil. Crops planted in the 120 DAT soils were harvested: endive at 78 days after planting (DAP), radish roots and leaves at 56 DAP, wheat forage at 22 DAP, wheat hay at 57 DAP and wheat grain and straw at 134 DAP. The 300 DAT crops were not harvested due to low residues found in 120 DAT crops. Crops with residues ≥ 0.01 mg/kg were extracted with acetonitrile/water and the extracted residues (> 0.01 mg/kg) were analysed by HPLC-radiodetector.

Analysis of soil cores showed that the residue had declined to 34% of the applied radioactivity (AR) after 120 days and to 15% AR after 300 days. In soil, mesotrione accounted for 0.1% AR after 120, MNBA for 8% and AMBA for 2% AR.

Total radioactive residues in crop commodities from the 120 DAT are shown in Table 53. Residue levels were ≤ 0.035 mg/kg for feed commodities and ≤ 0.014 mg/kg eq. for food commodities. Untreated soil and pots contained low levels of radioactivity (≤ 0.004 mg/kg eq.), resulting from mineralisation of [¹⁴C]mesotrione in the soil. Unextracted residues represented ≤ 0.015 mg/kg eq. and were not further analysed.

Commodities with residues > 0.01 mg/kg eq. were extracted with acetonitrile/water. From 46 to 59% TRRs were extracted. The major metabolite in wheat commodities was MNBA accounting for 36%TRR in forage (Table 53). AMBA was present mostly in conjugated form.

Table 53 Summary of total radioactive residues in rotational crop samples grown in soil treated at 0.165 kg/ai/ha with [phenyl-2-¹⁴C]-mesotrione–120 DAT

Crop	TRR, mg/kg eq.	Extracted, mg/kg (%TRR)	PES, mg/kg (%TRR)	Losses in sample work-up, mg/kg (%TRR)
Wheat forage, 22 DAP	0.031	0.018 (58)	0.006 (21)	0.007 (21)
Wheat hay, 57 DAP	0.019	0.011 (59)	0.007 (36)	0.001 (5)
Wheat straw, 134 DAP	0.035	0.016 (46)	0.015 (43)	0.004 (11)
Wheat grain, 134 DAP	0.006	–	–	–
Endive, 78 DAP	0.014	0.007 (51)	0.007 (51)	(+2)
Radish root, 56 DAP	0.004	–	–	–
Radish leaves, 56 DAP	0.004	–	–	–

Table 54 Summary of total radioactive residues in 120 DAT wheat feed commodities

Metabolite ¹⁾	Forage		Hay		Straw	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
MNBA	0.011	36	0.003	16	0.003	9
AMBA Sulphate conjugate	0.002	6	0.002	11	0.004	10
AMBA Conjugate	0.002	5	–	–	–	–
AMBA	–	–	0.001	5	0.001	3
Others (each < 0.001) ^a	0.004	11	0.005	26	0.009	24

^a All radioactivity that did not form discrete HPLC peaks

In another study, [phenyl-U-¹⁴C]-mesotrione was applied at 0.308 kg ai/ha and 0.462 kg ai/ha, the lower rate representative of the pre-emergence use and the higher rate being representative of a pre-emergent followed by post-emergent use (Gorder *et al.*, 1996; RR 96-035B). Each application was sprayed onto the bare surface of a sandy loam soil contained in 51 cm diameter pots. The pots were sown with combinations of wheat, soy, endive and radish at intervals of 30, 120 and 300 DAT. Leaf and root crops could not be planted at 30 DAT due to crop sensitivity

Samples having radioactive residues greater than or equal to 0.01 mg/kg were extracted 2–3 times with acetonitrile/water. The 30-DAT wheat grain sample was extracted by refluxing with 2 M HCl. Solids with residues > 0.05 mg/kg (> 10% TRR) were subjected to further extraction with 1 M/10M ammonium hydroxide and 6 M HCl at ambient temperature and/or under reflux. HPLC and TLC were used for the characterisation of residues.

Radioactive residues in soil declined to 27% AR at 300 DAT. Mesotrione levels were < 1% AR between 28 and 300 DAT, forming MNBA and to a lesser extent AMBA present at the 120 and 300 day sowing intervals.

Radioactive residues in wheat feed commodities declined with increasing sowing intervals, being highest in wheat straw (2.58 mg/kg eq. at 30 DAT). In wheat grain, it was 0.038 mg/kg at 30 DAT and < 0.02 mg/kg at all other planting intervals. Soya bean residues were 0.145 mg/kg at DAT with feed commodities containing 0.462 and 0.645 mg/kg for hay and forage respectively. Residues in endive and radish root declined to 0.019 and 0.005 mg/kg respectively after 300 DAT (Table 55). Residues in control did not exceed 0.007 mg/kg eq.

Table 55 Total Radioactive Residues (mg/kg eq.) in rotational crop samples grown in soil treated with [phenyl-U-¹⁴C]-mesotrione

Crop	30 DAT (0.308 kg ai/ha)		120 DAT (0.462 kg ai/ha)		300 DAT (0.462 kg ai/ha)	
	Treated	Control	Treated	Control	Treated	Control
Wheat straw	2.58	0.005	0.233	0.003	0.197	0.001
Wheat forage	1.011	0.001	0.303	0.001	0.100	0.000
Wheat hay	0.756	0.003	0.127	0.001	0.044	0.000
Wheat grain	0.038	0.004	0.014	0.004	0.015	0.001

Crop	30 DAT (0.308 kg ai/ha)		120 DAT (0.462 kg ai/ha)		300 DAT (0.462 kg ai/ha)	
	Treated	Control	Treated	Control	Treated	Control
Soy forage	0.645	0.001	Not planted	Not planted	Not planted	Not planted
Soy hay	0.462	0.003	Not planted	Not planted	Not planted	Not planted
Soya bean	0.145	0.007	Not planted	Not planted	Not planted	Not planted
Endive	Not planted	Not planted	0.053	0.000	0.019	0.000
Radish root	Not planted	Not planted	0.037	0.000	0.005	0.005
Radish leaves	Not planted	Not planted	0.048	0.000	0.009	0.000

The major identified metabolite in all wheat samples was MNBA, reaching 0.63 mg/kg in 30 DAT wheat forage (Tables 56 and 57). AMBA was mostly present as sulphate conjugate. Wheat grain samples from 30 DAT contained MNBA at 0.003 mg/kg and an unidentified glucose conjugate (0.013 mg/kg). About 70% TRR of grain samples from 120 and 300 DAT were retained in PES, but no further work was conducted in the samples.

Table 56 Radioactive residues in wheat RACs following treatment of soil with [phenyl- ^{14}C]-mesotrione

Commodity	0.308 kg ai/ha /30 DAT				0.462 kg ai/ha /120 DAT			
	Forage	Hay	Straw	Grain*	Forage	Hay	Straw	Grain
Component	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
MNBA	0.625 (62)	0.172 (23)	0.458 (18)	0.003 (8)	0.165 (54)	0.023 (18)	0.018 (8)	
AMBA	0.086 (9)	0.083 (11)	0.330 (13)	–	0.019 (6)	0.020 (16)	0.012 (5)	
AMBA sulphate	0.088 (9)	0.131 (17)	0.317 (12)	–	0.028 (9)	0.015 (12)	0.018 (8)	
AMBA conj.	0.036 (4)	0.021 (3)	0.021 (1)	–	0.021 (7)	0.001 (1)	0.002 (1)	
4-OH mesotrione	0.014 (1)	–	–	–	–	–	–	
Mesotrione	0.012 (1)	–	–	–	–	–	–	
Unknown M2	–	0.012 (2)	0.022 (1)	–	–	–	–	
Unknown M12	–	0.011 (1)	–	–	–	0.001 (1)	–	
Unknown M13	–	0.009 (1)	0.017 (1)	–	–	–	–	
Unknown M18	–	0.018 (2)	–	–	–	–	–	
Unknown M19	–	0.017 (2)	–	–	–	–	–	
Unknown M22	–	–	0.015 (1)	–	–	–	–	
Glucose conj.	–	–	–	0.013 (34)	–	–	–	
Others (each < 0.01)	0.118 (12)	0.184 (24)	1.01 (39)	0.009 (24)	0.035 (12)	0.032 (25)	0.119 (51)	
PES	0.051 (5)	0.073 (10)	0.35 (14)	0.011 (29)	0.014 (5)	0.012 (9)	0.034 (15)	0.001 (72)
Losses on workup	0.02 (–2)	0.03 (3)	0.04 (2)	< 0.01 (5)	0.02 (7)	0.02 (18)	0.03 (13)	< 0.01 (12)
Total	1.011 (100)	0.756 (100)	2.58 (100)	0.038 (100)	0.303 (100)	0.127 (100)	0.233 (100)	0.014 (100)

Table 57 Radioactive residues in wheat RACs following treatment of soil with [phenyl- ^{14}C]-mesotrione

Commodity	0.462 kg ai/ha / 300 DAT			
	Forage	Hay	Straw	Grain ^a
Component	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
Mesotrione	–	0.001 (2)	–	–
MNBA	0.043 (43)	0.007 (17)	0.015 (8)	–
AMBA	0.007 (7)	0.003 (7)	0.014 (7)	–
AMBA conj.	0.005 (5)	0.002 (4)	0.003 (1)	–
AMBA sulphate conj.	0.011 (11)	0.009 (21)	0.026 (13)	–
Unknown	–	0.002 (4)	0.005 (3)	–
Others (each < 0.01 mg/kg)	0.016 (16)	0.011 (24)	0.072 (37)	–
PES	0.015 (15)	0.013 (30)	0.029 (15)	0.001 (71)
Loss on Partitioning	0 (0)	< 0.01 (–7)	0.03 (15)	0 (0)
Total	0.100 (100)	0.044 (100)	0.197 (100)	0.015 (100)

^a The aqueous fraction for the wheat grain sample was a hydrolysate

Radioactive residues in soy forage represented 0.645 mg/kg, of which 61% TRR was extracted (Table 58). The PES containing 38% TRR was hydrolysed releasing a further 20% TRR (0.129 mg/kg). MNBA was the major metabolite in soy forage and hay (up to 48% TRR). Over 40% TRR in the soya beans was extracted. The PES containing 54% TRR (0.078 mg/kg) was further extracted with base, acid with hexane, which solubilised a further 24% TRR (0.036 mg/kg). AMBA was the major compound in soya bean (0.024 mg/kg), in addition to a unknown glucose metabolite (0.003 mg/kg). A number of unknown metabolites were observed in all commodities, no one representing > 0.011 mg/kg.

Table 58 Radioactive residues in soya bean RACs following treatment of soil with [phenyl-U-¹⁴C]-mesotrione 0.308 kg ai/ha / 30 DAT

Commodity	Forage		Hay		Bean	
Component	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
MNBA	0.312	48	0.166	35	0.014	10
AMBA	0.069	11	0.034	7	0.024	17
Unknown M1	0.004	1	0.003	1	0.01	7
Unknown M2	0.011	2	0.013	3	—	—
Unknown M18	0.009	1	0.009	2	0.005	4
Unknown M20	0.005	1	—	—	—	—
Unknown M14	—	—	0.004	1	—	—
Unknown M15	—	—	0.005	2	—	—
Glucose	—	—	—	—	0.004	3
Others (each < 0.01 mg/kg)	0.112	17	0.096	21	0.033	23
PES	0.104	16	0.124	27	0.026	18
Loss on Partitioning	0.02	3	< 0.01	2	0.03	19
Total	0.645	100	0.462	100	0.145	100

Not applicable

The major residue in endive was MNBA, accounting for up to 38% TRR (0.02 mg/kg) at 120 DAT (Table 59). Traces of AMBA were detected at 300 DAT. A number of minor unknowns were seen, each at ≤ 0.002 mg/kg. Residue levels in radish were low (≤ 0.05 mg/kg), and distributed with slightly more radioactivity in tops than roots, with MNBA accounting for up to 53% TRR (Table 59).

Table 59 Summary of radioactive residues in endive and radish RACs following treatment of soil with [phenyl-U-¹⁴C]-mesotrione at 0.462 kg ai/ha

Rotation Period	Endive		Radish Top	Radish Root
	120 DAT	300 DAT	120 DAT	
Component	mg/kg (% TRR)	mg/kg (%TRR)	mg/kg (% TRR)	mg/kg (%TRR)
MNBA	0.02 (38)	0.007 (35)	0.025 (53)	0.013 (36)
AMBA	—	0.001 (6)	—	—
AMBA conj.	—	—	—	0.003 (8)
Unknown M1	0.001 (3)	0.002 (13)	—	—
Unknown M5	0.001 (3)	—	—	—
Others (each < 0.001 mg/kg)	0.005 (9)	0.005 (25)	0.010 (20)	0.004 (10)
PES	0.023 (43)	0.007 (37)	0.012 (25)	0.017 (45)
Total	0.053 (100)	0.019 (100)	0.048 (100)	0.037 (100)

[Cyclohexane-2-¹⁴C]-mesotrione at a nominal rate of 0.165 kg ai/ha was sprayed once onto the bare surface of a sandy loam soil contained in pots in greenhouse (Spillner *et al.*, 1997; RR 95-042B). Endive, radish and wheat were sown into the treated soil (120 DAT). Soil cores were taken immediately after sowing and at 273 and 300 DAT. Endive was harvested 63 DAP, radish roots and leaves at 56 DAP, forage wheat at 22 DAP, forage hay at 57 DAP and wheat grain and straw at 131 DAP. Total radioactive residues in all samples ranged from < 0.001 mg/kg eq. in endive and radish to 0.004 mg/kg eq. in wheat straw. Residues in control reached 0.002 mg/kg in wheat grain and straw.

Radioactive residues in soil declined to 4% of the applied radioactivity by 120 DAT. No further analysis was conducted since the radioactive residues were < 0.01 mg/kg.

In another rotational study, [cyclohexane-2-¹⁴C]-mesotrione was sprayed once at 0.308 kg ai/ha or 0.462 kg ai/ha to sandy loam soil pots (Spillner *et al.*, 1996; RR 96-005B). Wheat was planted at 30, 120 and 300 DAT, soy at 30 DAT and endive and radish at 120 DAT. Samples with residues ≥ 0.01 mg/kg were extracted with acetonitrile:water. Soya beans were first extracted with hexane to remove the oil. The 30-DAT wheat grain sample was hydrolysed using 2 N hydrochloric acid; the hydrolysate partitioned with ethyl acetate to remove the organosoluble residues, and the aqueous phase was analysed for ¹⁴C-glucose. Reverse-phase HPLC was used for the characterisation mesotrione and its metabolites.

Radioactive residues in the soil declined to 10, 5 and 4% AR after 30, 120 and 300 DAT, respectively. Radioactive residues in wheat commodities declined over time, with the highest residues found in feed (Table 60). Residues in the soy ranged from 0.017 in beans to 0.026 mg/kg in forage at 30 DAT. In root crops at 120 DAT they reached 0.004 mg/kg in radish leaves and were not further investigated.

Table 60 Summary of total radioactive residues in rotational crop samples grown in soil treated with [cyclohexane-2-¹⁴C]-mesotrione

Crop	30 DAT (0.308 kg/ha)		120 DAT (0.462 kg/ha)		300 DAT (0.462 kg/ha)	
	Treated (mg/kg)	Control (mg/kg)	Treated (mg/kg)	Control (mg/kg)	Treated (mg/kg)	Control (mg/kg)
Wheat straw	0.059	0.002	0.043	0.002	0.006	< 0.001
Wheat forage	0.054	0.001	0.017	< 0.001	0.002	< 0.001
Wheat hay	0.048	0.001	0.013	< 0.001	0.002	< 0.001
Wheat grain	0.010	0.002	0.008	0.002	0.001	< 0.001
Soy forage	0.026	0.001	–	–	–	–
Soy hay	0.021	0.001	–	–	–	–
Soya bean	0.017	0.003	–	–	–	–
Endive	–	–	0.003	< 0.001	–	–
Radish root	–	–	0.002	< 0.001	–	–
Radish leaves	–	–	0.004	< 0.001	–	–

Mesotrione was a major residue in feed wheat commodities, but was not detected in grain, in which the only identified residue was a glucose conjugate (Table 61). Residues in wheat grain was 0.01 mg/kg at 30 DAT. 4-hydroxy mesotrione was the major metabolite identified, accounting for up to 15% TRR in forage (0.008 mg/kg). Over 70% of the residues were extracted from all matrices, and PES was not analysed further.

Table 61 Radioactive residues in wheat RACs following treatment with [cyclohexane-2-¹⁴C]-mesotrione

Commodity	0.308 kg ai/ha/ 30 DAT				0.462 kg ai/ha/120 DAT		
	Forage	Hay	Straw	Grain ^a	Forage	Hay	Straw
Component	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
mesotrione	0.009 (17)	0.003 (7)	0.0034 (5.7)	–	0.001 (5)	–	0.001 (3)
4-OH	0.008 (15)	0.004 (8)	0.0021 (3.6)	–	0.002 (12)	–	0.003 (6)
Unknown 1	–	–	0.0021 (3.6)	–	–	–	< 0.001 (1)
Unknown 2	0.002 (4)	0.003 (6)	–	–	0.001 (8)	–	0.002 (5)
XanH4	0.001 (2)	0.001 (2)	0.0021 (3.6)	–	< 0.001 (3)	–	0.002 (4)
Unknown 3	0.002 (3)	0.002 (4)	0.0025 (4.3)	–	–	–	0.001 (3)
4-OGlu	0.002 (4)	0.003 (6)	0.0017 (2.8)	–	0.001 (6)	–	0.002 (4)
5-OH	0.001 (1)	0.002 (4)	0.0025 (4.3)	–	< 0.001 (2)	–	0.001 (2)
Unknown 4	0.001 (1)	0.001 (3)	0.0029 (5.0)	–	< 0.001 (2)	–	0.001 (2)
Unretained	0.007 (12)	0.009 (20)	0.0147 (24.9)	–	0.002 (10)	–	0.010 (23)
Remainder	0.006 (10)	0.005 (10)	0.0080 (13.5)	–	0.001 (8)	–	0.006 (13)
Glucose				0.007 (69)			

	0.308 kg ai/ha/ 30 DAT				0.462 kg ai/ha/120 DAT		
Commodity	Forage	Hay	Straw	Grain ^a	Forage	Hay	Straw
acetone rinse + acetonitrile	0.009 (16)	0.004 (8)	< 0.001 (1)	< 0.001 (5)	0.003 (20)	0.0026 (20)	0.001 (2)
PES	0.008 (15)	0.011 (22)	0.017 (28%)	0.003 (26)	0.004 (24)	0.0027 (21)	0.014 (32)
Total	0.056 (100)	0.048 (100)	0.059 (100)	0.010 (100)	0.017 (100)	0.013 (100)	0.043 (100)

^a The aqueous fraction for the wheat grain sample was a hydrolysate, not an extract; 4-OH: 4-hydroxy-mesotrione; 4-OGlu: 4-hydroxy-mesotrione glucoside conjugate; 5-OH: 5-hydroxy-mesotrione; XanH4: 6-(methylsulfonyl)-1-oxo-1.2.3.4-tetrahydro-9H-xanthen-9-one

About 70% of radioactivity was extracted from soy forage and hay, and PES was not analysed further. 13% TRR was extracted from soya beans with hexane and 20% TRR with acetonitrile/water mixtures (< 0.01 mg/kg); PES accounted for 64% TRR (0.01 mg/kg). Neither the soya bean extracts nor PES were analysed further. Mesotrione was the major residue in soy forage and hay (up to 11% TRR, 0.003 mg/kg), while 5-hydroxy-mesotrione and 4-hydroxy-mesotrione represented 4 to 6% TRR (Table 62).

Table 62 Radioactive residues in soy samples following treatment with [cyclohexane-2-¹⁴C]-mesotrione (0.308 kg ai/ha/30DAT)

Commodity:		Forage		Hay		Bean	
	Component	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
	Mesotrione	0.003	11	0.002	10	—	—
	5- hydroxy-mesotrione	0.001	6	0.001	5	—	—
	4-hydroxy-mesotrione	0.001	4	—	—	—	—
	4-OH—glucose conjugate	—	—	0.001	5	—	—
	XanH4	0.001	3	0.001	4	—	—
	Unretained	0.005	20	0.005	22	—	—
	Unknown 1	—	—	< 0.001	3	—	—
	Unknown 3	—	—	0.001	5	—	—
	Remainder	0.005	19	0.003	13	—	—
	Acetone rinse + acetonitrile	0.002	7	0.001	6	0.004 ^a	23 ^a
	PES	0.08	29	0.006	27	0.011	64
	Total	0.026	100	0.021	100	0.017	100

^a Organic extract also included value obtained from the hexane extraction step; XanH4: 6-(methylsulfonyl)-1-oxo-1.2.3.4-tetrahydro-9H-xanthen-9-one

The proposed pathways of mesotrione in rotated crops obtained from studies using the mesotrione labelled in phenyl and the cyclohexane are shown in Figure 4.

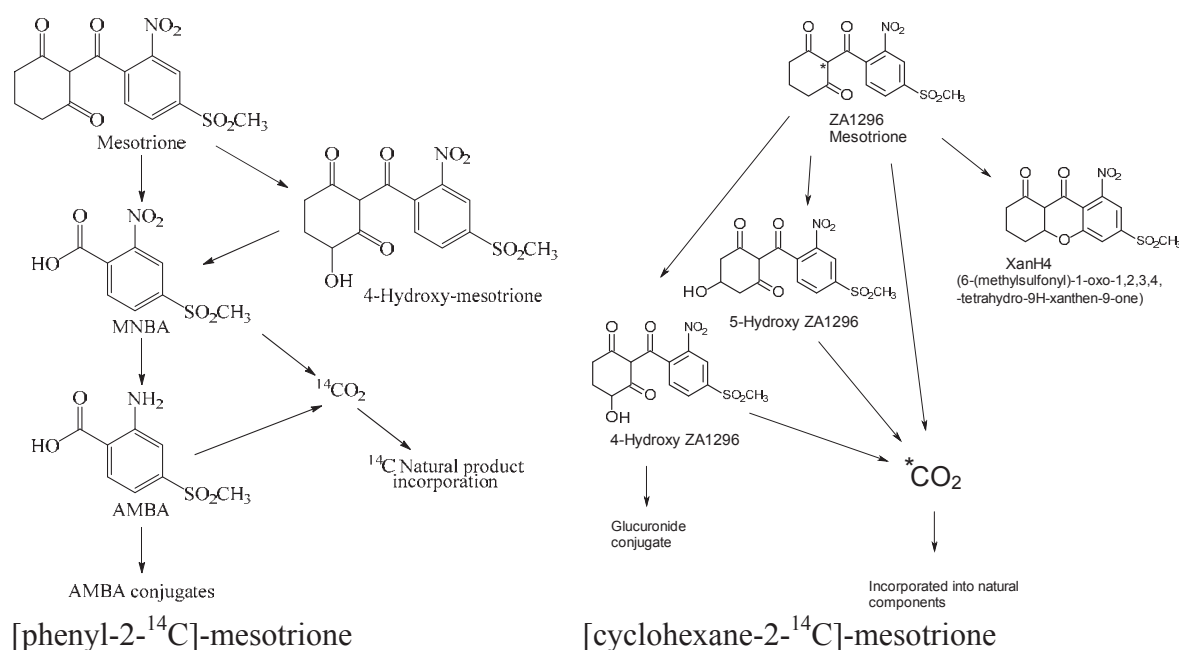


Figure 4 Proposed metabolic pathway of mesotrione in rotational crops

Analytical Methods

Vegetable crops

In Method RAM 366/01, residues of mesotrione and MNBA were extracted from crop samples with acetonitrile/water containing 10 g/L sodium chloride (50:50), an aliquot diluted with water and formic acid and cleaned up by Oasis[®] HLB SPE. The extract is partitioned into methylene chloride, evaporated to dryness, taken up with acetonitrile/water and analysed by HPLC-MS/MS, in negative ion mode, 290.9 m/z ion for quantification and no confirmation ion included. LOQ was 0.01 mg/kg in all crop matrices. Recovery data for mesotrione in corn (Hill, 2001; Report 2704 and Crook, 2001; RJ3253B), and cranberry (Salzman, 2005; 08903) are summarised in Table 63. The method was also successfully validated for MNBA.

Table 63 Recovery data for mesotrione by LC-MS/MS (negative ion mode; m/z=338 → 291)

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Corn/maize (whole plant)	0.01	5	95	5	2704 and RJ3253B
	0.10	5	100	3	
	1.0	5	91	9	
	10	5	105	4	
Corn/maize (grain)	0.01	5	86	12	
	0.10	5	84	7	
	1.0	5	96	5	
Cranberries	0.01	6	100	21	08903
	0.10	3	99	7	
	1.0	5	114	11	

Results of the recovery experiments conducted as part of the residue studies in which method RAM 366/01 was used are summarised in Table 64.

Table 64 Mesotrione procedural recovery data obtained during residue analysis

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
Cranberries	0.01	3	113	5	08903
	0.10	5	93	9	
Blueberries	0.01	9	84	7	T010288-04
	1.0	5	93	16	
Raspberries	0.01	6	90	11	
	1.0	3	94	10	
Blackberries	0.01	1	87	—	T021571-04
Okra	0.01	13	80	11	
	0.10	7	91	6	
	1.0	6	100	9	
Sweet corn (kernel)	0.01	1	80	—	04-7012
	0.02	1	81	—	
Sweet corn (cob)	0.01	1	103	—	
	0.02	1	90	—	
Field corn (kernel)	0.01	1	96	—	T000921-09-REG
	0.10	1	83	—	
Field corn (cob)	0.01	1	100	—	
	0.10	1	85	—	
Field corn (whole cob)	0.01	1	84	—	
	0.10	1	62	—	
Field corn (rest plant)	0.01	2	98	—	
	0.10	2	89	—	
Field corn (kernel)	0.01	1	97	—	T000920-09-REG
	0.10	1	98	—	
Field corn (cob)	0.01	1	115	—	
	0.10	1	78	—	
Field corn (whole cob)	0.01	1	91	—	
	0.10	1	85	—	
Field corn (rest plant)	0.01	2	104	—	
	0.10	2	88	—	
Sweet corn (forage w/o ears)	0.01	2	97	—	T001589-08
	0.50	2	90	—	
Sweet corn (ears)	0.01	1	99	—	
	0.50	1	96	—	
Sweet corn (stover)	0.01	1	105	—	
	0.50	1	81	—	
Soya bean (seed)	0.01	6	114	10	T005595-06
	0.10	6	108	13	
Soya bean (meal)	0.01	1	114	—	
	0.10	1	113	—	
Soya bean (hulls)	0.01	1	107	—	
	0.10	1	122	—	
Soya bean (oil)	0.01	1	134	—	T000908-07
	0.10	1	100	—	
Soya bean (seed)	0.01	21	83	15	
	0.10	21	87	11	
Soya bean (meal)	0.01	1	76	—	
	1.0	1	118	—	
Soya bean (hulls)	0.01	1	82	—	
	1.0	1	90	—	
Soya bean (crude oil)	0.01	1	86	—	
	1.0	1	104	—	
Soya bean (refined oil)	0.01	1	96	—	
	1.0	1	96	—	
Soya bean (AGF)	0.01	1	71	—	
	1.0	1	85	—	
Soya bean (flour)	0.01	1	87	—	
	1.0	1	95	—	
Soya bean	0.01	1	77	—	

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
(milk)	1.0	1	101	—	
Soya bean (tofu)	0.01	1	70	—	
	1.0	1	97	—	
Soya bean (soy sauce)	0.01	1	70	—	
	1.0	1	84	—	
Soya bean (miso)	0.01	1	86	—	
	1.0	1	90	—	
Asparagus	0.01	14	84	15	T021572-04
	0.05	1	82	—	
	0.10	2	88	—	
	0.50	1	97	—	
	1.0	2	94	—	
	10	1	93	—	
Rhubarb (petioles)	0.01	7	88	10	T014372-05
	0.10	5	86	8	
	1.0	2	95	—	
Millet (forage)	0.01	7	81	10	T010289-04
	1.0	7	91	7	
Millet (hay)	0.01	7	78	13	
	1.0	7	86	9	
Millet (straw)	0.01	6	95	19	
	1.0	5	93	13	
Millet (grain)	0.01	7	88	18	
	1.0	6	90	10	
Oat (forage)	0.01	14	77	8	T004407-05
	0.02	1	74	—	
	0.05	6	85	6	
	0.10	1	86	—	
	0.50	1	70	—	
	1.0	1	94	—	
Oat (hay)	0.01	12	73	7	
	0.02	3	71	6	
	0.05	4	76	4	
	0.10	3	84	2	
	0.20	1	83	—	
	0.50	1	74	—	
Oat (straw)	0.01	11	77	11	
	0.02	2	83	—	
	0.10	1	78	—	
	0.50	3	85	4	
Oat (grain)	0.01	10	75	11	
	0.02	1	70	—	
	0.05	5	80	3	
	0.20	1	84	—	
Sorghum (forage)	0.01	12	86	12	T020419-04
	0.05	3	87	11	
	0.10	2	91	—	
	0.20	2	94	—	
	0.50	1	80	—	
	1.0	1	94	—	
	2.0	1	119	—	
	20	2	99	—	
Sorghum (stover)	0.01	13	84	10	
	0.02	1	75	—	
	0.05	1	83	—	
	0.10	1	112	—	
	0.20	1	95	—	
	0.50	1	91	—	
	1.0	2	92	—	
	2.0	1	96	—	

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
Sorghum (grain)	0.01	12	90	11	
	0.05	4	78	6	
	0.10	3	92	1	
	0.20	3	101	14	
	0.50	3	98	12	
	1.0	1	94	—	
	2.0	1	105	—	
Sorghum (AGF)	0.01	2	82	—	
	0.10	2	88	—	
Sugarcane	0.01	16	71	6	T020420-04
	0.05	2	70	—	
	0.10	9	77	4	
	0.20	2	83	—	
	0.50	1	87	—	
	1.0	1	82	—	
	10	1	90	—	
Flax / Linseed (seed)	0.01	6	97	17	T010290-04
	1.0	6	98	10	
Flax / Linseed (meal)	0.01	1	85	—	
	1.0	1	102	—	

In Methods TMR0643B and TMR0882B, mesotrione and MNBA residues are extracted with acetonitrile/water (1:1). An aliquot is diluted with water, acidified, partitioned with ethyl acetate and subjected to silica SPE clean-up. Further clean-up is done by reversed phase HPLC. The MNBA, isolated in the first fraction, is chemically reduced to AMBA with SnCl_2 in HCl. The mesotrione residue, isolated in the second fraction, is oxidised to MNBA using hydrogen peroxide, which is reduced to AMBA using acidic SnCl_2 . After clean-up by C18 SPE, mesotrione and MNBA fractions are each analysed for the AMBA conversion product by a reversed-phase HPLC-fluorescence detection (ex 227 nm; em 424 nm). Mesotrione and MNBA standards were converted to AMBA concurrent with the samples. The method was validated for corn with a LOQ of 0.01 mg/kg for both mesotrione and MNBA. Independent validation was also provided. The recovery data for mesotrione are shown in Table 65.

Table 65 Mesotrione recovery data of methods TMR0643B and TMR0882B (HPLC-FL)

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Corn (forage)	0.01	13	82	16	Method TMR0643B ADD Alfness, 1996, 1997
	0.10	11	85	15	
Corn (fodder)	0.01	9	78	10	
	0.10	5	67	15	
Corn (grain)	0.01	2	109	—	Bolygo, 1996 RJ2149B Independent laboratory validation
	0.10	2	86	—	
Corn (forage)	0.01	2	85	—	
	0.05	2	94	—	
Corn (grain)	0.01	2	76	—	
	0.05	2	81	—	
Corn (fodder)	0.01	2	108	—	
	0.05	2	104	—	
Field corn (forage)	0.01	11	85	12	RR 96-018B Residue trials
	0.10	7	86	17	
Field corn (fodder)	0.01	11	78	10	
	0.10	3	82	11	
Field corn (grain)	0.01	13	95	13	
	0.10	3	100	12	
Corn (forage)	0.01	4	77	6	Method TMR0882B Alfness, 1999
	0.10	4	79	7	
Corn (fodder)	0.01	8	78	4	
	0.10	5	81	4	

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Corn (grain)	0.01	4	85	14	
	0.10	4	81	2	
Sugar cane	0.01	4	75	5	
	0.10	4	73	2	
Corn (fodder)	0.01	2	99	—	James, 1999; RR 99-062B Independent validation
	0.02	2	79	—	
Sweet corn (ears)	0.01	5	67	10	487-01 residue trial
	0.10	5	74	8	
	1.0	1	74	—	
Sweet corn (forage w/o ears)	0.01	6	88	20	
	0.10	6	84	11	
	1.0	3	90	15	
Sweet corn (forage with ears)	0.01	6	76	19	
	0.10	6	76	6	
	1.0	1	93	—	
Sweet corn (stover)	0.01	4	73	15	
	0.10	5	75	7	
	1.0	1	83	—	
	2.0	1	83	—	
Field corn (forage)	0.01	3	89	13	1847-01 residue trial
	0.10	5	78	5	
Field corn (stover)	0.01	4	87	22	
	0.10	4	80	5	
Field corn (grain)	0.01	4	80	10	
	0.10	4	84	9	
Sugarcane	0.05	2	80	—	RJ3076B; Residue trial

Method TMR0643B was radio-validated using incurred radioactive residues in a forage sample treated pre-emergence (Tarr & van Neste, 1997). An aliquot of the forage sample was extracted once with an acetonitrile:water mixture (1:1) using a high speed homogeniser, an aliquot of the supernatant partitioned three times into ethyl acetate, the combined fractions evaporated and reconstituted in acetonitrile. Residues of mesotrione and MNBA were quantified by TLC with storage-phosphor autoradiography. Levels of mesotrione and MNBA in forage were 0.008 and 0.073 mg/kg, respectively. Residues of mesotrione and MNBA obtained by exhaustive extraction within this metabolism study were 0.008 mg/kg and 0.070 mg/kg, respectively.

In Method TMR0689B (Meyers, 1996), mesotrione and MNBA residues are extracted from corn commodities with acetonitrile/water (1:1), an aliquot diluted with a sodium sulphate solution, acidified and partitioned with methylene chloride. The methylene chloride extract is evaporated and the residue heated with Jones Reagent (chromium^{VI} oxide acid solution) to convert mesotrione to MNBA. The total MNBA is extracted with ethyl acetate, evaporated to dryness, the residue dissolved in methyl ethyl ketone and the isopropyl ester of MNBA is formed by heating the mixture with 2-iodopropane and potassium carbonate. The methyl ethyl ketone is evaporated, the residue extracted with acetone, evaporated to dryness and the isopropyl-MNBA residue dissolved in toluene containing 0.05% (w/v) 5-nitrovanillin for analyse by GC-MS (monitoring ion: m/z 246). The method determines the sum of mesotrione and MNBA at a combined LOQ of 0.01 mg/kg (Table 66).

Table 66 Mesotrione procedural recovery data obtained during residue analysis by GC-MS

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Field corn (grain)	0.1	1	85	—	RR 97-043B
	0.3	2	98	—	
Field corn (silage)	0.01	1	94	—	
	0.03	3	93	8	
Field corn (grain)	0.01	3	80	5	RR 98-035B
Field corn (silage)	0.01	2	83	—	

Samples were analysed based on the QuEChERS multi-residue method (Watson, 2013). The method involves extraction with acetonitrile, addition of magnesium sulphate, sodium chloride and buffering citrate salts, centrifugation, an aliquot of the acetonitrile phase transferred to the freezer overnight (oilseed rape seed only) and cleaned up with magnesium sulphate prior to analysis by LC-MS/MS. Primary secondary amine (PSA) used in the original QuEChERS was not included due to low procedural recoveries for mesotrione. The LOQ was 0.01 mg/kg in all matrices. Recovery data in orange, maize and oilseed rape are summarised in Table 67.

Table 67 Mesotrione recovery data for method based on QuEChERS by LC-MS/MS (negative mode)

Matrix	Fortification Level (mg/kg)	n	Transition m/z=338 → 291		Transition m/z=338 → 212		Report
			Mean (%)	RSD (%)	Mean (%)	RSD (%)	
Orange (whole fruit)	0.01	5	77	14	81	15	Watson, 2013; Report S12-03251
	0.10	5	74	5.0	74	4.9	
Maize (grain)	0.01	5	89	2.0	87	5.2	
	0.10	5	89	2.7	90	3.8	
Maize (forage)	0.01	5	97	6.2	99	5.3	
	0.10	5	84	6.9	84	8.8	
Oilseed Rape (seeds)	0.01	5	80	2.5	88	14	
	0.10	5	81	4.2	82	4.2	
Maize (forage)	0.01	5	92	9	101	10	Independent validation (report S12-04607)
	0.10	4	72	5	71	8	
Maize (grain)	0.01	5	85	3	83	5	
	0.10	5	77	4	76	5	

Food of animal origin

In method TMR0914B, mesotrione and MNBA residues are extracted from milk and eggs with acetone and from animal tissues with an acetone/water (60:40). An aliquot is diluted with acidified water, partitioned twice into methylene chloride, and residues of mesotrione are converted to MNBA using hydrogen peroxide. After elimination of excess peroxide with catalase enzyme, MNBA is reduced to AMBA by heating with SnCl₂ and HCl and AMBA determined by reversed phase HPLC-fluorescence detection. The LOQ was 0.01 mg/kg in all matrices. Recovery data for mesotrione is summarised in Table 68.

Table 68 Mesotrione recovery data for Method TMR0914B

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
Meat (bovine)	0.01	6	88	4	Meyers, 1999 TMR0914B
	0.10	6	84	2	
Liver (bovine)	0.01	6	79	6	
	0.10	6	74	6	
Milk (cow)	0.01	6	90	4	
	0.10	6	93	3	
Egg (hen)	0.01	6	88	5	
	0.10	6	88	3	
Meat (bovine)	0.01	2	78	—	Brookey, 2000 RR 96-005B independent laboratory validation
	0.02	2	79	—	
Milk (cow)	0.01	2	88	—	
	0.02	2	86	—	

Mesotrione was determined in animal matrices using the modified QuEChERS, excluding PSA (Watson, 2013; Report no. S12-03250). LOQ was 0.01 mg/kg. The same procedure was independently validated (Bernal, 2013; Report no. S12-04608). The results are shown in Table 69.

Table 69 Mesotrione validation data (n=5 at each level)

Matrix	Fortification level (mg/kg)	Report no. S12-03250				Report no. S12-04608			
		m/z=338 → 291		m/z=338 → 212		m/z=338 → 291		m/z=338 → 212	
		Mean (%)	RSD (%)	Mean (%)	RSD (%)	Mean (%)	RSD (%)	Mean (%)	RSD (%)
Milk	0.01	92	1.8	93	3.9	84	3	82	4
	0.10	87	1.1	88	104	83	4	80	2
Eggs	0.01	77	3.1	77	3.0	90	6	97	5
	0.10	71	2.2	71	2.3	92	4	93	3
Muscle	0.01	92	2.6	93	3.5				
	0.10	86	2.7	88	2.9				
Fat	0.01	102	1.4	103	2.0				
	0.10	97	1.8	99	2.7				
Liver	0.01	86	2.7	88	3.9	79	5	71	9
	0.10	84	0.7	83	1.6	75	3	75	2
Kidney	0.01	99	2.0	92	3.0				
	0.10	91	0.9	87	2.2				

Storage stability under frozen conditions

Samples of maize grain, maize forage, maize fodder, radish root, and soya bean seed were homogenized and fortified at 0.1 mg/kg with either mesotrione or MNBA (Wiebe & Peyton, 1999). Duplicate samples were stored under frozen conditions ($-18^{\circ}\text{C} \pm 5^{\circ}\text{C}$) and analysed at intervals up to 44 months. Mesotrione and MNBA were quantified as AMBA by HPLC fluorescence detection (Method TMR0643B). The limit of quantification for mesotrione and MNBA in all matrices was 0.01 mg/kg. The results are shown in Table 70.

Table 70 Stability of residues in crop commodities during frozen storage, in % remaining (not corrected for analytical recovery). Mean of duplicate samples fortified at 0.1 mg/kg

Month of storage	Maize grain		Maize forage		Maize fodder		Radish root		Soya bean seed	
	mesotrione	MNBA	mesotrione	MNBA	mesotrione	MNBA	mesotrione	MNBA	mesotrione	MNBA
0	72	79	79	78	53 ^a	76	86	95	89	92
0.5					58	87				
1	89	88	70	89	61	85	79	83		
2									80	81
3			75	86	77	92			78	84
4	86	86								
5							94	89		
6									79	101
7					86	100	80	90		
8	81	80	75	84						
13							87	91 ^a		
14									102	114
15							77	80		
17	86	103			78	107				
18			74	105						
29		89				87			129	94
30					110	81				
31	109		81	85					97	
32							104	81		
40									91 ^a	92
42	86	100	65 ^a	85 ^a	73 ^a	91				
44							85	93		

^a Single value reported; duplicate sample lost during sample clean-up

Samples of blueberry, asparagus, sugarcane and okra were grounded in dry ice and fortified with mesotrione at 1.0 mg/kg (Link, 2007). Duplicate samples were prepared and stored under frozen conditions (-20°C) and analysed at intervals over 13 months for sugarcane and okra and over 22

months for blueberry and asparagus. Mesotrione was quantified by HPLC-MS/MS (LOQ of 0.01 mg/kg). The results shown in Table 71 are not corrected for procedural recovery.

Table 71 Stability of residues, in crop commodities, during frozen storage. Samples were fortified at 1.0 mg/kg of mesotrione

Commodity	Month of storage	% remaining individuals	% remaining, mean	Normalised to day 0 (%)
Blueberry	0	92; 93	93	100
	1	83; 81	82	89
	3	90; 89	90	97
	6	82; 90	86	93
	13	98; 101	100	108
	22	78; 79	79	85
Asparagus	0	92; 104	98	100
	1	97; 92	95	96
	3	92; 96	94	96
	6	94; 90	92	94
	13	98; 99	99	101
	22	93; 93	93	95
Sugarcane	0	77; 74	76	100
	1	89; 87	88	117
	3	81; 83	82	109
	6	83; 84	84	111
	13	80; 86	83	110
Okra	0	102; 101	102	100
	1	98; 96	97	96
	3	87; 91	89	88
	6	94; 99	97	95
	13	98; 97	98	96

USE PATTERNS

Mesotrione is an herbicide used pre-emergence and post-emergence for selective control of annual broad-leaved weeds, which cease growth soon after application (high or low volume sprayers). Mesotrione is registered in many countries, but only the relevant GAP information for this evaluation is given in Table 72. DAT means days after treatment.

Table 72 Registered uses of mesotrione relevant for the evaluation

Crop	Country	Formulation		Application		No	PHI (days)
		kg ai/L kg	Type	Method	Rate kg ai/ha		
Asparagus	USA	0.480	SC	Pre-emergence prior to spear emergence	0.270	1	–
Asparagus	USA	0.480	SC	Post-emergence after completion of harvesting	0.105	1	–
Asparagus	USA	0.480	SC	Pre-+ post-emergence	0.165 + 0.105	2	–
Bush and Cane berries	USA	0.480	SC	Pre-emergence	0.210	1	GS
		0.480	SC	Direct spray to the base of the plant, before bloom	0.105	2	
Cranberries	USA	0.480	SC	Broadcast foliar	0.280	2	45
Flax/Linseed	USA	0.480	SC	Post-emergence	0.210	1	NS
Maize	Canada	0.480	SC	Pre-or early post-emergence	0.14	1	GS
Maize	Canada	0.480	SC	Late post-emergence	0.10	1	GS
Maize	Germany	0.100	SC	Broadcast	0.150	1	GS
Maize	USA	0.480	SC	Pre-emergence	0.270	1	GS
Maize	USA	0.480	SC	Pre-+ post-emergence (up to 8 leaf stage)	0.165 + 0.105	2	
Millet	USA	0.480	SC	Pre-emergence	0.210	1	–
Oat	USA	0.480	SC	Pre-emergence	0.210	1	50
Oat	USA	0.480	SC	Post-emergence	0.105	1	

Crop	Country	Formulation		Application		No	PHI (days)
		kg ai/L kg	Type	Method	Rate kg ai/ha		
Okra	USA	0.480	SC	Pre-emergence	0.210	1	28
		0.480	SC	Post-emergence, directed to the weed	0.105	1	
Rhubarb	USA	0.480	SC	Pre-emergence	0.210	1	21
Rice	Republic of Korea	0.006	GR	Post planting into the water, 5–7 days after transplanting	0.09	1	Ns
Soy	USA	0.480	SC	Pre-emergence	0.210	1	Ns
Soy HT	Canada	0.480	SC	Pre-emergence	0.144	1	45
Soy HT	Canada	0.480	SC	Early Post-emergence	0.144	1	45
Soy HT	Canada	0.480	SC	Post-emergence	0.100	1	45
Soy HT	USA	0.480	SC	Pre-emergence	0.225	1	Ns
Soy HT	USA	0.480	SC	Early post-emergence, up to BBCH 13	0.225	1	Ns
Soy HT	USA	0.480	SC	Pre-+ post-emergence (BBCH 14 to 60)	0.225 + 0.125	2	GS
Sorghum	USA	0.480	SC	Pre-emergence	0.224	1	–
Sugar Cane	South Africa	0.480	SC	Broadcast	0.150	1	181
Sugar Cane	USA	0.480	SC	Pre-emergence	0.270	1	–
Sugar Cane	USA	0.480	SC	Post-emergence	0.105	2	114
Sugar Cane	USA	0.480	SC	Pre-+ post-emergence	0.36	1	114
Sweet corn	Germany	0.100	SC	Post-emergence (BBCH 12–18)	0.150	1	GS
Sweet corn	USA	0.480	SC	Pre-emergence	0.270	1	45
Sweet corn	USA	0.480	SC	Pre-+ post-emergence (up to 8 leaf stage)	0.165 + 0.105	2	45

NS= not specified

GS= growth stage

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised residue trials conducted with mesotrione on a variety of crops in Europe, Canada and USA were submitted to the Meeting. Studies were conducted according to GLP, and specified concurrent determination of residues in untreated crops gave residues < LOQ, unless specified. Residues of mesotrione arising from use patterns where rate or days after treatment (DAT) \pm 25% of GAP are underlined and considered for estimation of maximum residue levels and STMrs. When residues in samples harvested at a later stage were higher than those found at the critical PHI, they were used for the estimations. In trials conducted in the USA, duplicate or multiple field samples from replicate plots were taken for analysis at each sampling time and the mean was selected for the estimations. In total, 373 supervised trials were submitted and food commodities analysed for residues; in 193 cereal trials, feed commodities were also analysed. The data are summarized in Table 73.

Table 73 Summary of the supervised trials conducted with mesotrione

Codex group	Commodity	Region	No. of trials	Table No.
Berries and other small fruits	Blueberries, Raspberries, blackberries and cranberries	USA	25	74
Fruiting vegetables, other than Cucurbits	Okra	USA	20	75
	Sweet corn	France/USA	8/15	76
Pulses	Soy	USA	23	77
	HT Soy	USA	49	78
Stalk and stem vegetables	Asparagus	USA	24	79
	Rhubarb	USA	8	80
Cereal grains	Maize	EU/CAN/USA	3/24/24	81
	Millet	USA	15	82
	Oats	USA	34	83
	Rice	Japan/Korea	8/2	84
	Sorghum	USA	28	85
Grasses, for sugar or syrup production	Sugarcane	USA/South Africa	26/4	86

Codex group	Commodity	Region	No. of trials	Table No.
Oilseeds	Flax/Linseed	USA	17	87
Feed commodities	Corn/maize	EU/CAN/USA	101	88
	Millet	USA	15	89
	Oat	USA	34	90
	Rice	Japan/Korea	16	91
	Sorghum	USA	28	92

Berries and other small fruits

Twenty five supervised residue trials were conducted on berries in the USA using broadcast application during 2004 and 2005. Five trials were conducted on cranberries and ten trials were conducted on bush and cane berries. The results are shown in Table 74. Samples were stored deep-frozen for a maximum of 16.4 months and analysed for residues of mesotrione using method RAM 366/01 (LC-MS/MS).

Table 74 Summary of residue trials conducted with mesotrione (SC formulation) in USA on berries in 2004/2005

State	Crop (Variety)	Application rate, kg ai/ha	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Report, trial
Massachusetts	Cranberry (Early Black)	0.341 + 0.224 (+ NIS)	Fruit development Fruit sizing—Early colour	43	< 0.01 (2)	08903.05-CAR20, MA01
New Jersey	Cranberry (Early Black)	0.346 + 0.226 (+ NIS)	fruiting fruiting	44	< 0.01 (2)	08903.05-CAR20, NJ26
Oregon	Cranberry (McFarlin)	0.388 + 0.240	small green berry White fruit—Pink fruit	48	< 0.01 (2)	08903.05-CAR20, OR18
Wisconsin ^a	Cranberry (McFarlin)	0.343 + 0.220 (+ NIS)	fruiting	43	< 0.01 (2)	08903.05-CAR20, WI18
Wisconsin ^a	Cranberry (Stevens)	0.354 + 0.220 (+ NIS)	fruiting	43	< 0.01 (2)	08903.05-CAR20, WI19
New York	Blueberry (Blue Ray)	0.102	BBCH 59	77	< 0.01 (2)	T010288-04, 5A-HR04-5630
		0.211	BBCH 59	77	< 0.01 (2)	
North Caroline ^b	Blueberry (Reveille)	0.107	Early Fruit set	32	< 0.01 (2)	T010288-04, SJ-HR04-5631
				35	< 0.01 (2)	
				39	< 0.01 (2)	
				43	< 0.01 (2)	
	Blueberry (Reveille)	0.216	Early Fruit set	32	< 0.01 (2)	
				35	< 0.01 (2)	
				39	< 0.01 (2)	
				43	< 0.01 (2)	
North Caroline ^b	Blueberry (Reveille)	0.109	Early Fruit set	34	< 0.01 (2)	T010288-04, SJ-HR04-5632
		0.214	Early Fruit set	34	< 0.01 (2)	
Michigan (Freemont)	Blueberry (Blue Crop)	0.106	Early pink bud	72	< 0.01 (2)	T010288-04, NL-HR04-5633
		0.209	Early pink bud	72	< 0.01 (2)	
Michigan (Conklin)	Blueberry (Blue Ray)	0.105	Pink bud	64	< 0.01 (2)	T010288-04, NL-HR04-5634
		0.212	Pink bud	64	< 0.01 (2)	
Washington	Blueberry (Nelson and Iliot)	0.104	Fruit present—Blooming	88	< 0.01 (2)	T010288-04, WF- HR04-5635
		0.210		88	< 0.01 (2)	
Michigan (Belding)	Raspberry (K81-6)	0.106	Pre-bloom—Leaves present	64	< 0.01 (2)	T010288-04, NL-HR04-5636
				70	< 0.01 (2)	
				74	< 0.01 (2)	
				78	< 0.01 (2)	
	Raspberry (K81-6)	0.208	Pre-bloom—Leaves present	64	< 0.01 (2)	
				70	< 0.01 (2)	
				74	< 0.01 (2)	
				78	< 0.01 (2)	

State	Crop (Variety)	Application rate, kg ai/ha	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Report, trial
Oregon (Corvallis) ^c	Raspberry (Caroline)	0.105	BBCH 51	52	< 0.01 (2)	T010288-04, WG-WG-HR04-5637
	Raspberry (Caroline)	0.209	BBCH 51	52	<u>< 0.01</u> (2)	
Oregon (Hullsboro)	Blackberry (Kotata)	0.107	Pre-bloom	62	< 0.01 (2)	T010288-04, WG-HR04-5638
	Blackberry (Kotata)	0.218	Pre-bloom	62	<u>< 0.01</u> (2)	
Oregon (Corvallis) ^c	Raspberry (Caroline)	0.109	BBCH 51	83	< 0.01 (2)	T010288-04, WG-HR05-6370
	Raspberry (Caroline)	0.208	BBCH 51	83	<u>< 0.01</u> (2)	

NIS=Non-ionic surfactant

^a Trials conducted at the same location and dates, different variety

^b Trials conducted at the same location and dates, one value was considered

^c Trials conducted at the same location, but in different periods.

Okra

Twenty supervised residue trials were conducted on okra in the USA (Report T021571-04) during 2005 using either one pre-emergence application or one post-emergence application directed to the weed. A suspension concentrate (SC) formulation containing mesotrione was applied using soil surface spray pre-emergence (SS), post-emergence broadcast application over-the-top of the weed (POT) and or post-emergence directed application (PD). Samples were analysed for mesotrione residues using the method RAM 366. The results are summarized in Table 75.

Table 75 Summary of residue trials of mesotrione on okra (pods) using SC formulation in USA

State, trial	Okra variety	Application rate, kg	Growth stage	DAT (days)	Residues (mg/kg)
North Carolina, SJ-HR05-6260	Clemson Spineless	0.225 (SS)	00	73	< 0.01 (2)
		0.106 (POT)	2-3 leaf	45	< 0.01 (2)
		0.105 (PD)	(38) 10 leaf stage	28	< 0.01 (2)
		0.229 (SS) + 0.105 (POT)	00 + 2-3 leaf	45	< 0.01 (2)
		0.228 (SS) + 0.105 (PD)	00 + (36) 9 leaf stage	28	<u>< 0.01</u> (2)
Florida, VQ-HR05-6261	Clemson Spineless	0.105 (POT)	12 to 13	45	< 0.01 (2)
Mississippi, 3A-HR2005-6262	Perkins Long	0.109 (POT)	13	45	< 0.01 (2)
		0.106 (PD)	pre-bloom	28	<u>< 0.01</u> (2)
		0.232 (SS) + 0.105 (POT)	00 + 13	45	< 0.01 (2)
		0.233 (SS) + 0.103 (PD)	00 + pre-bloom	28	< 0.01 (2)
Texas, 3A-HR2005-6262	Louisiana Green Velvet	0.228 (SS)	00	84	< 0.01 (2)
				98	< 0.01 (2)
				104	< 0.01 (2)
				112	< 0.01 (2)
				119	< 0.01 (2)
		0.108 (POT)	89	0	0.19, 0.16
				15	< 0.01 (2)
				30	<u>< 0.01</u> (2)
				45	< 0.01 (2)
				52	< 0.01 (2)
		0.107 (PD)	89	0	< 0.01 (2)
				14	< 0.01 (2)
				21	< 0.01 (2)
				28	<u>< 0.01</u> (2)
				35	< 0.01 (2)
		0.228 (SS) + 0.105 (POT)	00 + 89	0	0.09, 0.2
				15	< 0.01 (2)
				30	< 0.01 (2)
				45	< 0.01 (2)
				52	< 0.01 (2)

State, trial	Okra variety	Application rate, kg)	Growth stage	DAT (days)	Residues (mg/kg)
		0.223 (SS) + 0.106 (PD)	00 + 89	0 14 21 28 35	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)
Oklahoma, SC-HR2005-6264	Clemson Spineless	0.220 (SS)	00	NA	< 0.01 (2)
		0.105 (POT)	13	45	< 0.01 (2)
		0.107 (PD)	53	28	< 0.01 (2)
		0.214 (SS) + 0.105 (POT)	00 + 13	45	< 0.01 (2)
		0.224 (SS) + 0.104 (PD)	00 + 59	28	< 0.01 (2)

SS=soil surface spray

POT=post over-the-top spray

PD=post directed application

Sweet corn

Eight post-emergence trials were conducted on sweet corn in France in 2003/2004, and 18 trials in the USA during 2001 and 2008, with either one pre-emergence and one post-emergence application or with two early post-emergence applications. Samples were analysed for mesotrione and MNBA using method RAM 366/1 (LC-MS/MS) or TM0643B (HPLC-FL). The data for mesotrione residue are summarised in Table 76. Residue levels for MNBA were < 0.01 mg/kg in all cases.

Table 76 Residue of mesotrione after the use of mesotrione on sweet corn

Country (Region) year	Sweet corn variety	Application rate (kg ai/ha) formulation	Growth Stage	DAT (days)	Crop Part	Residue (mg/kg)	Report; trial year
FRANCE 2003	620 Spirit	0.102 SC	BBCH 19	38	kernels	< 0.01	03-7049
				38	cob	< 0.01	
	620 Spirit	0.153 SC	BBCH 12	38	kernels	< 0.01	
				38	cob	< 0.01	
				61	kernels	< 0.01	
				61	cob	< 0.01	
FRANCE 2003	620 Spirit	0.98 SC	BBCH 19	38	kernels	< 0.01	03-7050
				38	cob	< 0.01	
	620 Spirit	0.143 SC	BBCH 12	62	kernels	< 0.01	
				62	cob	< 0.01	
				62	kernels	< 0.01	
				62	cob	< 0.01	
FRANCE 2004	620 Spirit	0.153 SC	BBCH 12	61	kernels	< 0.01	04-7012
				61	cob	< 0.01	
				74	kernels	< 0.01	
				74	cob	< 0.01	
	620 Spirit	0.104 SC	BBCH 59	39	kernels	< 0.01	
				39	cob	< 0.01	
FRANCE 2004	620 Spirit)	0.154 SC	BBCH 12	61	kernels	< 0.01	04-7012
				61	cob	< 0.01	
				74	kernels	< 0.01	
				74	cob	< 0.01	
	620 Spirit	0.103 SC	BBCH 59	39	kernels	< 0.01	
				39	cob	< 0.01	
USA (Wisconsin) 2001	NK 199	0.302 + 0.177 SC	pre-emergence + Vt (tassel fully emerged)	30	ears	< 0.01 (2)	487-01; NI-HR001-01
USA (Washington) 2001	Jubilee	0.303 + 0.179 SC		28	ears	< 0.01 (2)	487-01; WF-HR003-01
USA (California) 2001	Silver Queen	0.300 + 0.182 SC	BBCH 01 + BBCH 59	23	ears	< 0.01 (2)	487-01; W2-HR102-01
				30	ears	< 0.01 (2)	
				37	ears	< 0.01 (2)	

Country (Region) year	Sweet corn variety	Application rate (kg ai/ha) formulation	Growth Stage	DAT (days)	Crop Part	Residue (mg/kg)	Report; trial year
USA (N. Carolina) 2001	G90 F1	0.308 + 0.182 SC	BBCH 55	26	ears	< 0.01 (2)	487-01; SJ-HR003-01
USA (Ohio) 2001	Bodacious	0.314 + 0.190 SC		30	ears	< 0.01 (2)	487-01; NK-HR001-01
USA (New York) 2001	GH-2783	0.305 + 0.182 SC	at planting + 8–9 leaves	30	ears	< 0.01 (2)	487-01; EE-HR003-01
USA (Idaho) 2001	Sugar Buns	0.303 + 0.179 SC	post planting + pollen shed	30	ears	< 0.01 (2)	487-01; WG-HR005-01
USA (Illinois) 2001	Kandy King	0.301 + 0.186 SC	BBCH 00 + BBCH 34	31	ears	< 0.01 (2)	487-01; N4-HR003-01
USA (Pennsylvania) 2001	Argent	0.320 + 0.186 SC	BBCH 00 + early tassel	30	ears	< 0.01 (2)	487-01; EC-HR002-01
USA (Florida) 2001	Silver Queen	0.313 + 0.177 SC	Just planted + visible tassel	28	ears	< 0.01 (2)	487-01; VB-HR101-01
USA (Michigan) 2001	Excellency	0.306 + 0.180 SC	pre-emergence + Vt	23	ears	< 0.01 (2)	487-01; ED-HR006-01
				30	ears	< 0.01 (2)	
				36	ears	< 0.01 (2)	
USA (Maine) 2001	Maple Sweet	0.304 + 0.189 SC	0 + tassel initiation	30	ears	< 0.01 (2)	487-01; NF-HR002-01
USA (N. Carolina) 2008	Rogers WH0809	0.107 + 0.106 SC	BBCH 36 + BBCH 37	32	ears	< 0.01 (2)	T001589-08; E10NC081871
		0.104 + 0.104 WG	BBCH 36 + BBCH 37	32	ears	< 0.01 (2)	
USA (N. Dakota) 2008	Peaches & Cream	0.107 + 0.105 SC	BBCH 16 + BBCH 55	28	ears	< 0.01 (2)	T001589-08; C13ND081872
		0.107 + 0.106 WG	BBCH 16 + BBCH 55	28	ears	< 0.01 (2)	
USA (California) 2008	Sweetie 82	0.103 + 0.107 SC	BBCH 15 + BBCH 17	45	ears	< 0.01 (2)	T001589-08; W30CA081873
		0.105 + 0.105 WG	BBCH 15 + BBCH 17	45	ears	< 0.01 (2)	

Soya bean, dry

Twenty three supervised residue trials were conducted on soya bean in the USA during 2007 (Report T005595-06), with mesotrione applied pre-emergence direct to the soil. The DAT is driven by the normal vegetation period of the crop and is in the range of 113 to 160 days. Samples were analysed for mesotrione and MNBA using RAM 366/1. The results for mesotrione are shown in Table 77. MNBA residues were < 0.01 mg/kg in all samples.

Table 77 Summary of residue data for mesotrione (SC formulation) on soya bean seed from trials conducted in the USA in 2007

State (Location)	Soya bean variety	Application rate, kg ai/ha	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
North Carolina	S56-D7	0.212	BBCH 00	158	< 0.01 (2)	E10NC078240
South Carolina	S76-L9	0.215	BBCH 00	160	< 0.01 (2)	E11SC078241
Arkansas (New Port)	S56-D7	0.213	BBCH 00	137	< 0.01 (2)	C23AR078242
Louisiana	S56-D7	0.214	BBCH 00	140	< 0.01 (2)	E17LA078243
Arkansas (Proctor)	Garst 3512RR/	0.213	BBCH 00	113	< 0.01 (3)	C24AR078244
		1.07	BBCH 00	113	< 0.01 (3)	
Iowa (Richland)	S29-C)	0.212	BBCH 00	127	< 0.01 (2)	C18IA078245
				134	< 0.01 (2)	
				141	< 0.01 (2)	

State (Location)	Soya bean variety	Application rate, kg ai/ha	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
Iowa (Ollie)	Garst 3512 R/N	0.217	BBCH 00	152	≤ 0.01 (2)	C18IA078246
Illinois (Carlyle)	3512RR/N	0.213	BBCH 00	134	≤ 0.01 (2)	C06IL078247
		0.668	BBCH 00	134	< 0.01 (2)	
		1.111	BBCH 00	134	< 0.01 (2)	
Illinois (Wyoming)	S29-C9	0.214	BBCH 00	147	≤ 0.01 (2)	C06IL078248
Minnesota	CL081215	0.215	BBCH 00	126	≤ 0.01 (2)	C09MN078249
				133	< 0.01 (2)	
				140	< 0.01 (2)	
Indiana (Sheridan)	S29-C9	0.216	BBCH 00	141	≤ 0.01 (2)	C05IN078250
Indiana (Frankfort)	S29-C9	0.215	BBCH 00	141	≤ 0.01 (2)	C05IN078251
Missouri	S29-C9 RR	0.210	BBCH 00	122	≤ 0.01 (2)	C19MO078252
Nebraska	NK S19-R5 RR	0.218	BBCH 00	117	≤ 0.01 (2)	C17NE078253
Ohio	3512 RR/N	0.220	BBCH 00	126	≤ 0.01 (2)	C02OH078254
				134	< 0.01 (2)	
				139	< 0.01 (2)	
South Dakota	S19-R5	0.215	BBCH 01	119	≤ 0.01 (2)	C16SD078255
Kansas	04RM820605	0.212	BBCH 00	174	≤ 0.01 (2)	C22KS078256
Michigan	S19-R5	0.214	BBCH 00	156	≤ 0.01 (2)	C03MI078257
North Dakota	S10-T1	0.215	BBCH 00	140	≤ 0.01 (2)	C13ND078258
Wisconsin	CL081215	0.213	BBCH 00	127	≤ 0.01 (2)	C08WI078259

Mesotrione-tolerant soya bean, dry

Forty-nine supervised residue trials were conducted on herbicide-tolerant soya bean in the USA during 2009 and 2012 with either one pre-emergence and one post-emergence application at growth stage R1 (BBCH 60) or with one early post-emergence application at growth stage V2 (BBCH 13). Samples were analysed for mesotrione and MNBA using RAM 366/1. The results for mesotrione are shown in Table 78. MNBA residues were < 0.01 mg/kg in all samples.

Table 78 Summary of residue data for mesotrione on herbicide-tolerant soya bean (SC formulation)

State (location) year	HT Soya bean variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Portion	Residues (mg/kg)	Report; Trial
South Carolina 2009	Jack/SYHT04R	0.226 + 0.125	BBCH 00 + 60	89	Seed (m)	≤ 0.01 (2)	T000908-07; E11-9652
		0.225	BBCH 14	98	Seed (m)	≤ 0.01 (2)	
Arkansas 2009	Jack/SYHT04R	0.226 + 0.125	BBCH 00 + 60	50	Seed	< 0.01 (2)	T000908-07; C24-9653
Louisiana 2009				76	Seed (m)	≤ 0.01 (2)	
		0.226	BBCH 13	59	Seed	< 0.01 (2)	
				79	Seed (-	< 0.01	
				85	7d)	≤ 0.01 (2)	
				92	Seed (m)	< 0.01	
				99	Seed (+7d) Seed (+14d)	< 0.01	
Missouri (Fisk) 2009	Jack/SYHT04R	0.234 + 0.125	BBCH 00 + 61	45	Seed (m)	≤ 0.01 (2)	T000908-07; E18-9654
		0.234	BBCH 12	66	Seed (m)	≤ 0.01 (2)	
Iowa (Richland) 2009	Jack/SYHT04R	0.225 + 0.125	BBCH 00 + 63	49	Seed	< 0.01 (2)	T000908-07 C23-9655
				58	Seed (m)	0.02 (2)	
		0.226 + 0.124	BBCH 00 + 63	49	Seed	$0.01, 0.02$	
				58	Seed (m)	0.02 (2)	
		0.225	BBCH 12	67	Seed	< 0.01 (2)	
Missouri (La Plata) 2009	Jack/SYHT04R	0.226 + 0.126	BBCH 00 + 16	76	Seed (m)	≤ 0.01 (2)	T000908-07 C18-9656
				102	Seed (m)	≤ 0.01 (2)	
		0.222	BBCH 12.5	48	Seed	< 0.01 (2)	
				97	Seed (m)	≤ 0.01 (2)	
		1.130 + 0.623	BBCH 00 + 16	151	Seed (m)	≤ 0.01 (2)	
				102	Seed (m)	≤ 0.01 (3)	

State (location) year	HT Soya bean variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Portion	Residues (mg/kg)	Report; Trial
Iowa (Miles) 2009	Jack/SYHT04R	0.228 + 0.123	BBCH 0 + 18	45 85	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C18-0657
		0.224	BBCH 12	66 106	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Bagley) 2009	Jack/SYHT04R	0.227 + 0.122	BBCH 0 + 59	47 90	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C18-0658
		0.223 +	BBCH 12	73 116	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Berkley) 2009	Jack/SYHT04R	0.226 + 0.117	BBCH 00 + 63	45 76	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C30-9659
		0.222	BBCH 12	77 108	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
		1.147 + 0.640	BBCH 00 + 63	76	Seed (m)	0.04 (3)	
Missouri (Oregon) 2009	Jack/ SYHT04R	0.233 + 0.118	BBCH 00 + 63	43 75	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C30-9660
		0.224	BBCH 12	70 102	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Kansas 2009	Jack/ SYHT04R	0.226 + 0.121	BBCH 0 + 60- 65	47 69	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 2 C19-9661
		0.225	BBCH 13	82 93 104 110 117	Seed Seed Seed (m) Seed Seed	< 0.01 (2) < 0.01 ≤ 0.01 (2) < 0.01 < 0.01	
Michigan 2009	Jack/ SYHT04R	0.230 + 0.121	BBCH 00 + 60–65	47 70	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C19-9662
		0.229	BBCH 13	76 99	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Missouri (Dudley) 2009	Jack/ SYHT04R	0.224 + 0.124	BBCH 00 + 60	45 76	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C03-9663
		0.225	BBCH 13	81 112	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Nebraska (Stromsburg) 2009	HT Soya bean/ Jack/ SYHT04R	228 128	BBCH 00 + 61	47 55	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C23-9664
		0.223	BBCH 12	64 72	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Nebraska (Hampton) 2009	HT Soya bean/ Jack/ SYHT04R	0.228 + 0.123	BBCH 00 + 61	46 85	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E13-9665
		0.227	BBCH 12	76 115	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
North Carolina (York) 2009	Jack/ SYHT04R	0.225 + 0.125	BBCH 00 + 61	48 94	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E13-9666
		0.231	BBCH 12	83 129	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Nebraska (York) 2009	Jack/ SYHT04R	0.219 + 0.123	BBCH 00 + 61	49 86	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E13-9667
		0.230	BBCH 12	76 113	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Ohio (Richwood) 2009	Jack/ SYHT04R	0.227 + 0.125	BBCH 00 + 61	47 87	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C01-9668
		0.226	BBCH 12	66 106	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Ohio (Marysville) 2009	Jack/ SYHT04R	0.227 + 0.124	BBCH 00 + 61	47 87	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C01-9669
		0.230	BBCH 12	67 107	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Lime Springs) 2009	Jack/ SYHT04R	0.222 + 0.126	BBCH 00 + 61	46 123	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E19-9670
		0.231	BBCH 12	68 145	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	

State (location) year	HT Soya bean variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Portion	Residues (mg/kg)	Report; Trial
North Carolina (Seven springs) 2009	Jack/SYHT04R	0.226 + 0.125	BBCH 00 + 62	50 79	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E10-9671
		0.223	BBCH 13	67 96	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Richland) 2012	Jack/SYHT0H2	0.225 + 0.126	BBCH 00 + 60	94	Seed (m)	≤ 0.01 (2)	TK0112226 -1
Iowa (Bagley) 2012	Jack/SYHT0H2	0.237 + 0.125	BBCH 00 + 60	90	Seed (m)	0.02 (2)	TK0112226 -2
Iowa (Lime Spring) 2012	Jack/SYHT0H2	0.228 + 0.122	BBCH 00 + 60	97	Seed (m)	≤ 0.01 (2)	TK0112226 -3
Minneapolis 2012	Jack/SYHT0H2	0.225 + 0.127	BBCH 00 + 60	106	Seed (m)	≤ 0.01 (2)	TK0112226 -4
Nebraska 2012	Jack/SYHT0H2	0.228 + 0.126	BBCH 00 + 60	79	Seed (m)	≤ 0.01 (2)	TK0112226 -5
Iowa (Atlanta) 2012	Jack/SYHT0H2	0.235 + 0.127	BBCH 00 + 61-62	77	Seed (m)	≤ 0.01 (2)	TK0112226 -6

(m)—Mature

Asparagus

Twenty four supervised residue trials were conducted on asparagus in the USA (Report T021572-04 2005) during 2005 using either one pre-emergence soil surface spray (PSS), post-emergence over-the-top of the weeds (POT) or a combination of the two applications. Samples were analysed for mesotrione using RAM 366/1. The results are shown in Table 79.

Table 79 Summary of residue data for mesotrione on asparagus (spears) using SC formulation in the USA during 2005

State (location)	Asparagus variety	Application (kg ai/ha)	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Trial
North Caroline	Jersey Knight	0.276 (PSS)	BBCH 00	9	≤ 0.01 (2)	SJ-HR-05-6270
		0.106 (POT)	emerged	2	0.09 (2)	
		0.278 (PSS) + 0.105 (POT)	BBCH 00 + emerged	2	0.05, 0.18	
Michigan (Conklin)	Centennial	0.266 (PSS)	BBCH 00	13	≤ 0.01 (2)	NL-HR-05-6271
				14	< 0.01 (2)	
				15	< 0.01 (2)	
				16	< 0.01 (2)	
		0.106 (POT)	mature spears	0	0.61, 0.87	
				1	0.17, 0.21	
Michigan (Comstock)	Jersey Giant	0.271 (PSS) + 0.105 (POT)	BBCH 00 + mature spears	2	0.08, 0.06	NL-HR-05-6272
				3	0.05, 0.04	
				0	0.59, 0.66	
				1	0.14, 0.15	
		0.270 (PSS)	BBCH 00	2	0.09, 0.06	
				3	0.05, 0.07	
California (Gonzales)	UC 157	0.270 (PSS) + 0.105 (POT)	BBCH 00 + mature	2	0.25, 0.21	WC-HR-05-6273
		0.265 (PSS)	BBCH 00	25	≤ 0.01 (2)	
		0.104 (POT)	market size spears	2	0.05, 0.03	
California (Walnut Grove)	UC157	0.270 (PSS) + 0.105 (POT)	BBCH 00 + mature	2	0.04, 0.03	WD-HR-05-6274
		0.267 (PSS)	BBCH 08	11	≤ 0.01 (2)	
		0.106 (POT)	BBCH 45	3	≤ 0.01 (2)	
California (Fire)	UC 157	0.268 (PSS) + 0.105 (POT)	BBCH 08 + BBCH 45	3	< 0.01 (2)	WC-HR-05-6274
		0.275 (PSS)	BBCH 00	12	< 0.01 (2)	
		0.108 (POT)	emerging spears	2	0.13, 0.14	

State (location)	Asparagus variety	Application (kg ai/ha)	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Trial
Baugh)		0.280 (PSS) + 0.109 (POT)	BBCH 00 + emerging	2	0.16, 0.13	6275
Oregon	Martha Washington	0.274 (PSS)	BBCH 00	8	< 0.01 (2)	WF-HR-05-6277
		103 (POT)	BBCH 09	2	0.03, 0.05	
		0.274 (PSS) + 0.103 (POT)	BBCH 00 + BBCH 09	2	0.05, 0.08	
Washington	902-62	0.272 (PSS)	BBCH 00	10	< 0.01 (2)	WF-HR-05-6278
		0.107 (POT)	spears 2-12" tall	2	0.03 (2)	
		0.266 (PSS) + 0.106 (POT)	BBCH 00 spears 2-12" tall	2	0.04, 0.04	

PSS: Pre-emergence soil surface spray

POT: post-emergence over-the-top of the weeds

Rhubarb

Eight supervised residue trials were conducted on rhubarb in the USA (Report T014372-05) during 2006 with one application performed pre-emergence. The results are shown in Table 80.

Table 80 Summary of residue data for the use of mesotrione on rhubarb from trials conducted in USA in 2006 using SC formulation

Country (Region)	Rhubarb variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Residues (mg/kg)
Illinois		0.202	BBCH 00	28	< 0.01 (2)
				35	< 0.01 (2)
				42	< 0.01 (2)
				47	< 0.01 (2)
		0.350	BBCH 00	28	0.01, < 0.01
				35	< 0.01 (2)
				42	< 0.01 (2)
				47	< 0.01 (2)
Michigan	McDonald	0.212	pre-emergence	42	< 0.01 (2)
		0.340	pre-emergence	42	< 0.01 (2)
Oregon	Crimson	0.210	dormant	42	< 0.01 (2)
		0.343	dormant	42	< 0.01 (2)
Washington	RubyRed	0.217	BBCH 08	42	< 0.01 (2)
		0.337	BBCH 08	42	< 0.01 (2)

Field corn (maize)

Three trials with field corn were conducted in Europe in 2009, 24 in Canada in 1996/1997 and 42 in the USA in 1995 or 2001 with either one pre-emergence and one post-emergence application or with two early post-emergence applications. The results on maize grain are shown in Table 81.

Table 81 Summary of residue data for the use of mesotrione on maize (grain)

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
Canada (Ontario) 1996	Pioneer 3902	SC	0.300	Pre-emergence	123	< 0.01	RR 97-043B 94-CN-96-201
		SC	0.600	Pre-emergence	123	< 0.01	
		SC	0.200	5-6 Leaf	108	< 0.01	
		SC	0.400	5-6 Leaf	108	< 0.01	
		SC	0.300 0.200	Pre-emergence 5-6 leaf	108	< 0.01	
		SC	0.600 0.400	Pre-emergence 5-6 leaf	108	< 0.01	
Canada (Ontario) 1996	Ciba seeds G-4064	SC	0.300	Pre-emergence	122	< 0.01	RR 97-043B 94-CN-96-202
		SC	0.600	Pre-emergence	122	< 0.01	
		SC	0.200	4-5 Leaf stage	111	< 0.01	

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
		SC	0.400	4–5 Leaf stage (111	< 0.01	
		SC	0.300 0.200	Pre-emergence 4–5 leaf	111	< 0.01	
		SC	0.600 0.400	Pre-emergence 4–5 leaf	111	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	155	< 0.01	RR 98-035B 94-CN-97-901
		SC	0.600	Pre-emergence	155	< 0.01	
		SC	0.200	24	126	< 0.01	
		SC	0.400	24	126	< 0.01	
		SC	0.300 0.200	Pre-emergence 24	126	< 0.01	
		SC	0.600 0.400	Pre-emergence 24	126	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	145	< 0.01	RR 98-035B 94-CN-97-902
		SC	0.600	Pre-emergence	145	< 0.01	
		SC	0.200	23	126	< 0.01	
		SC	0.400	23	126	< 0.01	
		SC	0.300 0.200	Pre-emergence 23	126	< 0.01	
			0.600 0.400	Pre-emergence 23	126	< 0.01	
Germany 2009	Nescio	WG	0.138	BBCH 16–18	82 91 143	≤ 0.01 < 0.01 < 0.01	T000920-09- REG
Spain 2009	Castellano	WG	0.154	BBCH 15	42 71 82	< 0.01 < 0.01 < 0.01	T000921-09- REG
United Kingdom 2009	Ohio	WG	0.142	BBCH 16–17	98 112	≤ 0.01 < 0.01	T000920-09- REG
USA (Iowa, Sheffield), 1995	ICI 8543	SC	0.336 0.224	Pre-plant vegetative	109	< 0.01 (2)	RR 96-018B 63-IA-95-805
USA (Illinois, Brimfield), 1995	Hoblit 428	SC	0.336 0.224	pre-emergence	95	< 0.01 (2)	RR 96-018B 60-IL-95-806
USA (Indiana, Lafayette), 1995	Pioneer 3394	SC	0.336 0.224	pre-emergence 7–9 leaves	96	< 0.01 (2)	RR 96-018B 67-IN-95-807
USA (Michigan) 1995	NK 4640	SC	0.336 0.224	pre-bloom	114	< 0.01 (2)	RR 96-018B 24-MI-95-808
USA (Nebraska) 1995	Ottile RO 2455	SC	0.336 0.224	Pre-plant V6	88	< 0.01 (2)	RR 96-018B 68-NE-95-809
USA (Iowa, Albia), 1995	ICI 8532	SC	0.336 0.224	Pre-plant vegetative	102	< 0.01 (2)	RR 96-018B 63-IA-95-810
USA (Illinois, Towanda), 1995	Ainsworth 640	SC	0.336 0.224	Pre-plant post emergence	87	< 0.01 (2)	RR 96-018B 60-IL-95-811
USA (Indiana, Rochester), 1995	Pioneer 3394	SC	0.336 0.224	Early pre-plant 5–7 leaves	96	< 0.01 (2)	RR 96-018B 67-IN-95-812
USA (Nebraska, Waverly), 1995	Producers PH785	SC	0.336 0.224	Pre-plant V5–V6	96	< 0.01 (2)	RR 96-018B 68-NE-95-814
USA (Kansas, La Cynet), 1995	CIBA 4575	SC	0.336 0.224	early pre-plant post	68	< 0.01 (2)	RR 96-018B 37-KS-95-825
USA (Pennsylvania, Ephrata), 1995	CI 8541	SC	0.336 0.224	Pre-emergence vegetative	98	< 0.01 (2)	RR 96-018B 70-PA-95-815
		SC	0.336 0.224	pre-emergence vegetative	98	< 0.01 (2)	
USA (Minnesota, St. Peter)	Cenex 424	SC	0.336 0.224	Pre-emergence vegetative	92	< 0.01 (2)	RR 96-018B 36-MN-95-816

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
1995		SC	0.336 0.224	Pre-emergence vegetative	92	< 0.01 (2)	
USA (Ohio, Urbana) 1995	Vigoro V1122	SC	0.336 0.224	Pre-emergence	113	< 0.01 (2)	RR 96-018B 24-OH-95-817
		SC	0.336 0.224	Pre-emergence	113	< 0.01 (2)	
USA (Wisconsin, Baraboo) 1995	Cenex LOL 357	SC	0.336 0.224	Pre-emergence	104	< 0.01 (2)	RR 96-018B 79-WI-95-818
		SC	0.336 0.224	Pre-emergence	104	< 0.01 (2)	
USA (Texas) 1995	G4673B (D&PL Co.)	SC	0.336 0.224	Pre-emergence pre-tassel	92	< 0.01 (2)	RR 96-018B 25-TX-95-819
		SC	0.336 0.224	Pre-emergence pre-tassel	92	< 0.01 (2)	
USA (North Carolina) 1995	Field corn (Pioneer 3165)	SC	0.336 0.224	Pre-plant rapidly growing	95	< 0.01 (2)	RR 96-018B 01-NC-95-820
		SC	0.336 0.224	Pre-plant rapidly growing	88 95 101 108	< 0.01 < 0.01 (2) < 0.01 < 0.01	
USA (Iowa, Boone) 1995	ICI 8543	SC	0.336 0.224	Early pre-plant vegetative	112	< 0.01 (2)	RR 96-018B 63-IA-95-821
		SC	0.336 0.224	Early pre-plant vegetative	112	< 0.01 (2)	
USA (Illinois, Champagne) 1995	Pioneer 3394	SC	0.336 0.224	Early pre-plant post emergence	107	< 0.01 (2)	RR 96-018B 04-IL-95-822
		SC	0.336 0.224	Pre-plant post-emergence	100 107 113 121	< 0.01 < 0.01 (2) < 0.01 < 0.01	
USA (Indiana, New Richmond) 1995	Pioneer 3394	SC	0.336 0.224	Early pre-plant 6-8 leaf	86	< 0.01 (2)	RR 96-018B 67-IN-95-823
		SC	0.336 0.224	Early pre-plant 6-8 leaf	86	< 0.01 (2)	
USA (Nebraska, Crete) 1995	ICI 8541	SC	0.336 0.224	Early pre-plant V6-V7	89	< 0.01 (2)	RR 96-018B 68-NE-95-824
		SC	0.336 0.224	Pre-plant V6-V7	89	< 0.01 (2)	
USA Iowa) 2001	Pioneer 34B23)	SC	0.268 0.225	BBCH 00 BBCH 18-19	99	< 0.01 (2)	1847-01 NE-HR-003-01
		SE SC	0.267 0.233	BBCH 00 BBCH 18/19	99	< 0.01 (2)	
		SC	0.274 0.234	BBCH 13 BBCH 18/19	99	< 0.01 (2)	
		SE SC	0.267 0.223	BBCH 13 BBCH 18/19	99	< 0.01 (2)	
USA (North Carolina)	ICI 8543)	SC	0.269 0.226	BBCH 00 75 cm high	98	< 0.01 (2)	1847-01

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
2001		SE	0.269	BBCH 00	98	< 0.01	SJ-HR-013-01
		SC	0.227	75 cm high		(2)	
		SC	0.271	BBCH 12/13	98	< 0.01	
			0.227	75 cm high		(2)	
		SE	0.270	BBCH 12/13	98	< 0.01	
USA (Illinois) 2001	Pioneer 34B24	SC	0.285	BBCH 00	112	< 0.01	1847-01 N4-HR-005-01
			0.253	BBCH 36		(2)	
		SE	0.278	BBCH 00	112	< 0.01	
		SC	0.236	BBCH 36		(2)	
		SC	0.286	BBCH 12	112	< 0.01	
USA (Nebraska) 2001	ICI 8543		0.244	BBCH 36		(2)	
		SE	0.268	BBCH 12	112	< 0.01	1847-01 NB-HR-004-01
		SC	0.237	BBCH 36		(2)	
		SC	0.270	BBCH 00	103	< 0.01	
			0.227	BBCH 19		(2)	
		SE	0.273	BBCH 00	103	< 0.01	
		SC	0.227	BBCH 19		(2)	
		SC	0.266	BBCH 12/13	103	< 0.01	NB-HR-004-01
			0.226	BBCH 19		(2)	
		SE	0.268	BBCH 12/13	103	< 0.01	
		SC	0.226	BBCH 19		(2)	

Millet

Fifteen supervised residue trials were conducted on millet in the USA during 2004 (Report T010289-04) with one application performed pre-emergence. The results on millet grain are shown in Table 82.

Table 82 Summary of residue data for the use of mesotrione on millet (grain) using SC formulation in the USA in 2004

State	Millet variety	Application Rate (g ai/ha)	Growth Stage	PHI (days)	Residue (mg/kg)	Trial number
Illinois	Max Perl	0.109	at planting	130	< 0.01 (2)	4A-HR-04- 5640
		0.214	at planting	130	<u>< 0.01</u> (2)	
		0.108	BBCH 32	113	< 0.01 (2)	
Nebraska	Huntsman	0.103	at planting	84	< 0.01 (2)	4A-HR-04- 5641
		0.210	at planting	84	<u>< 0.01</u> (2)	
		0.103	BBCH 12	61	< 0.01 (2)	
South Dakota	Rise	0.104	at planting	95	< 0.01 (2)	4A-HR-04- 5642
		0.208	at planting	95	<u>< 0.01</u> (2)	
		0.105	BBCH 21	69	< 0.01 (2)	
Colorado (Ault) ^a	Early Bird	0.107	at planting	132	< 0.01 (2)	4A-HR-04- 5643
		0.212	at planting	132	<u>< 0.01</u> (2)	
		0.107	vegetative	92	< 0.01 (2)	
Colorado (Ault) ^a	Early Bird	0.110	at planting	104	< 0.01 (2)	4A-HR-04- 5644
				111	< 0.01 (2)	
				120	< 0.01 (2)	
		0.218	at planting	104	<u>< 0.01</u> (2)	
				111	< 0.01 (2)	
		0.108	vegetative	120	< 0.01 (2)	
				64	< 0.01 (2)	
				71	< 0.01 (2)	
				80	< 0.01 (2)	

^a Trials conducted at the same site during the same period. Only one was considered

Oat

Thirty four supervised residue trials were conducted in oat in the USA (Report T004407-05) during 2005 with either one pre-emergence or one post-emergence application. The results on oat grain are shown in Table 83.

Table 83 Summary of residue data for the use of mesotrione on oat (grain) using SC formulation in 2005 in the USA

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
New York	Armor	0.214	BBCH 00	90	< 0.01 (2)	5A-HR-05-6380
		0.104	BBCH 59–61	49	< 0.01 (2)	
Virginia	Coker	0.216	BBCH 00	246	< 0.01 (2)	SJ-HR-05-6381
		0.108	BBCH 75	51	< 0.01 (2)	
North Dakota (Northwood)	Morton	0.211	bare soil	90	< 0.01 (2)	NN-HR-05-6382
		0.104	BBCH 23	50	< 0.01 (2)	
South Dakota (Lesterville)	Jerry	0.209	BBCH 01	84	< 0.01 (2)	NF-HR-05-6383
		0.106	BBCH 32	54	< 0.01 (2)	
Wisconsin	Esker	0.216	pre-emergence	91	< 0.01 (2)	NI-HR-05-6384
		0.106	late boot -early head	49	< 0.01 (2)	
Minnesota	Drumlin	0.209	BBCH 00	80	< 0.01 (2)	NF-HR-05-6385
		0.107	BBCH 21	52	< 0.01 (2)	
Nebraska (York)	Jerry	0.213	BBCH 00	87	< 0.01 (2)	NB-HR-05-6386
		0.105	BBCH 31	49	< 0.01 (2)	
Iowa	Jerry	0.213	BBCH 00	96	< 0.01 (2)	NE-HR-05-6387
		0.106	BBCH 80	49	< 0.01 (2)	
Kansas (Sabetha)	Loyal	0.210	pre-emergence	83	< 0.01 (2)	ND-HR-05-6388
		0.107	1–3 tillers	50	< 0.01 (2)	
Michigan	Prairie	0.213	pre-emergence	88	< 0.01 (2)	NL-HR-05-6389
		0.104	5–6 leaves	50	< 0.01 (2)	
Illinois	VHS	0.209	BBCH 00	81	< 0.01 (2)	4A-HR-05-6390
		0.104	BBCH 26	52	< 0.01 (2)	
		0.317	BBCH 26	52	< 0.01 (2)	
		0.531	BBCH 26	52	< 0.01 (2)	
Texas	La-604	0.209	BBCH 00	180	< 0.01 (2)	SA-HR-05-6391
		0.106	BBCH 43	54	< 0.01 (2)	
North Dakota (New Rockford)	La-604	0.211	soil surface spray	92	< 0.01 (2)	NM-HR-05-6392
		0.104	BBCH 43	49	< 0.01 (2)	
South Dakota (Frederick)	Morton	0.211	BBCH 01	81	< 0.01 (2)	NF-HR-05-6393
		0.106	BBCH 21–31	51	< 0.01 (2)	
Nebraska (Grand Island)	Reeves	0.215	BBCH 00	90	< 0.01 (2)	NB-HR-05-6394
		0.105	BBCH 31	49	< 0.01 (2)	
Kansas (Larned)	Jerry	0.210	pre-emergence	83	< 0.01 (2)	NM-HR-05-6395
		0.107	1–2 tillers	50	< 0.01 (2)	

Rice

Two residue trials were conducted in rice in Korea in 2005 and eight in Japan in 2004 and 2006 using one or two applications into the water, post-transplanting. None of the trials were under GLP. The results in brown rice (husked rice) are shown in Table 84.

Table 84 Summary of residue data for mesotrione on rice (brown rice) after application using a granular formulation

Country, year	Rice variety	Application rate (g ai/ha)	Growth Stage	DAT (days)	Residue (mg/kg)	Trial
Korea 2005		0.180	5–15 days after transplanting	140	< 0.05 (3)	A14928B
		0.360	5–15 days after transplanting	140	< 0.05 (3)	

Country, year	Rice variety	Application rate (g ai/ha)	Growth Stage	DAT (days)	Residue (mg/kg)	Trial
Japan, 2004	Matsuribare	0.100	21 days after transplanting	91	< 0.002 (2)	A137723B
Japan, 2004	Hi-no-hikari	0.100	21 days after transplanting	89	< 0.002 (2)	A137723B
Japan 2006	Koshihikari	0.100	4.5 leaf period	76	< 0.002 (2)	A137723B
		0.100	growing period			
		0.100	4.5 leaf period	61	< 0.002 (2)	
		0.100	growing period			
Japan 2006	Hi-no-hikari	0.100	4.5 leaf period	45	< 0.002 (2)	A137723B
		0.100	before harvest			
		0.100	10 days after plant	75	< 0.002 (2)	
		0.100	75 days before harvest			
		0.100	10 days after plant	60	< 0.002 (2)	
		0.100	60 days before harvest			
		0.100	10 days after plant	45	< 0.002 (2)	
		0.100	45 days before harvest			
		0.100				
		0.100				

Sorghum

Twenty eight supervised residue trials were conducted on sorghum in the USA (T020419-04) during 2005, with one application made pre-emergence of the crop (SC formulation). The results on sorghum grain are shown in Table 85.

Table 85 Summary of residue data for mesotrione (SC formulation) in sorghum in the USA in 2005

State (location)	Sorghum variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
South Caroline	NK8416	0.225	Soil surface at planting	113	< 0.01 (2)	SJ-HR-05-6225
		0.227	Pre-planting incorporated	113	< 0.01 (2)	
		0.224 ^a	BBCH 17	86	< 0.01 (2)	
Louisiana	Pioneer 83G66	0.231	Soil surface at planting	120	< 0.01 (2)	SD-HR-05-6226
		0.228	Pre-planting incorporated	120	< 0.01 (2)	
		0.222 ^a	6–8 leaf	87	< 0.01 (2)	
Kansas	Dekalb DKS5400	0.228	Soil surface at planting	134	< 0.01 (2)	ND-HR-05-6227
		0.224 ^a	4–6 collar	111	< 0.01 (2)	
Nebraska (York)	NC + 6B50	0.225	Soil surface at planting	127	< 0.01 (2)	NB-HR-05-6228
		0.224 ^a	BBCH 16	94	< 0.01 (2)	
Missouri	Pioneer 8500	0.223	Soil surface at planting	127	< 0.01 (2)	ND-HR-05-6229
		0.221 ^a	BBCH 16	98	< 0.01 (2)	
South Dakota	Partner 251	0.224	Soil surface at planting	128	< 0.01 (2)	NF-HR-05-6230
		0.226 ^a	BBCH 18	101	< 0.01 (2)	
Texas (Wharton)	DK52	0.219	Soil surface at planting	102	< 0.01 (2)	SA-HR-05-6231
				108	< 0.01 (2)	
				116	< 0.01 (2)	
		0.225 ^a	BBCH 23–32	63	< 0.01 (2)	
Oklahoma	SG95207	0.229	Pre-planting incorporated	128	< 0.01 (2)	SC-HR-05-6232
		0.226 ^a	4–5 leaf	90	< 0.01 (2)	
Nebraska (Grand Island)	NC + 6B50	0.222	Soil surface at planting	132	< 0.01 (2)	NB-HR-05-6233
		0.225	Pre-planting incorporated	132	< 0.01 (2)	
		0.228 ^a	BBCH 16	99	< 0.01 (2)	
Texas (Levelland)	F222E	0.228	Pre-planting incorporated	128	< 0.01 (2)	SC-HR-05-6234
		0.223 ^a	BBCH 16	103	< 0.01 (2)	
Colorado	DG-720B	0.228	Pre-planting incorporated	145	< 0.01 (2)	NM-HR-05-6235
		0.231 ^a	vegetative	85	< 0.01 (2)	
New Mexico	7117	0.229	Soil surface at planting	113	< 0.01 (2)	NB-HR-05-6236
		0.226	Pre-planting incorporated	113	< 0.01 (2)	
		0.226 ^a	8 leaf	78	< 0.01 (2)	

^a Formulation with non-ionic surfactant

Sugarcane

Twenty six supervised residue trials were conducted on sugarcane in the USA during 2005 and four trials were conducted in South Africa during the growing period 1998/99. The trial data are summarised in Table 86.

Table 86 Summary of residue data for mesotrione in sugarcane (SC) formulation

Country (Region)	Sugarcane variety	Application Rate (g ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Report; trial
USA (Florida, South Bay)	CP-2143	0.27 (SS) + 0.11 (POT)	pre-emergence + 114 days to harvest	0	0.03, 0.02	T020420-04/VN-HR-05- 6240
				30	< 0.01 (2)	
				62	< 0.01 (2)	
				118	≤ 0.01 (2)	
				125	< 0.01 (2)	
		0.27 (SS) + 0.11 (PD)	pre-emergence + 100 days to harvest	0	0.02, < 0.01	
				30	< 0.01 (2)	
				61	< 0.01 (2)	
				104	< 0.01 (2)	
				111	≤ 0.01 (2)	
		0.10 (POT) + 0.11 (PD)	114 days + 100 days to harvest	0	0.02, 0.07	
				30	< 0.01 (2)	
				61	< 0.01 (2)	
				104	< 0.01 (2)	
				111	≤ 0.01 (2)	
USA (Florida, South Bay)	CP-2086	0.27 (SS) + 0.10 (POT)	pre-emergence+ 114 days to harvest	118	≤ 0.01 (2)	T020420-04/VN-HR-05- 6241
		0.27 (SS) + 0.10 (PD)	pre-emergence + 100 days to harvest	104	≤ 0.01 (2)	
		0.10 (POT) + 0.106 (PD)	114 days + 100 days to harvest	104	≤ 0.01 (2)	
		0.52 (POT) + 0.52 (PD) ¹	114 days + 100 days to harvest	104	< 0.01 (2)	
USA (Florida, South Bay)	CP-2086	0.28 (SS) + 0.10 (POT)	pre-emergence + 114 days to harvest	118	≤ 0.01 (2)	T020420-04/VN-HR-05- 6242
		0.27 (SS) + 0.11 (PD)	pre-emergence + 100 days to harvest	104	≤ 0.01 (2)	
		0.11 (POT) + 0.10 (PD)	114 days + 100 days to harvest	104	≤ 0.01 (2)	
USA (Louisiana, Bunkie)	LCP 85384	0.25 (SS) + 0.10 (POT)	BBCH 00 + pre-internode	0	3.72, 5.75	T020420-04/SD-HR- 2005
				30	< 0.01 (2)	
				60	< 0.01 (2)	
				114	≤ 0.01 (2)	
				121	< 0.01 (2)	
		0.26 (SS) + 0.10 (PD)	BBCH 00 + pre-internode	0	0.70, 0.31	
				30	< 0.01 (2)	
				60	< 0.01 (2)	
				100	< 0.01 (2)	
		0.11 (POT) + 0.10 (PD)	pre-internode + pre-internode	0	0.07, 0.20	
				30	< 0.01 (2)	
				60	< 0.01 (2)	
USA (Louisiana, Cheney Ville)	LCP85384 Kleentek	0.29 (SS) + 0.11 (POT)	BBCH 00 + pre-internode	113	≤ 0.01 (2)	T020420-04/SD-HR-05- 6244
				99	≤ 0.01 (2)	
		0.28(SS) + 0.10 (PD)	BBCH 00 + pre-internode	99	≤ 0.01 (2)	
				99	≤ 0.01 (2)	

Country (Region)	Sugarcane variety	Application Rate (g ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Report; trial
		0.56 (POT) + 0.54 (PD)	pre-internode + pre-internode	99	< 0.01 (2)	
USA (Louisiana, Washington)	384	0.27(SS) + 0.11 (POT)	BBCH 00 + 75	114	≤ 0.01 (2)	T020420-04/SD-HR-05- 6245
		0.27 (SS) + 0.11 (PD)	BBCH 00 + 43	100	≤ 0.01 (2)	
		0.11 (POT) + 0.11 (PD)	BBCH 00 + 43	100	≤ 0.01 (2)	
USA (Texas)	3388	0.28 (SS) + 0.11 (POT)	BBCH 08+ 37	114	≤ 0.01 (2)	T020420-04/SA-HR-05- 6246
		0.28 (SS) + 0.11 (PD)	BBCH 08 + 37	100	≤ 0.01 (2)	
		0.11 (POT) + 0.11 (PD)	BBCH 37 + 38	100	≤ 0.01 (2)	
USA (Hawaii)	65-7052	0.27 (SS) + 0.11 (POT)	prior emergence + pre-crop closure	114	≤ 0.01 (2)	T020420-04/WD-HR-05- 6247
		0.27(SS) + 0.10 (PD)	prior emergence + prior to layby	100	≤ 0.01 (2)	
		0.11 (POT) + 0.10 (PD)	pre-canopy close + prior to layby	100	< 0.01 (2)	
South Africa	N27	0.25	growth high 50–70 cm	182	< 0.01	RJ3076B/ZA18-99-H310
South Africa	N14	0.25	growth high 60–75 cm	182 273	< 0.01 < 0.01	RJ3076B/ZA18-99-H311
South Africa	N19	0.25	high 50–60 cm	181	< 0.01	RJ3076B/ZA22-99-H410
		0.50	growth high 50–60 cm	181	< 0.01	

SS= soil surface spray

POT= post-emergence over-the-top

PD= post-emergence application directed to the base of the sugarcane

Linseed

Seventeen supervised residue trials were conducted in linseed in the USA during 2004 with one pre-emergence application of a suspension concentrate (SC) formulation (Report T010290-04 2004). The results are shown in Table 87.

Table 87 Summary of residue data for the use of mesotrione on linseed in the USA in 2004 using SC formulation

State (location)	Linseed variety	Application Rate (g ai/ha)	Growth Stage	PHI (days)	Crop Part	Residues (mg/kg)	Trial number
North Dakota (Northwood)	Rehab	0.11	at planting	144	seed	< 0.01 (2)	NN-HR-04-5650
		0.21	at planting	144	seed	< 0.01 (2)	
		0.11	multiple stems and no buds	103	seed meal	< 0.01 (2)	
		0.32	multiple stems and no buds	103	seed	< 0.01 (2)	
		0.53	multiple stems and no buds	103	seed meal	< 0.01 (2)	
Minnesota	York	0.10	pre-emergence	170	Seed	< 0.01 (2)	NF-HR-04-5651
		0.21	pre-emergence	170	Seed	< 0.01 (2)	
		0.10	25 cm high	130	seed	< 0.01 (2)	
North Dakota (New Rockford)	Rehab	0.11	at planting	136	seed	< 0.01 (2)	NN-HR-04-5652
		0.21	at planting	136	seed	< 0.01 (2)	
		0.10	ca. 25 cm high	104	seed	< 0.01 (2)	
Montana	Neché	0.10	at planting	89	seed	< 0.01 (2)	NN-HR-04-5653
		0.21	at planting	89	seed	< 0.01 (2)	
		0.11	stem elongation	46	seed	< 0.01 (2)	

State (location)	Linseed variety	Application Rate (g ai/ha)	Growth Stage	PHI (days)	Crop Part	Residues (mg/kg)	Trial number
South Dakota	Webster	0.10	BBCH 01 (at planting)	133 140 147	seed	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-04-5654
		0.21	BBCH 01 (at planting)	133 140 147	seed	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.10	BBCH 39	95 102 109	seed	< 0.01 (2) < 0.01 (2) < 0.01 (2)	

Animal Feed

Cereal trials from the studies reported previously have included the analysis of feed samples. The results are shown in Tables 88 to 92.

Table 88 Summary of residue data for the use of mesotrione on feed from corn (sweet and field)

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
Canada (Ontario) 1996	Pioneer 3902	SC	0.300	Pre-emergence	102	silage	< 0.01	RR 97-043B 94-CN-96-201
		SC	0.60	Pre-emergence	102	silage	< 0.01	
		SC	0.200	5–6 Leaf	87	silage	< 0.01	
		SC	0.400	5–6 Leaf	87	silage	< 0.01	
		SC	0.300 0.200	Pre-emergence 5–6 leaf	87	silage	< 0.01	
		SC	0.600 0.400	Pre-emergence 5–6 leaf	87	silage	< 0.01	
Canada (Ontario) 1996	Ciba seeds G-4064	SC	0.300	Pre-emergence	101	silage	< 0.01	RR 97-043B 94-CN-96-202
		SC	0.600	Pre-emergence	101	silage	< 0.01	
		SC	0.200	4–5 Leaf	90	silage	< 0.01	
		SC	0.400	4–5 Leaf	90	silage	< 0.01	
		SC	0.300 0.200	Pre-emergence 4–5 leaf	90	silage	< 0.01	
		SC	0.600 0.400	Pre-emergence 4–5 leaf	90	silage	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	120 155	silage grain	< 0.01 < 0.01	RR 98-035B 94-CN-97-901
		SC	0.600	Pre-emergence	120	silage	< 0.01	
		SC	0.200	24	91	silage	< 0.01	
		SC	0.400	24	91	silage	< 0.01	
		SC	0.300 0.200	Pre-emergence 24	91	silage	< 0.01	
		SC	0.600 0.400	Pre-emergence 24	91	silage	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	112	silage	< 0.01	RR 98-035B 94-CN-97-902
		SC	0.600	Pre-emergence	112	silage	< 0.01	

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
		SC	0.200	23	93	silage	< 0.01	
France (South)	620 Spirit	SC	0.400	23	93	silage	< 0.01	03-7049 2003
		SC	0.300 0.200	Pre- emergence 23	93	silage	< 0.01	
		SC	0.600 0.400	Pre- emergence 23	93	silage	< 0.01	
		SC	0.102	BBCH 19	38	Cob	< 0.01	
		SC	0.153	BBCH 12	38 61 62	cob cob cob	< 0.01 < 0.01 < 0.01	
France (South)	620 Spirit		0.98	BBCH 19	38	Cob	< 0.01	03-7050 2003
			0.143	BBCH 12	62	cob	< 0.01	
France (South)	620 Spirit		0.153	BBCH 12	61 74	cob cob	< 0.01 < 0.01	04-7012 2004
			0.104	BBCH 59	39	Cob	< 0.01	
		SC	0.154	BBCH 12	61 74	cob cob	< 0.01 < 0.01	
France (South)	LG35.05	WG	0.148	BBCH 15– 16	39	Cob	< 0.01	T000921-09- REG 2009
					27	whole plant	< 0.01	
					35	whole plant	< 0.01	
					43	whole cob	< 0.01	
					43	remaining plant	< 0.01	
					61	whole plant	< 0.01	
					68	whole cob	< 0.01	
					68	remaining plant	< 0.01	
					99	whole plant	< 0.01	
					99	whole cob	< 0.01	
Germany	Nescio	WG	0.138	BBCH 16– 18	99	remaining plant	< 0.01	T000920-09- REG 2009
					127	whole cob	< 0.01	
					127	remaining plant	< 0.01	
					34	whole plant	< 0.01	
					47	whole plant	< 0.01	
					47	whole cobs	< 0.01	
					47	remaining plant	< 0.01	
					82	cobs	< 0.01	
					82	remaining plant	< 0.01	
					66	whole plant	< 0.01	
Spain	Castellano	WG	0.154	BBCH 15	91	cobs	< 0.01	T000921-09- REG 2009
					91	remaining plant	< 0.01	
					110	whole plant	< 0.01	
					143	cobs	< 0.01	
					143	remaining plant	< 0.01	
					7	whole plant	< 0.01	
					14	whole plant	< 0.01	
					42	cobs	< 0.01	
					42	remaining plant	< 0.01	
					63	whole plant	< 0.01	
Spain	Castellano	WG	0.154	BBCH 15	71	cobs	< 0.01	T000921-09- REG 2009
					71	remaining plant	< 0.01	
					82	cobs	< 0.01	
					82	remaining plant	< 0.01	
					82	remaining plant	< 0.01	

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
United Kingdom	Ohio	WG	0.142	BBCH 16– 17	20	whole plant	< 0.01	T000920-09- REG 2009
					35	whole plant	< 0.01	
					41	whole cobs	< 0.01	
					41	remaining plant	< 0.01	
					53	whole cobs	< 0.01	
					53	remaining plant	< 0.01	
					60	whole plant	< 0.01	
					87	whole plant	< 0.01	
					98	cobs	< 0.01	
					98	remaining plant	< 0.01	
					112	cobs	< 0.01	
					112	remaining plant	< 0.01	
USA (Wisconsin) 2001	NK 199	SC	0.302 0.177	pre- emergence Vt (tasseling)	14	forage with ears	< 0.01 (2)	487-01; NI- HR001-01
					30	forage without stover	< 0.01 (2)	
					56		< 0.01 (2)	
USA (Washington) 2001	Jubilee	SC	0.303 0.179		14	forage w/ ears	0.02, 0.07	487-01; WF- HR003-01
					28	forage w/o ears	< 0.01 (2)	
					71	stover	< 0.01 (2)	
USA (California) 2001	Silver Queen	SC	0.300 0.182	BBCH 01 BBCH 59	0	forage w/ ears	4.29, 4.96	487-01; W2-HR102-01
					7	forage w/ ears	0.82, 1.28	
					14	forage w/ ears	0.73, 0.88	
					23	forage w/o ears	0.45, 0.34	
					30	forage w/o ears	0.40, 0.38	
					37	forage w/o ears	0.23, 0.33	
					58	stover	1.2, 0.90 (1.1)	
USA (N. Carolina) 2001	G90 F1	SC	0.308 0.182	BBCH 55	14	forage w/ ears	< 0.01, 0.02	487-01; SJ- HR003-01
					26	forage w/o ears	< 0.01 (2)	
					54	stover	< 0.01 (2)	
USA (Ohio) 2001	Bodacious	SC	0.314 0.190		14	forage w/ ears	< 0.01 (2)	487-01; NK- HR001-01
					30	forage w/o ears	< 0.01 (2)	
					52	stover	< 0.01 (2)	
USA (New York) 2001	GH-2783	SC	0.305 0.182	at planting + 8–9 leaves	14	forage w/ ears	< 0.01 (2)	487-01; EE- HR003-01
					30	forage w/o ears	< 0.01 (2)	
					67	stover	< 0.01 (2)	
USA (Idaho) 2001	Sugar Buns		0.303 0.179	post planting + pollen shed	14	forage w/ ears	0.02, 0.01	487-01; WG- HR005-01
					30	forage w/o ears	0.05, < 0.01	
					61	stover	< 0.01 (2)	
USA (Illinois) 2001	Kandy King	SC	0.301 0.186	BBCH 00 + BBCH 34	14	forage w/ ears	< 0.01 (2)	487-01; N4- HR003-01
					31	forage w/o ears	< 0.01 (2)	
					60	stover	< 0.01 (2)	
USA (Pennsylvania) 2001	Argent	SC	0.320 0.186	BBCH 00 + early tassel	14	forage w/ ears	< 0.01 (2)	487-01; EC- HR002-01
					30	forage w/o ears	< 0.01 (2)	
					70	stover	< 0.01 (2)	
USA (Florida) 2001	Silver Queen	SC	0.313 0.177	Just planted + visible tassel	14	forage w/ ears	< 0.01 (2)	487-01; VB- HR101-01
					28	forage w/o ears	< 0.01 (2)	
					62	stover	< 0.01 (2)	
USA (Michigan) 2001	Excellency	SC	0.306 0.180	pre- emergence Vt	0	forage w/ ears	3.09, 3.96	487-01; ED- HR006-01
					7	forage w/ ears	0.02, 0.01	
					14	forage w/ ears	< 0.01 (2)	
					23	forage w/o ears	< 0.01 (2)	
					30	forage w/o ears	< 0.01 (2)	
					36	forage w/o ears	< 0.01 (2)	
					70	stover	< 0.01 (2)	
USA (Maine) 2001	Maple Sweet	SC	0.304 0.189	0 + tassel initiation	14	forage w/ ears	< 0.01 (2)	487-01; NF- HR002-01
					30	forage w/o ears	< 0.01 (2)	
					55	stover	< 0.01 (2)	
USA N. Carolina)	Rogers WH0809	SC	0.107 0.106	BBCH 36 + BBCH 37	32	forage w/o ears	< 0.01 (2)	T001589-08; E10NC081871
					53	stover	< 0.01 (2)	

Country (Region) 2008	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
		WG	0.104 0.104	BBCH 36 + BBCH 37	32 53	forage w/o ears stover	< 0.01 (2) < 0.01 (2)	
USA (N. Dakota 2008)	Peaches & Cream	SC	0.107 0.105	BBCH 16 + BBCH 55	28 66	forage w/o ears stover	< 0.01 (2) < 0.01 (2)	T001589-08; C13ND081872
		WG	0.107 0.106	BBCH 16 + BBCH 55	28 66	forage w/o ears stover	< 0.01 (2) < 0.01 (2)	
USA (California) 2008	Sweetie 82	SC	0.103 0.107	BBCH 15 + BBCH 17	45 69	forage w/o ears stover	0.12 (0.07, 0.17) 0.06, 0.09	T001589-08; W30CA081873
		WG	0.105 0.105	BBCH 15 + BBCH 17	45 69	forage stover	(0.06) 0.07, 0.06 0.09, 0.07	
USA (Iowa, Sheffield) 1995	ICI 8543	SC	0.336 0.224	Pre-plant vegetative	82 109	forage stover	< 0.01 (3) < 0.01 (2)	RR 96-018B 63-IA-95-805
USA (Illinois, Brimfield) 1995	Hoblit 428	SC-	0.336 0.224	pre- emergence	85 95	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 60-IL-95-806
USA (Indiana, Lafayette) 1995	Pioneer 3394	SC	0.336 0.224	pre- emergence 7–9 leaves	76 96 96	forage grain stover	< 0.01 (2) < 0.01 (2) < 0.01 (2)	RR 96-018B 67-IN-95-807
USA (Michigan) 1995	NK 4640	SC	0.336 0.224	pre-bloom	85 114	forage stover	< 0.01 (2) < 0.01	RR 96-018B 24-MI-95-808
USA (Nebraska) 1995	Ottolie RO 2455	SC	0.336 0.224	Pre-plant V6	62 88	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 68-NE-95-809
USA (Iowa, Albia) 1995	ICI 8532	SC	0.336 0.224	Pre-plant vegetative	74 102	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 63-IA-95-810
USA (Illinois, Towanda) 1995	Ainsworth 640	SC	0.336 0.224	Pre-plant post emergence	76 87	forage stover	< 0.01 (3) < 0.01 (2)	RR 96-018B 60-IL-95-811
USA (Indiana, Rochester) 1995	Pioneer 3394	SC	0.336 0.224	Early pre- plant 5–7 leaves	92 96	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 67-IN-95-812
USA (Nebraska, Waverly) 1995	Producers PH785	SC	0.336 0.224	Pre-plant V5–V6	71 96	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 68-NE-95-814
USA (Kansas, La Cygne) 1995	CIBA 4575	SC	0.336 224	early pre- plant post	46 68	forage stover	< 0.01 (3) < 0.01 (2)	RR 96-018B 37-KS-95-825
USA (Pennsylvania, Ephrata) 1995	CI 8541	SC	0.336 224	pre- emergence vegetative	76 98	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 70-PA-95-815
		SC	0.336 0.224	pre- emergence vegetative	76 98	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Minnesota, St. Peter) 1995	Cenex 424	SC	0.336 0.224	pre- emergence vegetative	76 92	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 36-MN-95-816
		SC	0.336 0.224	pre- emergence vegetative	76 92	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Ohio, Urbana) 1995	Vigoro V1122	SC	0.336 0.224	pre- emergence	85 113	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 24-OH-95-817
		SC	0.336 0.224	pre- emergence	85 113	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Wisconsin, 1995)	Cenex LOL 357	SC	0.336 0.224	pre- emergence	72 104	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 79-WI-95-818

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
Baraboo) 1995		SC	0.336 0.224	pre- emergence	72 104	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Texas) 1995	G4673B (D&PL Co.)	SC	0.336 0.224	pre- emergence pre-tassel	56 92	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 25-TX-95-819
		SC	0.336 0.224	pre- emergence pre-tassel	56 92	forage stover	< 0.01 (2) < 0.01 (2)	
USA (north Carolina) 1995	Field corn (Pioneer 3165)	SC	0.336 0.224	pre-plant rapidly growing	68 95	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 01-NC-95-820
		SC	0.336 0.224	pre-plant rapidly growing	62 68 75 82 88 95 101 108	forage forage forage stover stover stover stover	< 0.01 < 0.01 (2) < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	
USA (Iowa, Boone) 1995	ICI 8543	SC	0.336 0.224	Early pre- plant vegetative	86 112	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 63-IA-95-821
		SC	0.336 0.224	Early pre- plant vegetative	86 112	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Illinois, Champagne) 1995	Pioneer 3394	SC	0.336 0.224	pre-plant post emergence	74 107	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 04-IL-95-822
		SC	0.336 0.224	pre-plant post emergence	60 74 81 88 100 107 113 121	forage forage forage stover stover stover stover	< 0.01 < 0.01 (2) < 0.01 < 0.01 < 0.01 < 0.01 (2) < 0.01 < 0.01	
USA (Indiana, N. Richmond) 1995	Pioneer 3394	SC	0.336 20.24	pre-plant 6–8 leaf	64 86	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 67-IN-95-823
		SC	0.336 0.224	pre-plant 6–8 leaf	64 86	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Nebraska, Crete) 1995	ICI 8541	SC	0.336 0.224	pre-plant V6-V7	62 89	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 68-NE-95-824
		SC	0.336 0.224	pre-plant V6-V7	62 89	forage stover	< 0.01 (2) < 0.01 (2)	
USA Iowa) 2001	Pioneer 34B23)	SC	0.268 0.225	BBCH 00 BBCH 18– 19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	1847-01 NE-HR-003-01
		SE SC	0.267 0.233	BBCH 00 BBCH 18/19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	
		SC	0.274 0.234	BBCH 13 BBCH 18 19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	
		SE SC	0.267 0.223	BBCH 13 BBCH 18/19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	
USA (North Carolina) 2001	ICI 8543)	SC	0.269 0.226	BBCH 00 75 cm high	45 98	forage stover	< 0.01 (2) < 0.01 (2)	1847-01 SJ-HR-013-01
		SE SC	0.269 0.227	BBCH 00 75 cm high	45 98	forage stover	< 0.01 (2) < 0.01 (2)	

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
USA (Illinois) 2001	Pioneer 34B24	SC	0.271 0.227	BBCH 12/13 75 cm high	45 98	forage stover	< 0.01 (2) < 0.01 (2)	1847-01 N4-HR-005-01
		SE	0.270	BBCH 12/13	45	forage	< 0.01 (2)	
		SC	0.227	75 cm high	98	stover	< 0.01 (2)	
		SE	0.270	BBCH 12/13 75 cm high	45	forage	< 0.01 (2)	
USA (Nebraska) 2001	ICI 8543	SC	0.285 0.253	BBCH 00 BBCH 36	45 112	forage stover	< 0.01 (2) < 0.01 (3)	1847-01 NB-HR-004-01
		SE	0.278	BBCH 00	45	forage	< 0.01 (2)	
		SC	0.236	BBCH 36	112	stover	< 0.01 (2)	
		SC	0.286 0.244	BBCH 12 BBCH 36	45 112	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Nebraska) 2001	ICI 8543	SE	0.268	BBCH 12	45	forage	< 0.01 (2)	1847-01 NB-HR-004-01
		SC	0.237	BBCH 36	112	stover	< 0.01 (2)	
		SC	0.270 0.227	BBCH 00 BBCH 19	41 103	forage stover	< 0.01 (2) < 0.01 (2)	
		SE	0.273	BBCH 00	41	forage	< 0.01 (2)	
USA (Nebraska) 2001	ICI 8543	SC	0.227	BBCH 19	103	stover	< 0.01 (2)	1847-01 NB-HR-004-01
		SC	0.266 0.226	BBCH 12/13 BBCH 19	41 103	forage stover	< 0.01 (2) < 0.01 (2)	
		SE	0.268	BBCH 12/13	41	forage	< 0.01 (2)	
		SC	0.226	BBCH 19	103	stover	< 0.01 (2)	

Table 89 Summary of residue data for mesotrione on millet (feed) using SC formulation in the USA in 2004 (Report T010289-04)

State	Millet variety	Application Rate (g ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
Illinois	Max Perl	0.109	at planting	31 31	forage hay	< 0.01 (2) < 0.01 (2)	4A-HR-04- 5640
		0.214	at planting	31 31	forage hay	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	
		0.108	BBCH 32	31 31	forage hay	< 0.01 (2) < 0.01 (2)	
Nebraska	Huntsman	0.103	at planting	51 51 84	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-04- 5641
		0.210	at planting	51 51 84	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
		0.103	BBCH 12	28 28 61	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
South Dakota	Rise	0.104	at planting	34 34 95	forage hay straw	< 0.01 (2) 0.01, < 0.01 < 0.01 (2)	4A-HR-04- 5642
		0.208	at planting	34 34 95	forage hay straw	<u>0.01</u> , < 0.01 <u>0.01</u> (2) <u>< 0.01</u> (2)	
		0.105	BBCH 21	30 30 69	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Colorado (Ault)	Early Bird	0.107	at planting	70 70 132	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-04- 5643
		0.212	at planting	70 70 132	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	

State	Millet variety	Application Rate (g ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
		0.107	vegetative	30 30 92	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Colorado (Ault)	Early Bird	0.110	at planting	63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-04-5644
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
				63 63 70 70 77 77 104 111 120	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.108	vegetative	23 23 30 30 37 37 64 71 80	forage hay forage hay forage hay straw straw straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	

Table 90 Summary of residue data for the use of mesotrione on oats (feed) (SC formulation) in 2005 in the USA (Report T004407-05)

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT	Crop Part	Residues (mg/kg)	Trial number
New York	Armor	0.214	BBCH 00	70 70 90	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	5A-HR-05-6380
		0.104	BBCH 59–61	29 29 49	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.104	BBCH 59–61	29 29 49	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Virginia	Coker	0.216	BBCH 00	225 225 246	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	SJ-HR-05-6381
		0.108	BBCH 75	30 30 51	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
North Dakota (Northwood)	Morton	0.211	bare soil application	73 73 90	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NN-HR-05-6382
		0.104	BBCH 23	33 33 50	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
South Dakota (Lesterville)	Jerry	0.209	BBCH 01	60 60 84	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-05-6383
		0.106	BBCH 32	30 30 54	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT	Crop Part	Residues (mg/kg)	Trial number
Wisconsin	Oat (Esker)	0.216	pre-emergence	70 70 91	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NI-HR-05-6384
		0.106	late boot to early head	28 28 49	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Minnesota	Oat (Drumlin)	0.209	BBCH 00	58 58 80	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-05-6385
		0.107	BBCH 21	30 30 52	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Nebraska (York)	Oat (Jerry)	0.213	BBCH 00	54 54 63 63 68 68 75 75 87	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	NB-HR-05-6386
		0.105	BBCH 31	16 16 25 25 30 30 37 37 49	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Iowa	Oat (Jerry)	0.213	BBCH 00	75 75 96	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NE-HR-05-6387
		0.106	BBCH 80	28 28 49	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Kansas (Sabetha)	Oat (Loyal)	0.210	pre-emergence	63 63 83	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	ND-HR-05-6388
		0.107	tillering (1–3 tillers)	30 30 50	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Michigan	Oat (Prairie)	0.213	pre-emergence	55 55 61 61 68 68 75 75 88	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	NL-HR-05-6389
		0.104	5–6 leaves	17 17 23 23 30 30 37 37 50	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Illinois	Oat (VHS)	0.209	BBCH 00	62 62 81	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-05-6390

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT	Crop Part	Residues (mg/kg)	Trial number
		0.104	BBCH 26	33 33 52	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
		0.317	BBCH 26	33 33 52	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.531	BBCH 26	33 33 52	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Texas	Oat (La-604)	0.209	BBCH 00	156 156 180	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	SA-HR-05-6391
		0.106	BBCH 43	30 30 54	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
North Dakota (New Rockford)	Oat (La-604)	0.211	soil surface spray	73 73 92	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NM-HR-05-6392
		0.104	BBCH 43	30 30 49	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
South Dakota (Frederick)	Oat (Morton)	0.211	BBCH 01	60 60 81	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-05-6393
		0.106	BBCH 21–31	30 30 51	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Nebraska (Grand Island)	Oat (Reeves)	0.215	BBCH 00	70 70 90	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NB-HR-05-6394
		0.105	BBCH 31	29 29 49	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
Kansas (Larned)	Oat (Jerry)	0.210	pre-emergence	63 63 83	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NM-HR-05-6395
		0.107	tillering (1–2 tillers)	30 30 50	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	

Table 91 Summary of residue data for mesotrione on rice (feed) after application using a granular formulation

Country year	Rice variety	Application rate (kg ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
Korea 2005		0.180	5–15 days after transplanting	140	Straw	< 0.1 (3)	A14928B
		0.360	5–15 days after transplanting	140	Straw	< 0.1 (3)	
Japan 2004	Matsuribare	0.100	21 days after transplanting	63 91	Forage Straw	< 0.002 (2) < 0.01 (2)	A13723B
Japan 2004	Hi-no-hikari	0.100	21 days after transplanting	77 89	Forage Straw	< 0.002 (2) < 0.01 (2)	A13723B
Japan 2006	Koshihikari	0.100	4.5 leaf period growing period	76	Straw	< 0.01 (2)	A13723B
		0.100	4.5 leaf period growing period	61	Straw	< 0.01 (2)	
		0.100	4.5 leaf period growing period				
		0.100	4.5 leaf period before harvest	45	Straw	< 0.01 (2)	

Country year	Rice variety	Application rate (kg ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
Japan 2006	Hi-no-hikari	0.100	10 days after plant	75	Straw	< 0.01 (2)	A13723B
		0.100	75 days before harvest				
		0.100	10 days after plant	60	Straw	< 0.01 (2)	
		0.100	60 days before harvest				
Japan 2007	Koshihikari	0.100	10 days after plant	45	Straw	< 0.01 (2)	A13723B
		0.100	45 days before harvest				
		0.100	growing period	75	Forage	< 0.002 (2)	
		0.100	21 cm high				
Japan	Hi-no-hikari	0.100	growing period	60	Forage	< 0.002 (2)	A13723B
		0.100	growing period				
		0.100	growing period	45	Forage	< 0.002 (2)	
		0.100	before harvest				
Japan	Hi-no-hikari	0.100	24 days after plant	53	Forage	< 0.002 (2)	A13723B
		0.100	24 days after plant	46	Forage	< 0.002 (2)	
		0.100	35 days before harvest				
		0.100	24 days after plant	34	Forage	< 0.002 (2)	
		0.100	formation of young panicle				

Table 92 Summary of residue data for mesotrione in sorghum feed in USA in 2005 (SC formulation) (Report T020419-04)

State (location)	Sorghum variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Crop Part	Residues (mg/kg)	Trial
South Caroline	NK8416	0.225	pre-emergence	57 113	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	SJ-HR-05-6225
		0.227	before planting	57 113	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	
		0.224	BBCH 17	30 86	forage stover	< 0.01 (2) < 0.01 (2)	
Louisiana	Pioneer 83G66	0.231	pre-emergence	65 120	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	SD-HR-05-6226
		0.228	pre-plant	65 120	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	
		0.222	6-8 leaf	32 87	forage stover	< 0.01 (2) < 0.01 (2)	
Kansas	Dekalb DKS5400	0.228	pre-emergence	54 134	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	ND-HR-05-6227
		0.224	4-6 collar	31 111 111	forage stover AGF	< 0.01 (2) < 0.01 (2) < 0.01	
Nebraska (York)	NC + 6B50	0.225	pre-emergence	33 43 49 63 127	forage forage forage forage stover	< 0.01 (2) < 0.01 (2) < 0.01 (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	NB-HR-05-6228
		0.224	BBCH 16	0 10 16 30 94	forage forage forage forage stover	14.4, 11.8 < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Missouri	Pioneer 8500	0.223	pre-emergence	57 127	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	ND-HR-05-6229
		0.221	BBCH 16	28 98	forage stover	< 0.01 (2) < 0.01 (2)	
South Dakota	Partner 251	0.224	BBCH 00	57 128	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	NF-HR-05-6230
		0.226	BBCH 18	30 101	forage stover	< 0.01 (2) < 0.01 (2)	

State (location)	Sorghum variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Crop Part	Residues (mg/kg)	Trial
Texas (Wharton)	DK52	0.219	BBCH 00	39 49 55 70 102 108 116	forage forage forage forage stover stover stover	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	SA-HR-05-6231
		0.225	BBCH 23–32	0 10 16 31 63 69 77	forage forage forage forage stover stover stover	15.0, 15.1 < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Oklahoma	SG95207	0.229	pre-planting	71 128	forage stover	< 0.01 (2) < 0.01 (2)	SC-HR-05-6232
		0.226	4–5 leaf	33 90	forage stover	< 0.01 (2) < 0.01 (2)	
Nebraska (Grand Island)	NC + 6B50	0.222	pre-emergence	63 132	forage stover	< 0.01 (2) < 0.01 (2)	NB-HR-05-6233
		0.225	BBCH 00	63 132	forage stover	< 0.01 (2) < 0.01 (2)	
		0.228	BBCH 16	30 99	forage stover	< 0.01 (2) < 0.01 (2)	
Texas (Levelland)	F222E	0.228	pre-planting	55 128	forage stover	< 0.01 (2) < 0.01 (2)	SC-HR-05-6234
		0.223	BBCH 16	30 103 103	forage stover AGF	< 0.01 (2) < 0.01 (2) < 0.01	
Colorado	DG-720B	0.228	at planting	90 145	forage stover	< 0.01 (2) < 0.01 (2)	NM-HR-05-6235
		0.231	vegetative	30 85	forage stover	< 0.01 (2) < 0.01 (2)	
New Mexico	7117	0.229	pre-emergence	64 113	forage stover	< 0.01 (2) < 0.01 (2)	NB-HR-05-6236
		0.226	pre-plant	64 113	forage stover	< 0.01 (2) < 0.01 (2)	
		0.226	8 leaf	29 78	forage stover	0.02, 0.01 < 0.01 (2)	

Processing studies

A residue trial on soya beans conducted in the USA with one pre-emergence application at a 1.1 kg ai/ha (Table 77; report T005595-06). Soya beans harvested at 113 DAT were processed into meal, hulls and refined oil simulating commercial practices, and samples analysed using method RAM 366/01. Residues of mesotrione were < 0.01 mg/kg in the soy sample, meal, hulls and refined oil, and no processing factors were derived

Two residue trials on mesotrione-tolerant HT soya beans were conducted in the USA using pre- plus post-emergence applications at 1.1 and 0.6 kg ai/ha, respectively (Table 93, Report T000908-07). Soya bean samples were harvested at 76 or 102 DAT, processed according to commercial practices, and samples analysed using method RAM 366/01. The results are show in in Table 93.

Table 93 Processing factors for mesotrione residues in mesotrione-tolerant soya beans

Mesotrione	Soya bean	Meal	Hulls	Crude oil	Refined oil	AGF	Flour	Soya milk	Tofu	Soya sauce	Miso
Residue (mg/kg) ^a	0.04	0.01	0.02	< 0.01	< 0.01	0.02	0.07	< 0.01	< 0.01	< 0.01	< 0.01

Processing factor		0.25	0.5	< 0.25	< 0.25	0.5	1.8	< 0.25	< 0.25	< 0.25	< 0.25
Residue (mg/kg) ^b	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	0.02	< 0.01	< 0.01	< 0.01	< 0.01
Processing factor		–	–	–	< 0.01	–	> 2	–	–	–	–

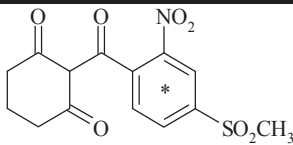
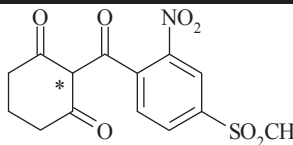
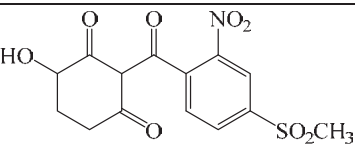
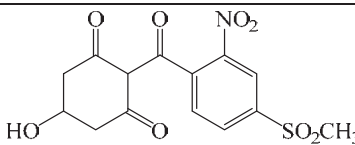
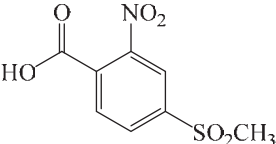
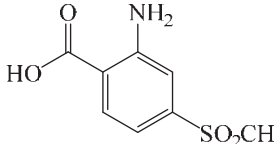
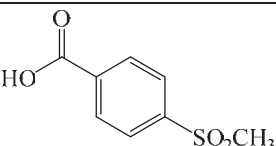
^a Trial C30-9659, 79 DAT^b Trial C18-9656, 102 DAT

RESIDUES IN ANIMAL COMMODITIES

No studies were provided

APPRAISAL

Mesotrione is a systemic pre-emergence and post-emergence herbicide for the selective contact and residual control of broadleaf weeds. The compound was scheduled for evaluation by 2014 JMPR as a new compound at the Forty-fifth Session of the CCPR (2013). Metabolism studies on animal and plants, confined rotational crops and environmental fate studies, analytical methods and residue trials on berries, okra, sweet corn, soybean, asparagus, rhubarb maize, millet, oat, rice, sorghum, sugarcane and linseed were submitted for evaluation. The structure of mesotrione and the main metabolites found in livestock, plant tissues and soil are shown below

 <p>[Phenyl-U-¹⁴C] Mesotrione</p>	 <p>[Cyclohexane-2-¹⁴C] Mesotrione</p>
 <p>4-Hydroxy-mesotrione</p>	 <p>5-Hydroxy-mesotrione</p>
 <p>MNBA [4-(methylsulphonyl)-2-nitrobenzoic acid]</p>	 <p>AMBA [(2-amino-4-(methylsulphonyl)-benzoic acid)]</p>
 <p>MBA [4-(methylsulphonyl) benzoic acid]</p>	

Animal metabolism

Rats

The metabolism of mesotrione was evaluated at the present Meeting by the JMPR WHO Panel. The compound is rapidly and extensively absorbed, minimally metabolized and excreted primarily in urine after a single or repeated dose. The majority of the radioactivity was excreted as the parent compound within 12 hours post-dose, accounting for 43–64% of the dose in urine. The metabolites found in the excreta includes 4 and 5-hydroxy mesotrione, MNBA and AMBA.

Livestock animals

Metabolism studies with mesotrione were conducted in lactating cows, swine and poultry. Additionally, the metabolism of AMBA, was investigated in the cow.

In two metabolism studies conducted in lactating cows, the animals were dosed with [phenyl- $U-^{14}C$]-mesotrione or [cyclohexane-2- ^{14}C]-mesotrione for 7 consecutive days at a nominal rate of 10 ppm in the diet, and sacrificed 16 hours after the final dose. Over 90% of the administered dose was found in excreta, mostly in faeces. TRR was higher in liver and kidney (0.07 to 0.11 mg eq./kg), reached 0.007 mg eq./kg in muscle, 0.013 mg/kg eq in fat and 0.08 mg eq./kg in milk (at least 90% TRR in skimmed milk). Mesotrione accounted for 10–18% TRR in liver and kidney (0.01–0.02 mg eq./kg). AMBA was identified in kidney of the phenyl label experiment (0.01 mg eq./kg).

One female swine was dosed orally with [phenyl- $U-^{14}C$]-mesotrione for 5 consecutive days at 6 ppm, and sacrificed 23 hours after the final dose. About 90% of the administered dose was recovered in the excreta, mostly in the faeces. Highest TRRs were found in liver (1.75 mg eq./kg) and kidney (0.12 mg eq./kg), with 0.01 mg eq./kg in muscle and 0.006 mg eq./kg in fat. Mesotrione was the main identified residues (90% TRR in liver, 73% TRR in kidney and 78% TRR in muscle). AMBA accounted for up to 2% TRR in tissues (up to 0.029 mg eq./kg in liver). MNBA was only detected in liver (0.005 mg eq./kg).

Two metabolism studies were conducted in poultry, in which hens were dosed for 10 consecutive days at 11 ppm either with [phenyl- $U-^{14}C$]-mesotrione or [cyclohexane-2- ^{14}C]-mesotrione; the hens were sacrificed 16 hours after the final dose. The radioactivity in excreta accounted for over 90% of the administered dose, and contained mesotrione (up to 55% TRR) and AMBA (18% TRR). TRRs were similar in both experiments for liver (1.1–1.2 mg eq./kg) and kidney (0.06–0.07 mg eq./kg), but were higher in the cyclohexane experiment in muscle (up to 0.012 mg/kg eq), fat (up to 0.048 mg eq./kg), reaching 0.094 mg eq./kg in egg yolk, and 0.025 mg eq./kg in the white. Mesotrione was not detected in muscle in any experiment, and was the only compound identified in tissues and eggs in both experiments, corresponding to at least 70% TRR in the liver and fat. In egg yolk, mesotrione accounted for 81% TRR in the phenyl experiment, and 19.5% TRR in the cyclohexane experiment, in which about 15% TRR was shown to be incorporated into palmitic/oleic acid.

A lactating cow received [phenyl- $U-^{14}C$]-AMBA for 7 days at 12.2 ppm in the diet and was sacrificed 23 hours after the final dose. About 90% of the dose was recovered in the excreta, mostly in the faeces. Highest residues were found in kidney (0.053 mg eq./kg), with AMBA accounting for 79% TRR. Perineal fat contained 0.018 mg eq./kg, 62% identified as AMBA. TRR in liver were 0.005 mg eq./kg, and reached a maximum of 0.009 mg eq./kg in milk (day 6), but were not characterized. No radioactive residues were detected in muscle.

In summary, the biotransformation of mesotrione in livestock involves the oxidative cleavage of the parent molecule to yield MNBA, which is reduced in the nitro group to give AMBA. Highest residues were found in liver and kidney, and the levels in muscle were low, reaching a maximum of 0.012 mg eq./kg Mesotrione accounted for up to 18% TRR in cow liver and kidney, at least 70% TRR in tissues of swine and poultry, and up to 80% TRR in egg yolk. No single compound was detected in muscle. The metabolism of Mesotrione in rats was found to be similar to that described for livestock.

Plant metabolism

Metabolism studies were conducted in cranberries, tolerant soya bean, maize, rice and peanuts. [Phenyl- U - ^{14}C]-mesotrione was applied twice to cranberry plants at $0.331 + 0.242$ kg ai/ha ($1\times$) or $0.919 + 0.642$ kg ai/ha ($3\times$ rate), and samples harvested 46 days after the last treatment (DAT). TRRs in mature foliage were 16.8 mg eq./kg and 31.8 mg eq./kg for $1\times$ and $3\times$, respectively. TRRs in the mature cranberry fruit were 2.6 mg eq./kg and 4.9 mg eq./kg, respectively, mostly as mesotrione (60–67% TRR) and AMBA (24–35% TRR), MNBA accounted for up to 3% TRR.

Mesotrione tolerant soya bean seeds grown in sandy loam soil were treated with either [phenyl- U - ^{14}C]- or [cyclohexane-2- ^{14}C]-mesotrione using three GAP application regimes: one pre-emergence at 0.225 kg ai/ha (T1), a combined pre-emergence at 0.225 kg ai/ha followed by a post-emergence at 0.125 kg ai/ha (T2), or one post-emergence at 0.225 kg ai/ha (T3). Forage was sampled at 22–28 DAT, hay at 40–42 DAT and seeds at 90–123 DAT.

Higher radioactivity was recovered from the phenyl label experiment. In forage, TRR were 0.16 to 0.5 mg eq./kg, mostly as MNBA (13 to 24% TRR; 0.04 to 0.06 mg eq./kg); mesotrione and its 4 and 5-hydroxy metabolite accounted for up to 14.6% TRR each (0.01 to 0.08 mg eq./kg). In hay, TRR ranged from 0.14 mg/kg eq (T1) to 2 mg eq./kg (T2), mostly MNBA (up to 20% TRR) and 4/5-hydroxy-mesotrione (up to 16% TRR); mesotrione accounted for up to 9% TRR. AMBA was only detected in T2 hay (0.055 mg eq./kg; 2.7% TRR). Residues in soya bean seed ranged from 0.052 to 0.104 mg eq./kg, with mesotrione and 4/5-hydroxy-mesotrione the main compounds identified (< 10% TRR). Low levels of MNBA and AMBA were found in the T1 and T2 samples (< 5% TRR, 0.005 mg eq./kg).

Results from the cyclohexane experiment showed mesotrione accounting for up to 18% TRR in forage, 8.2% TRR in hay and 5.1% TRR in seed (0.02 mg eq./kg). 4 and 5-hydroxy-mesotrione accounted for up to 19% TRR in forage and hay, and 7% TRR in seeds.

Three studies were conducted with maize, two with [phenyl- U - ^{14}C]-mesotrione and/or one with [cyclohexane-2- ^{14}C]-mesotrione. In all cases, the compound was applied to the soil surface after planting the seeds at a rate of 0.3 kg ai/ha (pre-emergence; T1) or post-emergence at 0.16–0.18 kg ai/ha, 28 days after planting (T2).

Results from the phenyl label experiments showed higher total residues in fodder/stover (0.8 to 1.1 mg eq./kg) and forage (0.244 to 0.356 mg eq./kg). Over 60% of the residues in fodder were not extracted with ACN/water. In T1 forage, MNBA and AMBA were the major residues (up to 19.7 and 12.2% TRR, respectively). In fodder, AMBA was the major residue (up to 28% TRR in T2). 4-hydroxy-mesotrione was mainly present in forage (up to 8% TRR, about 50% conjugated). Mesotrione was a minor component of the residues in all cases, present at a higher level in T1 forage samples (2.2% TRR, 0.008 mg eq./kg). TRR in grain was 0.01 mg/kg eq, and was not further characterized.

In the cyclohexane experiment, TRR reached 0.1 mg eq./kg in forage and 0.33 mg eq./kg in fodder. In forage, the identified residues were mesotrione (up to 3% TRR) and 4-hydroxy-mesotrione (up to 10% TRR). About 18% TRR was incorporated into lignin and cellulose. Residues in grain were low (up to 0.011 mg eq./kg) and were not be further characterized.

Rice plants were treated at the 2–3 leaf stage with [phenyl- U - ^{14}C]-mesotrione added directly to the paddy water at either 0.09 kg ai/ha ($1\times$) or 0.225 kg ai/ha ($2.5\times$). TRRs were higher in whole tops and straw (0.03 to 0.06 mg eq./kg at $1\times$), with 60–71% extracted by ACN/water. Residues in grain and husk (109 DAT) reached 0.01 mg eq./kg, about 33% being extracted (acid released up to 75.1% TRR in grain). Immature whole tops from $1\times$ rate contained mesotrione and 5-hydroxy-mesotrione at up to 0.01 mg/kg eq. each (11 to 15% TRR), and traces of MNBA and AMBA (< 5% TRR from $1\times$). In $1\times$ stalk and straw, mesotrione and its metabolites represented < 10% TRR each. No characterization was performed in grain. Residues from $2.5\times$ samples were 2–5 times higher (0.02 mg eq./kg in grain).

[Phenyl- ^{14}C] or [cyclohexane-2- ^{14}C]-mesotrione were applied to the soil surface after planting peanut seed (pre-emergence) at 0.3 (T1) or 0.8 kg ai/ha (T2). Peanut foliage was harvested 90 DAT (50% maturity), mature peanuts and peanut hay at 153 DAT. Residues from [phenyl- ^{14}C] treatment were higher in foliage (0.028 and 0.064 mg eq./kg, in T1 and T2, respectively) and approximately 0.01 mg eq./kg in hay, hull and nutmeat. Traces of MNBA, MBA, AMBA and 4-hydroxy-misotrione were found in hay (< 6% TRR, ≤ 0.002 mg eq./kg), but only AMBA was found in nutmeat (up to 15% TRR, 0.002 mg eq./kg, in T1). TRR from [cyclohexane-2- ^{14}C] treatment were ≤ 0.01 mg eq./kg in T1 samples ranged from 0.01 to 0.02 mg eq./kg in T2. 4-hydroxy-mesotrione was only identified in hulls (7% TRR). The peanut oil fraction was shown to be composed primarily of ^{14}C -labelled neutral lipids.

In summary, the metabolic pathway of mesotrione following pre- and/or post-emergence foliar applications in cranberries, maize, rice, peanut and tolerant soya bean are similar. It proceeds via cleavage of the parent molecule to yield MNBA and reduction to AMBA, which either conjugated or degraded to MBA. Mesotrione is also hydroxylated in the cyclohexane-dione ring to give 4 or 5-hydroxy-mesotrione. Incorporation of radioactive residues into natural products (lignin cellulose sugar or lipid) was seen in all crops, except cranberry fruit. Residues in cranberry fruits were mostly mesotrione and AMBA (over 20% TRR each). Maize, soya and rice feed commodities contained mostly MNBA and AMBA (> 10% TRR in most cases). Residues in grains were low and mesotrione only represented higher than 10% TRR in tolerant soya bean seed.

Environment fate

The photolysis of [phenyl- ^{14}C] mesotrione and [cyclohexane- ^{14}C]-mesotrione was studied in silt loam soil treated at 0.3 kg ai/ha and incubated in local sunlight (latitude 37° 56') at 20 to 24°C. About half of the radioactivity was present as mesotrione at 12–13 DAT. MNBA and AMBA accounted for 2–8% TRR at 5 DAT, increasing up to 8% TRR at 30 DAT.

The metabolism of [phenyl-2- ^{14}C] or [cyclohexane- ^{14}C]-mesotrione applied to various soils at rates ranging from 0.165 to 0.85 kg ai/ha and kept under aerobic conditions in the dark at 25±1°C for 28 to 60 days was investigated. Mesotrione degrades relatively fast, with DT_{50} values ranging from 4.5 to 32 days. DT_{50} for MNBA was < 2 days in these studies.

In two water sediment systems experiments conducted with either [phenyl-2- ^{14}C] or [cyclohexane- ^{14}C]-mesotrione at 0.20 kg ai/ha and incubated in the dark for 101 days, showed DT_{50} were from 3 to 6 days, with mesotrione in the sediment never exceeding 4% AR. MNBA and AMBA were found in both water and sediment, starting at day 3.

The aerobic degradation of [phenyl-2- ^{14}C]-AMBA was studied in soils incubated up to 60 days in the dark, showing DT_{50} ranging from 2 to 6 days.

Field studies

In six studies conducted with soils collected from different regions of Europe, mesotrione was applied at 0.15–0.2 kg ai/ha. MNBA and AMBA were detected at 6 DAT in 0–10 cm horizon (0.031 and 0.006 mg eq./kg, respectively). No residues of mesotrione or metabolites were detected in the soil below 10 cm. DT_{50} ranged from 2 to 8 days.

In one study conducted with four soils from England and USA treated with MNBA at 0.22 kg ai/ha, DT_{50} ranged from 0.6 to 10.6 days.

Confined rotational crops

Endive, radish and wheat were sown 120 days after a sandy loam soil being treated with [phenyl- ^{14}C] or [cyclohexane-2- ^{14}C]-mesotrione at 0.165 kg ai/ha. Endive was harvested at 78–63 days after planting (DAP), radish roots and leaves at 56 DAP, wheat forage at 22 DAP, wheat hay at 57 DAP and wheat grain and straw at 134–131 DAP. In the [phenyl- ^{14}C] experiment, residues in soil declined to 34% of the applied radioactivity (AR) at 120 DAT, with the most abundant metabolites being MNBA (8% AR) and AMBA (2% AR); mesotrione accounted for only 0.1% AR. TRR were

0.02 to 0.04 mg eq./kg in wheat forage, hay and straw, mostly MNBA (0.011 mg eq./kg in forage). Residues were 0.006 mg eq./kg in wheat grain, 0.014 mg eq./kg in endive and 0.004 mg eq./kg in radish root and leaves. TRR in all cyclohexane-2-¹⁴C samples were < 0.005 mg eq./kg in endive, and wheat straw, and were not further characterized.

[Phenyl-U-¹⁴C] or [cyclohexane-2-¹⁴C]-mesotrione was applied at 0.308 kg ai/ha (T1, characteristic of pre-emergence) and 0.462 kg ai/ha (T2, characteristic of pre + post-emergence) onto a sandy loam soil, and wheat, soya, endive or radish planted at 30, 120 and/or 300 DAT. Residues in soil declined to 27% AR at 300 DAT. Residues in wheat commodities from the [phenyl-U-¹⁴C] experiment were higher in straw (2.58 mg eq./kg at 30 DAT, T1). In wheat grain, residues were 0.038 mg eq./kg at 30 DAT (T1) and 0.014–0.015 mg eq./kg at 120 and 300 DAT (T2). At 30 DAT (T1), the major identified metabolite was MNBA, with residues ranging from 0.17 to 0.63 mg eq./kg in wheat forage, hay and straw and 0.003 mg eq./kg in grain. AMBA was mostly present as sulphate conjugate (total of 0.67 mg eq./kg in straw), and mesotrione and its 4-OH metabolite were only detected in forage (0.01 mg eq./kg).

At 30 DAT (T1), residues were 0.145 mg eq./kg in soya bean, and 0.46–0.64 mg eq./kg in soya feed. MNBA was 0.17–0.31 mg eq./kg in forage and hay and 0.014 mg eq./kg in soya bean. AMBA levels were 0.02–0.07 mg eq./kg. Residues in endive and radish ranged from 0.037 to 0.053 mg eq./kg at 120 DAT, declining to 0.005 to 0.019 mg eq./kg at 300 DAT (T2). The major residue was MNBA (0.02 mg eq./kg at 120 DAT, T2, in endive and radish tops).

Highest residues from [cyclohexane-2-¹⁴C] experiment were found at 30 DAT, T1: 0.05 - 0.06 mg eq./kg in wheat feed, 0.01 mg eq./kg in wheat grain, and 0.02–0.03 mg eq./kg in soya bean samples. Residues in endive and radish were < 0.01 mg eq./kg. Mesotrione and 4-hydroxy mesotrione were identified in wheat and soya bean feed (< 0.01 mg eq./kg at 30 DAT, T1).

In summary, mesotrione degrades quickly in soil under aerobic conditions. Although mesotrione is relatively stable to hydrolysis at pH 5–9 (less than 10% degradation after 30 days at 25 °C), it degrades rapidly in flooded systems with a half-life of approximately 4 days. Mesotrione metabolites, mainly MNBA, are expected in wheat and soya bean feed, endive and radish root when the crops are planted up to 120 days after the soil is treated with mesotrione at 0.3 kg ai/ha rate or higher. As currently the compound is used at rates lower than 0.3 kg ai/ha, no residues arriving from the use of mesotrione are expected in rotational crops.

Methods of analysis

Mesotrione residues in vegetable crops may be analysed by LC-MS/MS (negative mode, m/z=338 → 291) after extraction with acetonitrile/water and cleaned up by SPE. Recovery data for mesotrione in maize commodities and cranberries showed good performance (84–114% recovery, 3–21% RSD, n=3–6) at the 0.01 mg/kg (LOQ) to 10 mg/kg range. The method was used in various supervised trials, with recovery data for mesotrione and MNBA within the acceptable levels at the LOQ or higher.

A modified QuEChERS LC-MS/MS multi-residue method (no clean up with primary-secondary amine (PSA) is used) was validated for mesotrione in oranges, maize and oilseed rape, with a LOQ of 0.01 mg/kg.

In a reversed-phase HPLC-fluorescence method, mesotrione and MNBA residues are extracted with acetonitrile:water (1:1) and cleaned up on silica SPE. The extract is submitted to reversed phase HPLC, the mesotrione fraction converted to MNBA with H₂O₂ and reduced to AMBA using acidic SnCl₂ and the MNBA fraction reduced to AMBA. Each fraction is cleaned-up by C18 SPE, and the AMBA conversion product quantified by HPLC-fluorescence. The method was validated for corn commodities with a LOQ of 0.01 mg/kg.

In a GC-MS method, mesotrione and MNBA residues are extracted from corn commodities with acetonitrile:water (1:1), acidified, partitioned with methylene chloride, which is evaporated and the residue heated with Jones Reagent (Cr^{VI} oxide acid solution) to oxidize mesotrione to MNBA. The total MNBA is extracted with ethyl acetate, evaporated to dryness, and the residue reacted with 2-

iodopropane and potassium carbonate to form isopropyl ester of MNBA for analyse by GC-MS. The method determines both mesotrione and MNBA at a combined LOQ of 0.01 mg/kg.

The acetonitrile:water (1:1) extraction efficiency was radio-validated using incurred radioactive residues in forage. After extraction using a high speed homogeniser, an aliquot was partitioned three times into ethyl acetate, and residues of mesotrione and MNBA quantified by TLC with storage-phosphor autoradiography. Levels of mesotrione and MNBA in forage were similar to the results obtained after exhaustive extraction within the metabolism study.

Mesotrione and MNBA residues are extracted from milk and eggs with acetone and from animal tissues with an acetone:water, the extract acidified, partitioned into methylene chloride, and residues of mesotrione oxidised to MNBA using H₂O₂. MNBA is reduced with acidic SnCl₂ and AMBA determined by reversed phase HPLC-fluorescence detection. The LOQ was 0.01 mg/kg in all matrices. Mesotrione may also be determined in animal matrices using the modified QuEChERS, excluding PSA, at an LOQ of 0.01 mg/kg.

The analytical methods were considered fit for purpose to determine mesotrione alone or in combination with MNBA in plant and animal commodities at a LOQ of 0.01 mg/kg.

Stability under frozen conditions

Residues of mesotrione and/or MNBA in fortified samples of maize commodities, radish root, and soya bean seed at 0.1 mg/kg were stable under frozen conditions for at least 32 months (at least 80% of the residues remained, quantified as AMBA by HPLC-FL). Samples of blueberry, asparagus, sugarcane and okra fortified with mesotrione at 1.0 mg/kg were shown to be stable for at least 13 months when stored frozen (quantified by HPLC-MS/MS). The residue trials reports also include additional information on storage stability, and the samples were stored within the period that guaranteed the integrity of the residues at the time of analysis.

Definition of the residue

Metabolism studies conducted in cow, swine and poultry fed with ¹⁴C mesotrione at 6 to 11 ppm showed higher residues in liver and kidney, and ranged from 0.01 to 0.08 mg eq./kg in muscle, milk and in eggs. When detected, mesotrione was the main residue found in animal commodities, accounting for up to 18% TRR in cow liver and kidney, at least 70% TRR in tissues of swine and poultry, and up to 80% TRR in egg white. When cow was fed with ¹⁴C AMBA, residues reached a maximum of 0.05 mg eq./kg in kidney and fat, with over 60% as AMBA. Residues in other tissues and in milk were < 0.01 mg eq./kg

The Meeting agreed that the residue definition of mesotrione in animal commodities for enforcement and dietary exposure assessment is mesotrione.

The residues do not concentrate in fat and mesotrione has a log P_{ow} of 0.1, confirming that mesotrione is not fat soluble.

Mesotrione is a herbicide that can be applied to the soil pre and/or post emergence of the plant, with exception of cranberry, for which the use is foliar. The compound is rapidly degraded in soil. Metabolism study showed residues in cranberry fruits mostly as mesotrione (over 60% TRR) and AMBA (over 20% TRR). Metabolism studies conducted in tolerant soya bean, maize, rice and peanut showed higher residues in feed commodities, mostly as mesotrione (up to 28% TRR in rice tops), MNBA (up to 24% TRR in soya bean forage) and AMBA (up to 29% TRR in maize fodder). Total residues in edible commodities were low (≤ 0.03 mg eq./kg) and when characterized, showed mesotrione as the main residue.

The Meeting concluded that MNBA and AMBA appear to be of low toxicological concern. When the information was available, MNBA was not detected in any sample from the residue trials.

The Meeting agreed that mesotrione is an adequate marker for the uses of mesotrione in plants and is suitable for dietary intake assessment

The Meeting agreed in the following residue definition for both plant and animal commodities for enforcement and dietary risk assessment: *Mesotrione*

The residues are not fat soluble.

Residues of supervised residue trials on crops

Cranberry

GAP in USA for cranberries is 2 broadcast foliar applications at 0.28 kg ai/ha, PHI 45 days. In five trials using 2 applications, the first being at 0.388 kg ai/ha, residues were: < 0.01 mg/kg (5), indicating that no residues are expected when the product is applied at the GAP rate.

The Meeting agreed to recommend a maximum residue level of 0.01* mg/kg, and a STMR of 0 mg/kg for mesotrione in cranberries

Bush berries and cane berries

GAP in USA for bush and cane berries is 1 post-direct spray at 0.21 kg ai/ha before bloom, with no PHI specified.

In one trial conducted in blueberry in USA according to GAP (application at BBCH 59), residues were < 0.01 mg/kg (77 DAT). In five other trials where the application was done after bloom residues at 32 to 88 DAT were < 0.01 mg/kg.

In four trials conducted with raspberry at GAP, residues at 32 to 88 DAT were < 0.01 mg/kg (4) at 52 to 83 DAT.

As no residues above the LOQ were found even in the late application trials, the Meeting agreed to estimate a maximum residue level of 0.01* mg/kg and a STMR of 0 mg/kg for bush berries and cane berries

Okra

In USA, mesotrione can be applied either at pre-emergence (0.21 kg ai/ha) or post emergence (0.105 kg ai/ha). PHI in both cases is 28 days. Five post-emergence trials conducted according to GAP gave residues < 0.01 mg/kg (5).

The Meeting agreed to recommend a maximum residue level of 0.01* mg/kg, and a STMR of 0.01 mg/kg for mesotrione in okra.

Sweet corn

Mesotrione is registered in Germany for post-emergence use on sweet-corn (BBCH 12–18) at 0.15 kg ai/ha. In four trials conducted in France at this GAP rate gave residues were: < 0.01 mg/kg (4) in the kernels and in the cob at 38 to 61 DAT.

In USA, mesotrione can be used in sweet corn via three application regimes: 1) one pre-emergence application at 0.27 kg ai/ha, 2) two post emergence applications, with a maximum of 0.21 kg ai/ha; or 3) 1× pre + 1× post emergence, with a maximum of 0.27 kg ai/ha. In all cases, the PHI is 45 days. The second application should be done up to the 8 leaf stage. In one trial, conducted according to regime 2, residues in the ears were: < 0.01 mg/kg (2); other two trials conducted at the same rate residue were the same 28 to 32 DAT. In 12 trials conducted a higher rate (0.48 to 0.50 kg ai/ha; regimes 1 or 2 residues were: < 0.01 mg/kg from 23 to 36 DAT.

Although only one trial was conducted in USA according to GAP, 14 trials conducted at higher rates and/or lower PHI showed that no residues are expected in the ears of sweet corn after treatment according to GAP.

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg and a STMR of 0 mg/kg for mesotrione in sweet corn (kernels plus cob without husk).

Soya bean, dry

In USA, GAP for mesotrione in conventional soya is one pre-emergence application at 0.21 kg ai/ha, with no PHI specified. In 20 trials conducted according to GAP, residues at 117 to 174 DAT were < 0.01 mg/kg (20). Three trials conducted at higher rates (0.6–1 kg ai/ha) gave the same results.

GAPs for mesotrione tolerant soya are i) one pre-emergence or ii) early post-emergence (up to BBCH 13) application at 0.225 kg ai/ha, or iii) one pre + one post emergence application (BBCH 14–60) at 0.225 kg ai/ha and 0.125 kg ai/ha, respectively. Forty seven trials were conducted with tolerant soya using application using regimes 2 or 3, residues in the mature seeds were: < 0.01 (44) and 0.02 (3) mg/kg.

Using the data from trials conducted in tolerant crops, the Meeting agreed to estimate a maximum residue level of 0.03 mg/kg and a STMR of 0.01 mg/kg for mesotrione in soya bean, dry.

Asparagus

In the USA, mesotrione can be use in asparagus either as a pre-emergence application on the soil surface at 0.27 kg ai/ha in the spring prior to spear emergence, one application after completion of harvesting directed to the weed at 0.105 kg ai/ha, or both at a maximum of 0.27 kg ai/ha. In eight trials conducted in USA using the pre-emergence GAP, residues at 8 to 18 DAT were < 0.01 mg/kg (8). In 16 other trials the application was done after emergence of the plant.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0.01 mg/kg for mesotrione in asparagus.

Rhubarb

In USA, mesotrione can be use in rhubarb as pre-emergence application on the soil surface prior to any spring green-up at 0.21 kg ai/ha and 21 days PHI. In four trials conducted at GAP rate, residues at 28 to 42 DAT were < 0.01 mg/kg. Four trials were conducted at higher rates and gave the same result.

As the PHI is not relevant to a pre-emergence application, the Meeting agreed to estimate maximum residue level of 0.01* mg/kg and a STMR of 0.01 mg/kg for mesotrione in rhubarb.

Maize

Mesotrione is registered in Germany for post-emergence use on maize (BBCH 12–18) at 0.15 kg ai/ha. In two trials conducted in Germany and UK at this GAP gave results at 112 to 143 DAT of < 0.01 mg/kg (2).

In the USA, mesotrione can be used in maize in three application regimes: 1) one pre-emergence at 0.27 kg ai/ha, 2) two post emergence, with a maximum of 0.21 kg ai/ha; or 3) 1× pre + 1× post emergence, with a maximum of 0.27 kg ai/ha. The second application should be done up to the 8 leaf stage. In all cases, the PHI was 45 days. Eight trials were conducted in Canada and the USA using regime 1 and 32 trials using regime 3 at rates higher than USA GAP. Grain harvested at 68 to 145 DAT gave residues < 0.01 mg/kg.

The results from North American trials conducted at higher rate show that no residues are expected in maize grain after treatment according to GAP.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in maize grain.

Millet

Mesotrione is registered in the USA as one pre-emergence use at 0.21 kg ai/ha, and no PHI specified. In five trials conducted according to GAP residues were: < 0.01 mg/kg (5) in millet grain (84 to 132 DAT).

With the support from the data from other cereals, the Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in millet grain.

Oat

Mesotrione is registered in USA either as one pre-emergence use at 0.21 kg ai/ha or as a post-emergence application at 0.105 kg ai/ha. PHI is 50 days. In sixteen post-emergence trials conducted at GAP, residues in oat grain were < 0.01 mg/kg (16). Two trials conducted at up to 5 times the rate gave the same results.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in oat grain.

Rice

Mesotrione is registered in paddy rice in Republic of Korea as post-planting into the water (5–7 days after transplanting) at 1×0.09 kg ai/ha and no PHI specified. Ten trials were conducted in Republic of Korea and Japan using either a single application at higher rate, two applications at the GAP rate and/or applying latter in the season. In all cases, residues at 45 to 140 DAT were < 0.01 mg/kg. Although the trials were not according to GAP, these results indicate that no mesotrione residues are expected when the product is used according to GAP.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in rice grain, husked.

Sorghum

The registered use for mesotrione in sorghum in the USA is one pre-emergence application at 0.224 kg ai/ha up to 21 days before planting. In nine trials conducted according to GAP, residues at 78 to 134 DAT were < 0.01 mg/kg (9). Twelve post-emergence trials gave the same results.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in sorghum.

Sugar cane

The GAP for mesotrione in sugarcane in the USA is either two post-emergence applications at 0.10 kg ai/ha to the base of the sugar cane or a combination of one pre- and one post-emergence application not exceeding a total rate of 0.36 kg ai/ha. PHI is 114 days. In twenty four trials conducted according to either of the GAPs in USA gave residues of < 0.01 mg/kg (24) from 30 to 118 DAT. Two trials conducted at 3 or 5× the rate gave the same results within 114 days PHI.

In South Africa, GAP is a single early post-emergence at 0.15 kg ai/ha, and 181 days PHI. In four trials conducted at higher rate no residues were detected.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in sugar cane.

Linseed

Mesotrione is registered in the USA for linseed at one pre or one post-emergence application at 0.21 kg ai/ha, and no PHI specified. Five pre-emergence trials conducted according to GAP gave residues of < 0.01 mg/kg (5). Two post-emergent trials conducted at higher rates gave the same result.

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg and a STMR of 0.01 mg/kg for mesotrione in linseed.

*Animal feed**Forage*

Mesotrione is registered in Germany for post-emergence use on maize (BBCH 12–18) at 0.15 kg ai/ha, and no PHI specified. In three trials conducted in maize in France, Germany and the UK matching German GAP, residues in stover (remaining plant) at 41–47 DAT were 0.01 mg/kg (3).

In the USA, mesotrione can be used in maize under three application regimes: 1) one pre-emergence at 0.27 kg ai/ha; 2) two post emergence, with a maximum of 0.21 kg ai/ha; or 3) 1× pre + 1× post emergence, with a maximum of 0.27 kg ai/ha. In all cases, PHI was 45 days for forage and stover. The second application should be made up to the 8 leaf stage (or BBCH 19).

In one trial conducted in USA according to GAP, residues in maize forage were 0.12 mg/kg.

Mesotrione is registered in USA in millet as one pre-emergence use at 0.21 kg ai/ha. In five trials conducted at GAP, residues in forage were < 0.01 mg/kg (5).

The Meeting estimated a median residue and a highest residue of 0.01 mg/kg for mesotrione in millet forage

Mesotrione is registered in oats in USA either as one pre-emergence use at 0.21 kg ai/ha or as post-emergence application at 0.105 kg ai/ha. In 16 pre-emergence trials and 16 post-emergence US trials matching GAP, residues in oat forage were < 0.01 mg/kg (32).

The Meeting estimated a median residue and a highest residue of 0.01 mg/kg for mesotrione in oat forage.

The registered use for mesotrione in sorghum in USA is one pre-emergence application at 0.224 kg ai/ha up to 21 days before planting. In 13 trials conducted according to GAP, residues in sorghum forage were < 0.01 mg/kg (13).

The Meeting estimated a median residue and a highest residue of 0.01 mg/kg for mesotrione in sorghum forage.

Hay

In 16 pre-emergence trials and 16 post-emergence trials conducted in oats in USA, matching GAP, residues in oat hay were < 0.01 mg/kg (32). Post-emergence application trials gave the same results.

In five trials conducted in millet at GAP, residues in hay were < 0.01 mg/kg.

The Meeting estimated a median residue of 0.01 mg/kg and a highest residue of 0.01 mg/kg for mesotrione in oat hay and millet hay.

Straw

Mesotrione is registered in paddy rice in Korea as post-planting into the water (5–7 days after transplanting) at 1×0.09 kg ai/ha and no PHI specified. In eight trials conducted in Japan at this GAP gave residues in straw of < 0.002 mg/kg.

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg for mesotrione in rice straw and fodder, dry

The Meeting estimates a median residue and a highest residue of 0.01 mg/kg for mesotrione in rice straw

Fate of residues during processing

A processing study conducted with soya bean containing 0.04 mg/kg mesotrione showed residues of 0.01 mg/kg in the meal and 0.07 mg/kg in flour, with calculated processing factors of 0.25 and 1.8 mg/kg, respectively. Residues in soya oil, milk, tofu, sauce and miso were < 0.01 mg/kg, with an estimated processing factor of < 0.25. Based on a STMR of 0.01 mg/kg for soya bean, dry, the Meeting estimated a STMR-P of 0.018 mg/kg in soya bean flour, and of 0.002 mg/kg for soya oil, milk, tofu, sauce and miso.

Residue in animal commodities

Farm dietary burden

The Meeting estimated the dietary burden of mesotrione in farm animals on the basis of the OECD Animal Feed data published in the 2009 FAO Manual, and the median and highest residue levels estimated at the present Meeting for oat and sorghum forage, oat hay and rice straw.

The maximum and the mean dietary burden was 0.03 ppm for cattle, 0.01 and 0.001 ppm, for swine, respectively, and 0 ppm for poultry.

Animal commodity maximum residue level

No feeding study on mesotrione was provided to the Meeting. The metabolism study conducted with cattle at 10 ppm, gave residues of mesotrione up to 0.02 mg/kg in tissues and milk. Swine fed with radiolabeled mesotrione at 6 ppm gave residues of 1.5 mg/kg in liver, 0.09 mg/kg in kidney and 0.01 mg/kg in muscle. Interpolation of the residues found in the metabolism studies to what would be expected at the calculated dietary burden indicates that no residue will exceed 0.0025 mg/kg (in swine liver).

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg for mesotrione in milks, edible offal (mammalian) and meat (from mammals other than marine mammals).

The Meeting also estimated a STMR of 0 for mesotrione in milk and meat (from mammals other than marine mammals), and edible offal (mammalian).

Metabolism study conducted with poultry at 11 ppm showed mesotrione residues of 1.1 mg/kg in liver, 0.03 mg/kg in fat, < 0.01 mg/kg in meat and 0.02 mg/kg in eggs. As the dietary burden for poultry is 0, the Meeting agreed to estimate a maximum residue level of 0.01* mg/kg, and a STMR and a HR of 0 mg/kg for mesotrione poultry meat, poultry offal and eggs.

RECOMMENDATIONS

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

The residue is not fat soluble.

CCN	Commodity name	Maximum residue level (mg/kg)	STMR (P) (mg/kg)
VS 0621	Asparagus	0.01*	0.01
FB 2006	Bush berries	0.01*	0
FB 2005	Cane berries	0.01*	0
FB 0265	Cranberry	0.01*	0
MO 0105	Edible offal (mammalian)	0.01*	0
PE 0112	Eggs	0.01*	0
SO 0693	Linseed	0.01*	0.01
GC 0645	Maize	0.01*	0
MM 0095	Meat (from mammals other than marine mammals)	0.01*	0
GC 0646	Millet	0.01*	0
MI 0106	Milks	0.01*	0
GC 0647	Oat	0.01*	0
VO 0442	Okra	0.01*	0.01
PO 0111	Poultry, Edible offal of	0.01*	0
PM 0110	Poultry meat	0.01*	0
CM 0649	Rice, husked	0.01*	0
VS 0627	Rhubarb	0.01*	0.01
GC 0651	Sorghum	0.01*	0

VD 0541	Soya bean, dry	0.03	0.01
	Miso		0.002
	Soya flour		0.018
	Soya milk		0.002
	Soya oil		0.002
	Soya sauce		0.002
	Tofu		0.002
GS 0659	Sugarcane	0.01*	0
VO 0447	Sweet corn (kernels plus cob without husk)	0.01*	0

Animal Feed items

	Median residue, mg/kg	Highest residue, mg/kg
Millet forage	0.01	0.01
Oat forage	0.01	0.01
Sorghum forage	0.01	0.01
Millet hay	0.01	0.01
Oat hay	0.01	0.01
Rice straw	0.01	0.01

DIETARY RISK ASSESSMENT

Long-term intake

The IEDI of mesotrione based on the STMRs estimated by this Meeting for the 17 GEMS/Food regional diets were 0% of the maximum ADI of 0–0.3 mg/kg bw (see Annex 3 of the 2014 Report). The Meeting concluded that the long-term dietary intake of residues of mesotrione is unlikely to present a public health concern.

Short-term intake

The 2014 JMPR decided that an ARfD is unnecessary for mesotrione. The Meeting therefore concluded that the short-term intake of residues of mesotrione is unlikely to present a public health concern.

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