HALOXYFOP (194) AND HALOXYFOP-P

The first draft was prepared by Mr. D. J. Hamilton Department of Primary Industries and Fisheries Brisbane, Australia

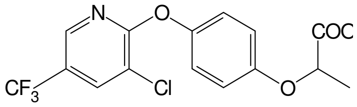
EXPLANATION

Residue and analytical aspects of haloxyfop were evaluated by the JMPR in 1995, 1996 and 2001. The compound was listed in the Periodic Re-Evaluation Program at the 39th Session of the CCPR for periodic review by the 2009 JMPR. The most recent toxicological review was in 2006 when a group ADI of 0–0.0007 mg/kg bw was established for racemic haloxyfop, haloxyfop-R and their methyl esters. The 2006 JMPR also established a group ARfD of 0.08 mg/kg bw for racemic haloxyfop, haloxyfop-R and their methyl esters.

The primary manufacturer provided a full residue data package. GAP information was also provided by Australia and The Netherlands.

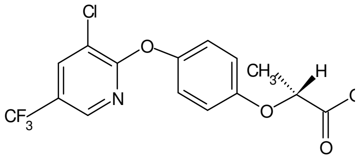
IDENTITY

Haloxyfop was developed as a selective herbicide for the control of grass weeds in broad-leaf crops. Originally it was produced as a racemic mixture. The R-isomer is the herbicidal active compound. Currently, the only compound available commercially is haloxyfop-R as methyl ester. When applied to plants, the ester is rapidly hydrolysed to acid, which has herbicidal activity.

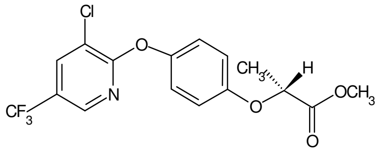
HALOXYFOP	
ISO common name (Wood, 2009)	haloxyfop
IUPAC name (Wood, 2009)	(RS)-2-{4-[3-chloro-5-(trifluoromethyl)-2-pyridyloxy]phenoxy}propionic acid
Chemical Abstracts name (Wood, 2009)	2-[4-[[3-chloro-5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid
CAS Number (Wood, 2009)	69806-34-4
CIPAC Number	438
Molecular formula	$C_{15}H_{11}ClF_3NO_4$
Molecular mass	361.7
Structural formula	
HALOXYFOP-P	
ISO common name (Wood, 2009)	haloxyfop-P
Synonyms:	haloxyfop-P: DE-535 acid, haloxyfop-R haloxyfop-P-methyl: XRD-535 (until 1989); XDE-535 (1990-1993); DE-535 (since 1993)
IUPAC name (Wood, 2009)	(*R*)-2-{4-[3-chloro-5-(trifluoromethyl)-2-pyridyloxy]phenoxy}propionic acid
Chemical Abstracts name (Wood, 2009)	(2*R*)-2-[4-[[3-chloro-5-(trifluoromethyl)-2-pyridinyl]oxy]phenoxy]propanoic acid
CAS Number (Wood, 2009)	haloxyfop-P: 95977-29-0 haloxyfop-P-methyl: 72619-32-0

CIPAC Number	haloxyfop-P: 526 haloxyfop-P-methyl: 526.201
Molecular formula	haloxyfop-P: $C_{15}H_{11}ClF_3NO_4$ haloxyfop-P-methyl: $C_{16}H_{13}ClF_3NO_4$
Molecular mass	haloxyfop-P: 361.7 haloxyfop-P-methyl: 375.7

Structural formula



haloxyfop-P



haloxyfop-P-methyl

Physical and chemical properties

Pure active ingredient: Haloxyfop-P-methyl

Property	Result	Ref
Description (purity 99%)	Viscous, clear, colourless liquid	GHE P 2140
Vapour pressure (purity 99.7%)	2.6×10^{-5} Pa at 20 ºC 5.5×10^{-5} Pa at 25 ºC	GHE-P-4060
Water solubility (purity 99.7%) at 20 °C, OECD 105	9.1 mg/L purified water 6.9 mg/L pH 5 buffer 7.9 mg/L pH 7 buffer	GHE-P-4060
Water solubility (purity 99%) at 25 °C, OECD 105	8.7 mg/L distilled water 9.3 mg/L pH 5 buffer 7.8 mg/L pH 7 buffer	GHE P 2140
Octanol/water partition coefficient (purity 99%), OECD 107	log K_{OW} = 4.05 at 20 ºC	GHE P 2140
Octanol/water partition coefficient (purity 99.7%), OECD 107	log K_{OW} = 4.00 at 20 ºC	GHE-P-4060
Hydrolysis rate (radiochemical purity 96.5–> 99%[2]), 31 days at 0.5 mg/L at 20 ºC.	pH 4 buffer, sterile: approx 5% loss in 31 days pH 7 buffer, sterile: DT_{50} = 43 days pH 9 buffer, sterile: DT_{50} = 0.63 days Natural water, pH 8: DT_{50} = 3 days. Main hydrolysis product: haloxyfop-P acid, stable to further hydrolysis. Minor products < 3% of starting material.	000133
Photolysis rate (radiochemical purity 97.8% and 98.2%[2]), 2 mg/L aqueous at 20 ºC. Average summer sunlight at 40ºN latitude. 400 hours continuous irradiation.	pH 5 buffer, sterile: photolysis DT_{50} 135 hours pH 5 buffer, sterile: dark control, stable Natural water, pH 8.5, non-sterile: photol DT_{50} 11 hours Natural water, pH 8.5, non-sterile: dark control DT_{50} 37 hours	000421
Dissociation constant in water	Dissociation not predicted for haloxyfop-P-methyl	

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[2] [^{14}C]phenyl label and [^{14}C]3,5-pyridine label.

Technical material: Haloxyfop-P-methyl

		Ref
Description (purity 91.7%)	Viscous brown liquid	GHE P 2140
Description (purity 98.6%)	A light amber, viscous liquid with a faint, characteristic odour.	FAPC003116
Vapour pressure (purity 91.7%) OECD 104	3.28×10^{-4} Pa at 25 °C	GHE P 2140
Solubility in organic solvents at 20 °C (purity 98.6%)	Highly soluble (more than 50% by weight) in these solvents: acetone, Aromatic 100, cyclohexanone, dichloromethane, DMF, ethanol, ethyl acetate, hexane, isopropanol, methanol, toluene and xylene.	GH-C 2162

Pure active ingredient: Haloxyfop-P

		Ref
Melting point (purity 99.3%)	70.4–74.5 °C	NAFST272
Vapour pressure, EEC Test A4	3.5×10^{-6} Pa at 25 °C [a]	NAFST272 9^{d}1314 DES/182
Water solubility (purity 99.3%) at 20 °C, EEC Test A6	375 mg/L purified water 28.2 g/L buffer pH 5.0 (solubility of Na salt) > 25% w/V buffer pH 7.0 (solubility of Na salt) > 25% w/V buffer pH 9.0 (solubility of Na salt)	NAFST272
Octanol-water partition coefficient (purity 99.3%), EEC Test A8	log K_{OW} = 2.8 buffer pH 4 log K_{OW} = 0.27 buffer pH 7 [b] log K_{OW} = 0.21 buffer pH 10 [b]	NAFST272
Photolysis rate (radiochemical purity 97.5% and 99.5%[a]), 1 mg/L aqueous at 20 °C. Average summer sunlight at 40°N latitude. 210 hours continuous irradiation.	pH 5 buffer, sterile: photolysis DT_{50} 68 hours pH 5 buffer, sterile: dark control, stable	000421
Dissociation constant (purity 99.3%), OECD 112	pKa = 4.27	NAFST272
Solubility in organic solvents (purity 99.3%) at 20 °C, CIPAC MT 157.1 and a flask shaking method [c]	Haloxyfop-P was highly soluble (> 2000 g/L) in acetone, acetonitrile, ethyl acetate and methanol; also in dichloroethane (> 1300 g/L). Xylene 639 g/L n-Octanol 1510 g/L n-Heptane 3.93 g/L	NAFST272

[a] The test material may have been haloxyfop rather than haloxyfop-P. Report DES/182 described the test material as a white lumpy powder with melting point 107–108 °C. Vapour pressure measurements were made over a temperature range of 59.35 °C to 103.9 °C, where the material was assumed to be solid, for extrapolation to the solid at 25 °C.

[b] The buffers were sodium and potassium salts, so haloxyfop-P would be present as a sodium or potassium salt.

[c] Method MT 157.1 is a method for water solubility (Dobrat and Martijn, 1995), but here adapted for solvents. Although report NAFST272 mentions MT 157.2, it was not used. In addition to MT 157.1, a flask shaking method was used for measuring solubility in xylene, n-octanol and n-heptane.

Identified photolytic products

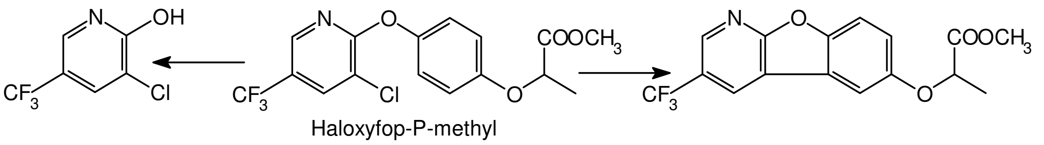


Figure 1 Identified products of photolysis of haloxyfop-P-methyl in a sterile pH 5 buffer (Cook and Balcer, 2002, 000421)

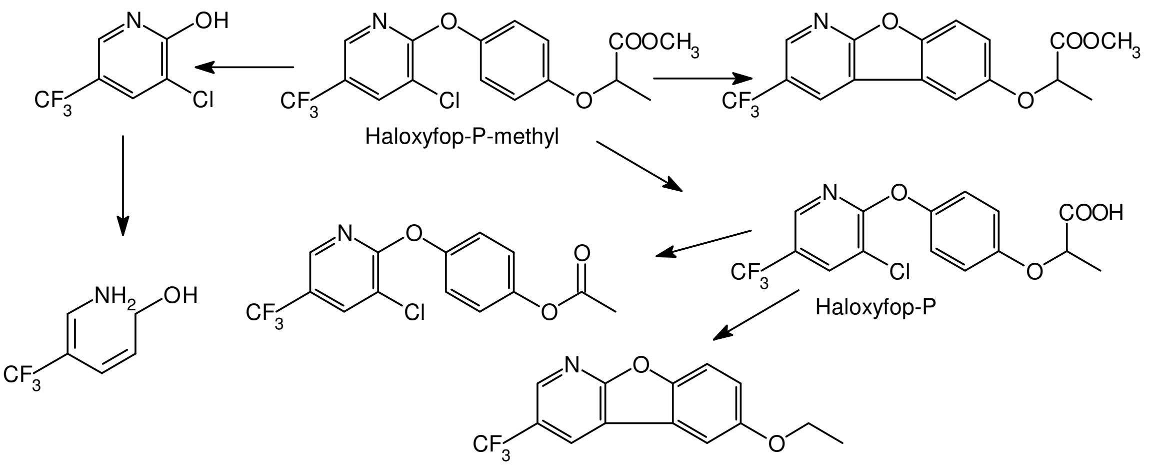


Figure 3 Identified products of photolysis (and hydrolysis) of haloxyfop-P-methyl in a non-sterile natural water (Cook and Balcer, 2002, 000421)

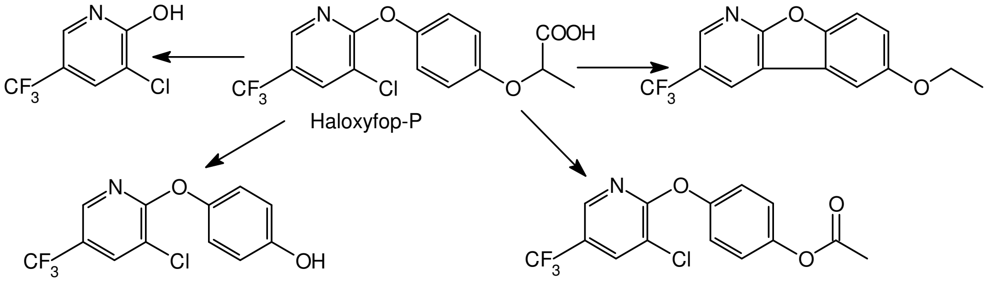


Figure 3 Identified products of photolysis of haloxyfop-P in a sterile pH 5 buffer (Cook and Balcer, 2002, 000421)

Technical material: Haloxyfop-P

		Ref
Description (purity 98.8%)	Off-white, odourless, powder.	FAPC003117
Density (purity 98.8%)	1.46 g/mL at 21 °C	46028

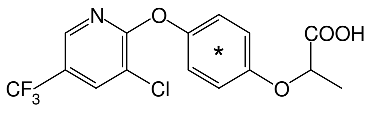
Formulations

The main formulation type is EC with haloxyfop or haloxyfop-P formulated as esters. Neither haloxyfop nor haloxyfop-P is co-formulated with other pesticides.

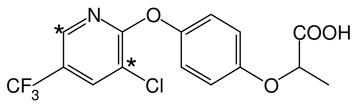
Code	Description		Examples
EC	emulsifiable concentrate	haloxyfop-P, methyl ester	Galant R, Mirage, Verdict R, Verdict 600, Dowco 535.
EC	emulsifiable concentrate	haloxyfop, ethoxyethyl ester	Galant, Gallant.

METABOLISM AND ENVIRONMENTAL FATE

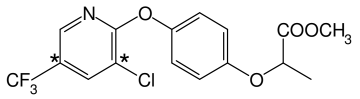
Metabolism and environmental fate studies used haloxyfop (as esters) ^{14}C labelled in the phenyl ring or in the 3,6 positions of the pyridine ring. Haloxyfop-P-methyl labelled in the 3,5 positions of the pyridine ring was also used .



phenyl ring



3,6 positions of pyridine ring



3,5 positions of pyridine ring

Animal metabolism

The Meeting received animal metabolism studies with haloxyfop in lactating goats and laying hens. The 2006 JMPR received information on the metabolism in laboratory animals.

After oral dosing of livestock with haloxyfop, much of the residue is readily excreted. Parent compound and its conjugates constitute the main component of the residue in tissues, milk and eggs. The residue, including the triacylglycerides, is fat soluble.

Laboratory animals

The metabolic fate of orally administered racemic haloxyfop and haloxyfop-P-methyl in mice, rats, dogs and monkeys was reported by the 2006 JMPR (JMPR, 2006). Oral doses were rapidly and extensively absorbed. The pharmacokinetics and metabolic data on haloxyfop-R methyl ester (haloxyfop-P-methyl) and haloxyfop suggest that the results of studies of oral toxicity with racemic haloxyfop methyl ester, haloxyfop or haloxyfop sodium salt should be relevant to the investigation of the toxicity of haloxyfop–R methyl ester and haloxyfop-R (haloxyfop-P), as all stereoisomeric forms and esterified forms end up as the de-esterified R enantiomer.

Lactating goats

Two lactating goats were dosed with [^{14}C]phenyl ring labelled haloxyfop via gelatin capsule twice daily for 10 consecutive days at the equivalent of 16 ppm haloxyfop in the feed, i.e. approximately 0.6 mg/kg bw per day (Yackovich and Miller, 1983, GH-C 1624). The feed consumption during dosing for one goat was 1.6 kg/day and for the other was 0.80 kg/day (it had been 1.6 kg/day during the acclimatisation period). The feed was high efficiency dairy ration and alfalfa hay (1 + 1). Initial body weights were 45.5 and 39.5 kg. Milk was collected twice daily. Average milk production during the dosing period was 1.1 and 0.67 litres per milking for the two goats. Animals were slaughtered approximately 12 hours after the final dose for tissue collection.

Accountability of the administered ^{14}C was 96% and 90% for the two goats. Most of the administered dose (92% and 84%) was excreted in urine, with 1.9% and 1.5% in faeces. Milk accounted for 1.9% and 3.2% of the dose. Tissues accounted for less than 0.5% of the dose. Expired air was monitored from one goat for 10 hours, but no $^{14}CO_2$ or [^{14}C]volatiles were detected. The level of residues in tissues and milk are summarised in Table 1.

Table 1 ^{14}C radiolabel, expressed as haloxyfop, in tissues and milk from 2 lactating goats dosed with [^{14}C]phenyl ring labelled haloxyfop via gelatin capsule for 10 consecutive days at the equivalent of 16 ppm haloxyfop in the feed (Yackovich and Miller, 1983, GH-C 1624)

Tissue, Milk	^{14}C expressed as haloxyfop, mg/kg Goat #3	Goat #4
Muscle	0.02	< 0.01
Heart	0.07	0.04

Tissue, Milk	^{14}C expressed as haloxyfop, mg/kg Goat #3	Goat #4
Fat	0.06	0.11
Liver	0.45	0.31
Kidney	1.45	1.07
Milk, day 10	0.25	0.20
Milk fat (ether extracted from milk), day 10 [a]	1.2	2.8

[a] Report GH-C 1624 explains that milk fat was extracted from acidified whole milk with ether. The Appendix document (McConnell *et al.*, 1982, ADC #704) explains that whole milk was separated into milk fat and skim milk by centrifugation. The data should not be used for determining the distribution of the residue between fat and non-fat portions of the milk because of doubts about the procedure.

The ^{14}C residues in extracted milk fat were nonpolar and were susceptible to alkaline hydrolysis or lipase hydrolysis to release haloxyfop. The behaviour was consistent with haloxyfop conjugated as triacylglycerides. Residues in body fat were of the same nature as the residues in milk fat. Residues in milk reached a plateau very quickly, within 24 hours of the first dose.

The residues in kidney and liver consisted mostly of parent haloxyfop, but some may have been present as labile conjugates.

Laying hens

Four laying hens were dosed with [^{14}C]phenyl ring labelled haloxyfop via gelatin capsule for 11 consecutive days at 1.1 to 1.3 mg/kg bw/day, the equivalent of 12 ppm haloxyfop in the feed (Yackovich and Miller, 1983, GH-C 1635). Eggs were collected daily. Birds were slaughtered 24 hours after the final dose for tissue collection.

Accountability of the administered ^{14}C was high at 93–102% for the four hens, with most (81.9–89.7%) of the ^{14}C excreted in the droppings or present as gut contents (5.8–8.6%). Eggs accounted for an average of 1.6% of the ^{14}C and tissues approximately 2%. ^{14}C residue levels were highest in kidney and liver and lowest in muscle tissues (Table 2). ^{14}C residue levels were much higher in the yolk than in the whites and reached a plateau in yolks on approximately the 7th day of dosing (Figure 4).

Table 2 ^{14}C radiolabel, expressed as haloxyfop, in tissues and eggs from 4 laying hens dosed with [^{14}C]phenyl ring labelled haloxyfop via gelatin capsule for 11 consecutive days at the equivalent of 12 ppm haloxyfop in the feed (Yackovich and Miller, 1983, GH-C 1635)

Tissue, Eggs	^{14}C expressed as haloxyfop Mean residue, mg/kg	Range (4 hens), mg/kg
Light muscle	0.06	0.02–0.11
Dark muscle	0.18	0.06–0.35
Heart	0.28	0.09–0.46
Skin	0.39	0.11–0.93
Fat	0.99	0.46–2.0
Liver	1.82	1.2–2.5
Kidney	4.2	2.5–7.0
Eggs, day 10	1.05	0.74–1.24
Egg yolks, day 10	2.87	2.0–4.0
Egg whites, day 10	0.27	0.12–0.37

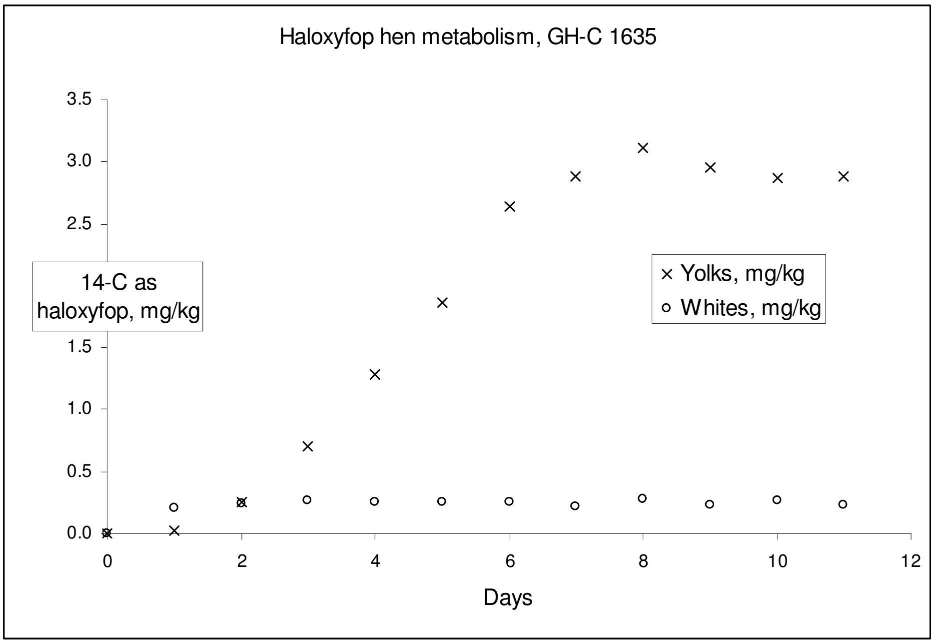


Figure 4 ^{14}C radiolabel, expressed as haloxyfop, in yolks and whites of eggs from 4 laying hens dosed with [^{14}C]phenyl ring labelled haloxyfop via gelatin capsule for 11 consecutive days at the equivalent of 12 ppm haloxyfop in the feed (Yackovich and Miller, 1983, GH-C 1635). Residues are the mean from eggs of the 4 hens

Approximately 3% of ^{14}C in egg yolks was present as free haloxyfop with a large part of the residue in the non-polar fraction. Mild alkaline hydrolysis produced haloxyfop as the single product. Lipase hydrolysis of an egg yolk extract also produced haloxyfop as the single product, demonstrating that haloxyfop was incorporated into the triacylglycerides in egg lipids.

Solvent extracts of fat, skin, liver and kidney produced varying proportions of haloxyfop, polar conjugates and non-polar conjugates. Alkaline hydrolysis of the extracts from liver, kidney and fat converted the residues almost quantitatively to a single product, haloxyfop. Most likely, parent haloxyfop was largely incorporated into lipids from which it could be readily released by hydrolysis.

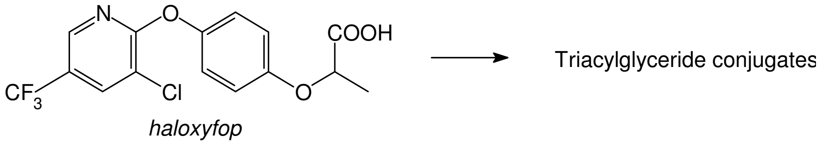


Figure 5 Haloxyfop livestock metabolism

Plant metabolism

The Meeting received plant metabolism studies with haloxyfop-butyl in cotton; haloxyfop-methyl, haloxyfop-butyl and haloxyfop-ethoxyethyl in soya beans; and haloxyfop-P-methyl in sugar beet and lettuce.

When applied to plants, the esters of haloxyfop or haloxyfop-P are broken down quickly to release free acid which is readily translocated throughout the plants. The haloxyfop (or haloxyfop-P) becomes conjugated, typically as glycosides (polar metabolites) or as triglycerides (non-polar metabolites).

Metabolism of haloxyfop butyl ester in cotton

Haloxyfop butyl ester, [^{14}C]phenyl ring labelled, was applied as an EC formulation to the foliage of 1-month old cotton plants (variety D and PL 16) at a rate equivalent to 0.56 kg ai/ha (Stafford and Miller, 1983, GH-C 1634). Lint and seeds were collected for analysis at two intervals after application, 78 and 105 days. The lint and seeds were separated and air dried. Seeds were further de-linted by a brief treatment with concentrated sulfuric acid. All remaining parts of the cotton plant after final harvest of lint and seed became 'field trash', which was also air dried.

Residue levels in seed (0.23 mg/kg) and seed coat (0.16 mg/kg) were similar for the 105 days sample. The plant components were subjected to solvent extraction and partition to separate polar and non-polar metabolites, which were further subjected to alkaline hydrolysis. Enzyme (porcine pancreatic lipase) hydrolysis was also used on non-polar metabolites. Results of the identification are summarised in Table 3. No haloxyfop butyl ester remained as a residue. Haloxyfop free acid and haloxyfop conjugates were major parts of the residue in cotton seed lint and trash.

The ^{14}C in the oil was associated with the triglycerides. Lipase hydrolysis and alkali hydrolysis released 91–99.8% of the ^{14}C as haloxyfop, suggesting that the non-polar residues were triglyceride esters of haloxyfop.

Table 3 ^{14}C radiolabel, expressed as haloxyfop, in cotton seed, oil, lint and dry plant (field trash) sampled 78 and 105 days from cotton plants after treatment with [^{14}C]haloxyfop butyl ester at a rate equivalent to 0.56 kg ai/ha (Stafford and Miller, 1983, GH-C 1634). Identified components of the residue are expressed as percentage of the totals

	Cotton seed (seed + seed coat)		Oil		Lint		Field trash
	78 days	105 days	78 days	105 days	78 days	105 days	105 days
Total ^{14}C, mg/kg	0.78	0.20	1.12	0.38	0.19	0.04	1.09
Haloxyfop-butyl ester	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Haloxyfop free acid	41.5%	31.9%	0.0%	0.0%	76.0%	75.5%	39.1%
Haloxyfop conjugates	54.6%	66.4%	100%	100%	10.0%	10.1%	55.2%
Non-extractable	3.9%	1.7%	0.0%	0.0%	14.0%	14.4%	5.7%

Metabolism of haloxyfop methyl, n-butyl and ethoxyethyl esters in soya beans

The mature second and developing third trifoliate leaves of 20-days old soya bean plants (Corsoy) were treated with [^{14}C]labelled haloxyfop in the form of an ester (Bauriedel and Miller, 1982, GH-C 1507). Haloxyfop methyl ester was available as [^{14}C]labelled in the phenyl ring or in the 3,6 positions of the pyridyl ring. The *n*-butyl and ethoxyethyl esters were [^{14}C]labelled in the phenyl ring. The dose for each plant was 0.2 mg haloxyfop acid equivalent. Plants were sampled 2, 4 and 8 days after treatment. Two samples were taken from each plant: treated leaves and remainder of above-ground plant. Total ^{14}C was measured and the constituents of the residue were characterised or identified.Concentrations in the leaves were not reported. The composition of the residues in treated and untreated plant is summarised in Table 4.

Esters were rapidly hydrolysed to haloxyfop. Even after 2 days, little of the applied ester remained in the treated leaves. Applied ester did not appear in untreated portions of the plant. In the treated leaves, polar metabolites increased as a percentage of the residue with time after treatment. The two label positions in the methyl ester apparently produced the same residue behaviour, suggesting that the molecule remained intact. Unconjugated haloxyfop was the main part of the residue in untreated parts of the plant, irrespective of the ester used for treatment.

Mild alkaline hydrolysis of the polar translocated residue released haloxyfop, demonstrating that at least part of the polar fraction consists of haloxyfop conjugates.

Table 4 Nature of the residue in treated and untreated portions of soya bean plants after application of [^{14}C]labelled esters of haloxyfop (Bauriedel and Miller, 1982, GH-C 1507). Residues are expressed as percentages of the extractable residue.

	Haloxyfop methyl [^{14}C]phenyl	Haloxyfop methyl [^{14}C]3,6-pyridyl	Haloxyfop n-butyl [^{14}C]phenyl	Haloxyfop ethoxyethyl [^{14}C]phenyl
TREATED LEAVES 2 days after treatment				
Polar metabolites	34%	30%	29%	25%
Haloxyfop	66%	66%	60%	58%
Haloxyfop ester	0%	4%	11%	17%
4 days after treatment				
Polar metabolites	52%	49%	51%	52%
Haloxyfop	48%	50%	43%	39%
Haloxyfop ester	0%	1%	6%	9%
8 days after treatment				
Polar metabolites	58%	65%	62%	65%
Haloxyfop	40%	34%	38%	34%
Haloxyfop ester	2%	1%	0%	1%
UNTREATED PLANT 2 days after treatment				
Polar metabolites	17%	20%	18%	22%
Haloxyfop	83%	80%	82%	78%
Haloxyfop ester	0%	0%	0%	0%
4 days after treatment				
Polar metabolites	28%	30%	27%	25%
Haloxyfop	72%	70%	73%	75%
Haloxyfop ester	0%	0%	0%	0%
8 days after treatment				
Polar metabolites	36%	38%	39%	35%
Haloxyfop	64%	62%	61%	65%
Haloxyfop ester	0%	0%	0%	0%

Soya bean plants (Corsoy) were treated with [^{14}C]haloxyfop butyl ester at two rates (0.28 and 0.56 kg ai/ha)and at two stages of plant growth (Yackovich and Miller,1983, GH-C 1618). The early treatment was 90 days before harvest and the late treatment was 60 days before harvest. Both [^{14}C]phenyl ring labelled and [^{14}C]3,6-pyridyl labelled haloxyfop were used.

The plant samples were extracted with acetonitrile/water mixtures. Beans were first extracted with hexane to remove lipids before acetonitrile/water extraction. Methanol was routinely avoided for sample extraction, sample solvent and HPLC mobile phase because haloxyfop is very easily methylated in methanol solutions. The distribution and nature of the ^{14}C are summarised in Table 5.

Table 5 Residue levels and nature of the residue in forage, beans and straw of soya bean plants after application of [^{14}C]labelled haloxyfop butyl ester (Yackovich and Miller,1983, GH-C 1618). Residues are expressed as percentages of the total residue in the forage, beans or straw

	Days after treatment	Application rate 0.28 kg ai/ha	Application rate 0.56 kg ai/ha	
		[^{14}C]phenyl ring label	[^{14}C]phenyl ring label	[^{14}C]3,6-pyridyl label
Forage	15	10.5 mg/kg	14.9 mg/kg	
Haloxyfop		30%	27%	
Polar conjugates		66%	65%	
Forage, new growth	15		5.9 mg/kg	
Haloxyfop			42%	
Polar conjugates			57%	

	Days after treatment	Application rate 0.28 kg ai/ha	Application rate 0.56 kg ai/ha	
		[^{14}C]phenyl ring label	[^{14}C]phenyl ring label	[^{14}C]3,6-pyridyl label
Beans	89	0.8 mg/kg	1.3 mg/kg	
Haloxyfop		57%	58%	
Polar conjugates		17%	17%	
Non-polar conjugates		18%	19%	
Beans	61	3.1 mg/kg	5.8 mg/kg	4.3 mg/kg
Haloxyfop		57%	59%	58%
Polar conjugates		17%	18%	20%
Non-polar conjugates		18%	18%	17%
Straw	89	1.0 mg/kg	1.6 mg/kg	
Haloxyfop		65%	62%	
Polar conjugates		27%	32%	
Straw	61	1.3 mg/kg	3.0 mg/kg	2.1 mg/kg
Haloxyfop		66%	64%	60%
Polar conjugates		24%	28%	29%

The parallel behaviour of the phenyl ring label and 3,6-pyridyl label showed that the molecule remained intact and essentially all of the residue contained both the phenyl and pyridyl rings. HPLC analysis comparisons of residue in treated foliage and new growth showed that the butyl ester was not translocated. The free acid or haloxyfop conjugates were readily translocated.

Alkaline and lipase hydrolysis of the non-polar residue from the beans suggested that haloxyfop was incorporated into the oil triglycerides. Most of the polar conjugates also produced haloxyfop on hydrolysis.

Metabolism of haloxyfop-P-methyl in sugar beet

Sugar beet plants (cultivar Wildcat), at the 6-leaf growth stage, in field plots were foliar sprayed with [^{14}C]3,5-pyridyl haloxyfop-P-methyl formulated as a 108 g ai/L EC at a rate of 0.113 kg ai/ha (Chapleo *et al*., 2002, 20625). Plants were harvested on the day of application, 28 days later and at maturity (92 days after application). In this study, haloxyfop-P-methyl was described as the active ingredient.

Residue levels (TRR, expressed as haloxyfop-P-methyl) were much lower in the roots than the shoots and decreased with time after treatment (Table 6).

Table 6 Residue levels and nature of the residue in roots and tops of sugar beet after application of [^{14}C]3,5-pyridyl haloxyfop-P-methyl at a rate of 0.113 kg ai/ha (Chapleo *et al.*, 2002, 20625)

Residue	Day 0 Plants	Day 28 Shoots	Day 28 Roots	Maturity Shoots	Maturity Roots
Total ^{14}C, as haloxyfop-P-methyl (TRR)	6.6 mg/kg	0.18 mg/kg	0.083 mg/kg	0.079 mg/kg	0.019 mg/kg
Unextracted, % TRR	1.1%	8.1%	5.5%	12%	20%
Extractable residue, % TRR	99%	95%	97%	79%	87%
Haloxyfop-P-methyl, % TRR	99%	n.d	n.d	0.6%	n.d
Haloxyfop-P acid, % TRR	n.d	47%	74%	33%	31%
Haloxyfop-P conjugate 1, % TRR	n.d	17%	n.d	24%	19%
Haloxyfop-P glycoside conjugate 1, % TRR	–	18%	17%	14%	20%
Haloxyfop-P glycoside conjugate 2, % TRR	n.d	8.6%	n.d	n.d	n.d
Haloxyfop-P conjugate 2, % TRR	n.d	3.5%	n.d	4.1%	n.d

n.d = not detected

Apart from one treatment day, haloxyfop-P acid and its conjugates were major parts of the residue. The residue readily translocated to the roots.

Metabolism of haloxyfop-P-methyl in lettuce

Lettuce plants (cultivar Set), at approximately ¼ of their final size, in field plots were foliar sprayed with [^{14}C]3,5-pyridyl haloxyfop-P-methyl formulated as a 108 g ai/L EC at a rate of 0.106 kg ai/ha (Chapleo and White, 2002, 20626). Lettuces were harvested on the day of application, 14 days later and at maturity (29 days after treatment). In this study, haloxyfop-P-methyl was described as the active ingredient. Results are summarised in Table 7.

Higher residues were found in outer leaves than on inner leaves. Haloxyfop-P-methyl had disappeared by day 14. A large part of the residues in inner and outer leaves consisted of haloxyfop-P in free or conjugated form. Haloxyfop-P was readily translocated to the inner (unsprayed) leaves of the lettuces.

Table 7 Residue levels and nature of the residue in inner and outer leaves of lettuces after application of [^{14}C]3,5-pyridyl haloxyfop-P-methyl at a rate of 0.106 kg ai/ha (Chapleo and White, 2002, 20626)

	Day 0	Day 14 [a]			Maturity [a]		
	Foliage	Outer leaves	Inner leaves	Whole lettuce	Outer leaves	Inner leaves	Whole lettuce
Total ^{14}C, as haloxyfop-P-methyl (TRR), mg/kg	3.37	0.40	0.16	0.32	0.16	0.048	0.093
Unextracted, % TRR	0.8%	8.7%	1.8%		10.3%	0.6%	
Extractable residue, % TRR	104%	93%	98%		92%	101%	
Haloxyfop-P-methyl, % TRR	72%	n.d	n.d		n.d	n.d	
Haloxyfopn.dP acid, % TRR	31%	55%	97%	62%	38%	93%	55%
Haloxyfop-P conjugate 1, % TRR	n.d	5.8%	0.6%	4.9%	24%	1.2%	17%
Haloxyfop-P glycoside conjugate 1, % TRR	n.d	4.1%	n.d	3.4%	6.9%	n.d	5.4%
Haloxyfop-P glycoside conjugate 2, % TRR	n.d	29%	0.8%	24%	23%	2.7%	17%
Haloxyfop-P conjugate 2, % TRR	n.d	n.d	n.d		0.4%	1.5%	1.1%

n.d = not detected.

[a] %TRR values for whole lettuce are calculated from the measured values on outer and inner leaves.

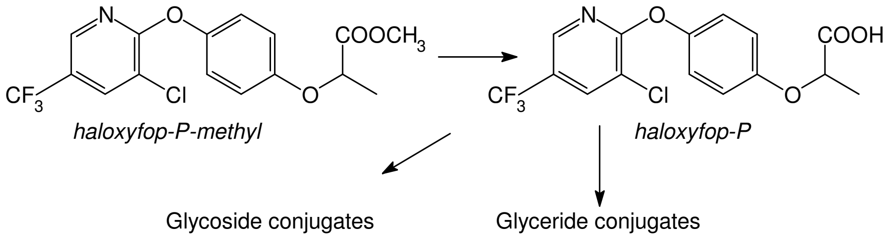


Figure 6 Proposed metabolic pathway for haloxyfop-P-methyl in plants

Environmental fate in soil

The 2003 JMPR (JMPR, 2003) explained the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For haloxyfop-P-methyl, supervised residue trials data are available for sugar beet, which means that aerobic degradation in soil is relevant, as well as the normal requirements for hydrolysis and rotational crop studies. The 2003 report does not mention soil photolysis studies; however, such studies should be relevant for the same reasons as for aerobic soil degradation—nature and magnitude of residues in soil.

The Meeting received information on soil aerobic metabolism and soil photolysis properties of haloxyfop-P-methyl. Studies were also received on the behaviour of [^{14}C]labelled haloxyfop-butyl in a rotational crop situation and haloxyfop-methyl in an unconfined rotational crop situation.

Haloxyfop residues are generally not persistent in soils. Haloxyfop residues in soils resulting from recommended uses should not contribute to the residues in root vegetables or to residues in succeeding crops.

Soil metabolism

Hale and Trigg (1994, GHE-P-3594) dosed four soils with: [^{14}C]3,4 pyridyl haloxyfop-P-methyl and maintained aerobic conditions for 182 days. The methyl ester was converted to haloxyfop-P very quickly. Haloxyfop-P was also readily metabolised, with half-lives in the range of 8–20 days. After 182 days, 6.5–24% of the dose was mineralised and 23–35% was unextracted.

Goodyear (2001, 295/100) studied the aerobic fate of [^{14}C]haloxyfop-P-methyl in four soils for 120 or 268 days. Parent haloxyfop-P-methyl typically disappeared with a half-life of approximately 0.5 days. The first metabolite was haloxyfop-P acid, which mostly disappeared with half-lives in the range of 9–21 days, but in subsoils with low organic carbon its disappearance half-lives were 28 and 129 days. After 268 days, mineralization and unextracted residues accounted for approximately 6–33% and 28–46% of the dose, respectively. The metabolites 'phenol metab', 'pyridinone metab' and 'pyridinol metab' were consistently produced, with the 'pyridinone metab' apparently the most persistent. See Figure 7 for structures of these metabolites.

Haloxyfop-P-methyl was hydrolysed just as quickly in a sterile soil as in a fresh soil, demonstrating that the methyl ester is chemically labile. In the sterile soil, haloxyfop-P was persistent and very little of the ^{14}C was mineralised.

Aerobic soil metabolism		Ref: Hale and Trigg, 1994, GHE-P-3594
Test material: [^{14}C]3,4 pyridyl haloxyfop-P-methyl		Dose rate: 0.108 mg ai/kg
Duration: 182 days	Temp: 20 °C	Moisture: 40% max water holding capacity
Soil: sandy clayey loam	pH: 8.3	Organic matter: 3.8%
Half-life haloxyfop-P-methyl: < 1 day		^{14}C accountability: 87–100%
% haloxyfop-P-methyl remaining, day 182 = 0.52% of dose		% mineralization, day 182 = 6.5% of dose
Half-life haloxyfop-P: 8.8 days		% unextracted, day 182 = 35% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	73%	1
Phenol metab	12%	3
Pyridinol metab	37%	91

Aerobic soil metabolism		Ref: Hale and Trigg, 1994, GHE-P-3594
Test material: [^{14}C]3,4 pyridyl haloxyfop-P-methyl		Dose rate: 0.108 mg ai/kg
Duration: 182 days	Temp: 20 °C	Moisture: 40% max water holding capacity
Soil: loamy sand	pH: 8.2	Organic matter: 1.9%
Half-life haloxyfop-P-methyl: < 1 day		^{14}C accountability: 86–103%
% haloxyfop-P-methyl remaining, day 182 = 0.60% of dose		% mineralization, day 182 = 11% of dose
Half-life haloxyfop-P: 9.1 days		% unextracted, day 182 = 30% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	91%	1
Phenol metab	8.3%	7
Pyridinol metab	39%	91

Aerobic soil metabolism		Ref: Hale and Trigg, 1994, GHE-P-3594
Test material: [^{14}C]3,4 pyridyl haloxyfop-P-methyl		Dose rate: 0.108 mg ai/kg
Duration: 182 days	Temp: 20 °C	Moisture: 40% max water holding capacity
Soil: loamy clay	pH: 7.8	Organic matter: 6.3%
Half-life haloxyfop-P-methyl: < 1 day		^{14}C accountability: 86–102%
% haloxyfop-P-methyl remaining, day 182 = 0.39% of dose		% mineralization, day 182 = 7.2% of dose
Half-life haloxyfop-P: 17 days		% unextracted, day 182 = 30% of dose

Metabolites	Max (% of dose)	Day

Haloxyfop-P	82%	1
Phenol metab	12.6%	14
Pyridinol metab	53%	91

Aerobic soil metabolism
Ref: Hale and Trigg, 1994, GHE-P-3594
Test material: [^{14}C]3,4 pyridyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg
Duration: 182 days
Temp: 20 °C
Moisture: 40% max water holding capacity
Soil: slightly loamy sand
pH: 6.8
Organic matter: 3.5%
Half-life haloxyfop-P-methyl: < 1 day
^{14}C accountability: 84–104%
% haloxyfop-P-methyl remaining, day 182 = 0.61% of dose
% mineralization, day 182 = 24% of dose
Half-life haloxyfop-P: 20 days
% unextracted, day 182 = 23% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	84%	1
Phenol metab	7.0%	14
Pyridinol metab	36%	91

Aerobic soil metabolism
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]3,5 pyridyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 268 days
Temp: 20 °C
Moisture:?
Soil: Marcham sandy loam
pH: 7.6
Organic matter: 1.9%
Half-life haloxyfop-P-methyl: 0.5 days
^{14}C accountability: 89–99%
% haloxyfop-P-methyl remaining, day 268, not detected
% mineralization, day 268 = 23% of dose
Half-life haloxyfop-P: 9.4 days
% unextracted, day 268 = 29% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	59%	2
Phenol metab	7.2%	7
Pyridinone metab	11%	120
Pyridinol metab	29%	59

Aerobic soil metabolism
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]phenyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 120 days
Temp: 20 °C
Moisture:?
Soil: Marcham sandy loam
pH: 7.6
Organic matter: 1.9%
Half-life haloxyfop-P-methyl: 0.6 days
^{14}C accountability: 80–99%
% haloxyfop-P-methyl remaining, day 120:0.6%
% mineralization, day 120 = 33% of dose
Half-life haloxyfop-P: 12 days
% unextracted, day 120 = 46% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	53%	2
Phenol metab	7.4%	7

Sterile soil—aerobic conditions
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]phenyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 120 days
Temp: 20 °C
Moisture: ?
Soil: Marcham sandy loam, sterilised by gamma irradiation
pH: 7.6
Organic matter: 1.9%
Half-life haloxyfop-P-methyl: 0.5 days
^{14}C accountability: 90–99%
% haloxyfop-P-methyl remaining, day 120: 1.4%
% mineralization, day 120 = 0.5% of dose
Half-life haloxyfop-P: persistent
% unextracted, day 120 = 15% of dose

Degradation products	Max (% of dose)	Day
Haloxyfop-P	85%	30
Phenol metab	1.2%	2

Aerobic soil metabolism
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]3,5 pyridyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 268 days
Temp: 10 °C
Moisture: ?
Soil: Marcham sandy loam
pH: 7.6
Organic matter: 1.9%
Half-life haloxyfop-P-methyl: 0.5 days
^{14}C accountability: 88-99%
% haloxyfop-P-methyl remaining, day 268: 2.2%
% mineralization, day 268 = 6.1% of dose
Half-life haloxyfop-P: 21 days
% unextracted, day 268 = 30% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	70%	2
Phenol metab	6.3%	14
Pyridinone metab	11.5%	268
Pyridinol metab	35%	90

Aerobic soil metabolism
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]3,5 pyridyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 268 days
Temp: 20 °C
Moisture: ?
Soil: Borstel (0-30 cm), sandy loam
pH: 6.4
Organic matter: 2.6%
Half-life haloxyfop-P-methyl: 0.5 days
^{14}C accountability: 90–98%
% haloxyfop-P-methyl remaining, day 268: not detected
% mineralization, day 268 = 22% of dose
Half-life haloxyfop-P: 12 days
% unextracted, day 268 = 30% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	72%	2
Phenol metab	7.5%	7
Pyridinone metab	7.7%	120
Pyridinol metab	37%	90

Aerobic soil metabolism
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]3,5 pyridyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 268 days
Temp: 20 °C
Moisture: ?
Soil: Borstel (30-60 cm), sandy loam
pH: 6.5
Organic matter: 1.2%
Half-life haloxyfop-P-methyl: 0.7 days
^{14}C accountability: 93–99%
% haloxyfop-P-methyl remaining, day 268: 0.6%
% mineralization, day 268 = 26% of dose
Half-life haloxyfop-P: 28 days
% unextracted, day 268 = 28% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	68%	2
Phenol metab	9.3%	14
Pyridinone metab	17%	120
Pyridinol metab	29%	59

Aerobic soil metabolism
Ref: Goodyear, 2001, 29^{e}100
Test material: [^{14}C]3,5 pyridyl haloxyfop-P-methyl
Dose rate: 0.108 mg ai/kg soil
Duration: 268 days
Temp: 20 °C
Moisture: ?
Soil: Borstel (60-100 cm), sand
pH: 6.4
Organic matter: 0.2%
Half-life haloxyfop-P-methyl: 0.6 days
^{14}C accountability: 90–102%
% haloxyfop-P-methyl remaining, day 268: 2.7%
% mineralization, day 268 = 17% of dose
Half-life haloxyfop-P: 129 days
% unextracted, day 268 = 22% of dose

Metabolites	Max (% of dose)	Day
Haloxyfop-P	86%	14
Phenol metab	4.0%	120
Pyridinone metab	9.5%	268
Pyridinol metab	22%	181

Cook (2002, 010107) studied the photolysis of [^{14}C]haloxyfop-P-methyl on the surface of a sandy clay loam. Photolysis had negligible effect compared with hydrolysis and metabolism. Degradation rates in the dark controls and the photolysis samples were similar.

Soil photolysis
Ref: Cook, 2002, 010107
Test material: [^{14}C]3,5 pyridyl haloxyfop-P-methyl
Dose rate: 3.2 mg ai/kg soil
Xenon lamp simulating average summer sunlight at 40 ºN latitude
Duration: 353 hours
Temp: 20 °C
Soil conditions: air dried, 0.2 cm layer
Soil: sandy clay loam
pH: 7.9
Organic carbon: 2.2%
Half-life haloxyfop-P-methyl: < 22 hours (dark control < 22 hours)
^{14}C accountability: 96–105%
% haloxyfop-P-methyl remaining after 353 hours = 9.1% of dose
(dark control 1.6%)

Metabolites and photolysis products	Max (% of dose)	Hours	
Haloxyfop-P	83%	209	75% dark control
Phenol metab	5.4%	118	8.3% dark control

Soil photolysis
Ref: Cook, 2002, 010107
Test material: [^{14}C]phenyl haloxyfop-P-methyl
Dose rate: 3.2 mg ai/kg soil
Xenon lamp simulating average summer sunlight at 40 ºN latitude
Duration: 353 hours
Temp: 20 °C
Soil conditions: air dried, 0.2 cm layer
Soil: sandy clay loam
pH: 7.9
Organic carbon: 2.2%

Half-life haloxyfop-P-methyl: < 22 hours (dark control < 22 hours)		^{14}C accountability: 91–98%	
% haloxyfop-P-methyl remaining after 353 hours = 8.1% of dose		(dark control 0.8%, after 209 hours)	
Metabolites and photolysis products	Max (% of dose)	Hours	
Haloxyfop-P	83%	22	87% dark control
Phenol metab	4.2%	118	9.9% dark control

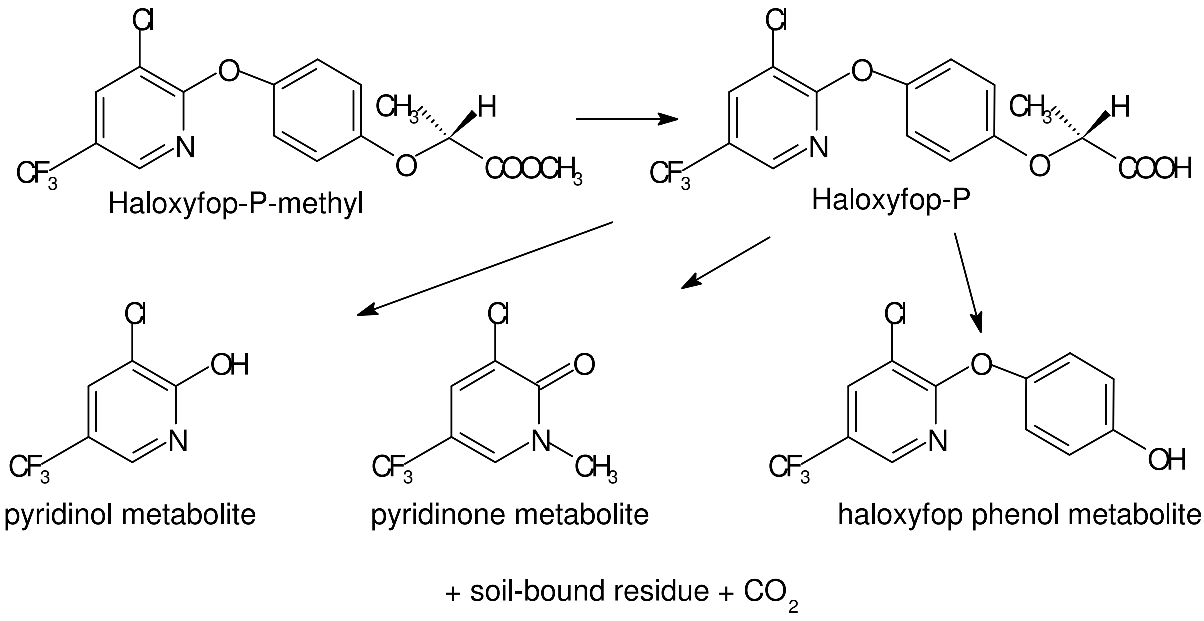


Figure 7 Soil metabolism of haloxyfop-P-methyl

Rotational crops

In a confined rotational crop study, uniformly phenyl-ring-labelled haloxyfop butyl ester was formulated as an EC and applied at 0.56 kg ai/ha to a plot of sandy loam soil in Michigan USA in the late spring (Yackovich and Miller, 1983, GH-C 1598). Thirty days later, the plot was weeded and portions of the plot were sown with spring wheat (var *Eureka*), soya beans (var *Corsoy*), leaf lettuce (var *Black Seeded Simpson*), carrots (var *Denver's half-long*) and turnips (var *Purple Top White Globe*). Crops were harvested at various intervals after sowing: lettuce 49 days, soya bean forage 56 days, turnips 64 days, carrots 124 days, wheat 110 days and soya beans 145 days.

Samples containing high percentages of water were freeze dried and dry tissues were milled ready for combustion to determine the ^{14}C content.

The ^{14}C content of the plant tissues was calculated as haloxyfop and expressed on fresh weight: lettuce 0.01 mg/kg, turnip foliage < 0.01 mg/kg, turnip root < 0.01 mg/kg, wheat grain 0.01 mg/kg, wheat straw 0.02 mg/kg, soya bean forage 0.07 mg/kg, soya bean grain < 0.01 mg/kg, soya bean straw 0.01 mg/kg, carrot foliage < 0.01 mg/kg and carrot root < 0.01 mg/kg.

Attempts at isolation and identification of the residues were not successful. The study demonstrated that very little haloxyfop residue would transfer to rotational crops, at least for conditions similar to those of the study.

In an unconfined rotational crop study, Bjerke *et al.,* (1985, GH-C 1758) applied a formulation of haloxyfop-methyl at 0.28 kg ai/ha to plots of soya beans and at 0.56 kg ai/ha to plots of cotton. Approximately 30 and 120 days after treatment, rotational crops of lettuce, sugar beets and wheat were sown into the plots. The crops were grown to maturity. Samples of mature crops and wheat forage were taken for residue analysis. The data are summarised in Table 8.

Residues generally did not occur in the rotational crops at levels exceeding LOQs (0.01 and 0.02 mg/kg). Haloxyfop residues of 0.01–0.02 mg/kg were detected in wheat green forage in two trials, but the results were confounded in one case by residues at the LOQ (0.01 mg/kg) in a sample from the control plot and in the other case by detection of apparent residues just below the LOQ in samples from the control plot.

Table 8 Haloxyfop residues in rotational crops resulting from treatments on the first crop

First crop, country, year, ref.	Application				Rotational crop	TSI [a] days	THI [b] days	Sample	Residue, haloxyfop mg/kg
	Compound	Form	No	kg ai/ha					
Soya beans, USA (MI), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.28	lettuce (G-240 Montello)	25	64	lettuce heads	< 0.01 (4)
Cotton, USA (MS), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	lettuce (Romaine)	34	99	lettuce heads	< 0.01 (4)
Cotton, USA (CA), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	lettuce (Royal Oak Leaf)	31	144	lettuce heads	< 0.01 (4)
Cotton, USA (CA), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	lettuce (Oak Leaf)	112	372	lettuce heads	< 0.01 (4)
Soya beans, USA (MI), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.28	sugar beets (Monitor Sugar Lot 260-201-2)	25	119 119	beet tops beet roots	< 0.01 (4) < 0.01 (4)
Cotton, USA (MS), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	sugar beets (US-H-9)	32	139 139	beet tops beet roots	< 0.01 (4) < 0.01 (4)
Soya beans, USA (IL), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.28	sugar beets (Monitor Sugar Lot 260-201-2)	31	128 128	beet tops beet roots	< 0.01 (4) < 0.01 (4)
Cotton, USA (CA), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	sugar beets (US-H-9)	123	426 426	beet tops beet roots	< 0.01 (4) < 0.01 (4)
Soya beans, USA (MI), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.28	wheat (West Bend Aim)	25	110	green forage	0.01 0.01 < 0.01 (2)
Soya beans, USA (MI), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.28	wheat (Frankenmuth)	92	391 391	grain straw	< 0.01 (4) < 0.02 (4)
Cotton, USA (MS), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	wheat (Coher 747)	34	104	green forage	0.02 < 0.01 (3) c 0.01 < 0.01 (3)
Cotton, USA (MS), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	wheat (Coher 747)	148	408	grain straw	< 0.01 (4) < 0.02 (4)

First crop, country, year, ref.	Application				Rotational crop	TSI [a]	THI [b]	Sample	Residue, haloxyfop mg/kg
	Compound	Form	No	kg ai/ha		days	days		
Soya beans, USA (IL), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.28	wheat (Centurk)	128	415	grain straw	< 0.01 (4) < 0.02 (4)
Cotton, USA (CA), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	wheat (Anza)	31	350	grain straw	< 0.01 (4) < 0.02 (4)
Cotton, USA (CA), 1983 GHC 1758	haloxyfop-methyl	EC	1	0.56	wheat (Anza)	123	353	grain straw	< 0.01 (4) < 0.02 (4)

[a] TSI: interval between treatment on first crop and sowing of rotation crop, days

[b] THI: interval between treatment on first crop and sampling or harvest of rotation crop, days

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of haloxyfop in animal and plant matrices.

The methods rely on an initial extraction and hydrolysis step, usually with methanolic NaOH to release haloxyfop from conjugates. After solvent partition cleanup, the haloxyfop is methylated or butylated ready for GC analysis or further cleanup before the GC analysis. Typically, haloxyfop residues can be measured in most matrices to an LOQ of 0.01–0.05 mg/kg.

None of the methods separates the haloxyfop enantiomers. The methods effectively measure 'total' haloxyfop present as acid, salts, esters and conjugates (esters with natural compounds).

Haloxyfop residues are not suitable for analysis by multiresidue methods because the extraction step is typically also a base-hydrolysis step designed to release haloxyfop from non-polar and polar conjugates found in animal and plant tissues. Such an extraction-hydrolysis step is not suitable for many other pesticides.

Animal commodities

Bovine tissues (Kutschinski, 1984, ACR 84.1)

Analyte:	haloxyfop	GLC-ECD	Method ACR 84.1
LOQ:	0.01 mg/kg.		
Description	Muscle, liver and kidney tissues are homogenised and extracted with methanolic sodium hydroxide. The extract is cleaned up by solvent partition between ether and aqueous solutions (acid and alkaline) and then by C-18 column chromatography. The residue is methylated with BF_3-methanol and further cleaned up on a small alumina column. The eluate, in benzene-isooctane, is analysed by GLC-ECD. Rendered fat is dissolved in benzene and hydrolysed with ethanolic sodium hydroxide for 2 hours at 50 °C to release conjugates. The hydrolysate is cleaned up by solvent partitions between benzene and aqueous solutions (acid and alkaline), followed by a passage through a silica gel column. The eluted residue is ready for methylation, further cleanup and analysis as for the other tissues.		

Milk, cream (Gardner RC, 1988, ACR 84.6)

Analyte:	haloxyfop	GLC-ECD	Method ACR 84.6R
LOQ:	0.01 mg/kg for milk. 0.02 mg/kg for cream.		

Description Haloxyfop residues are extracted from milk or cream with diethyl ether. The ether is evaporated and the residue is hydrolysed in benzene-KOH-ethanol at 50 °C to release conjugates. The solution is diluted with water, washed with benzene, then acidified and the residues extracted into benzene, which is washed with water. The benzene solution is applied to a silica gel column and the haloxyfop residue is eluted with acetic acid in methylene chloride. After evaporation of the solvent the residue is methylated with BF_3-methanol and further cleaned up on a silica gel column, before GLC-ECD analysis.

Animal tissues, eggs, fat, milk (Yeh *et al.,* 2001, GRM 01 09)

Analyte: haloxyfop LC-MS-MS Method GRM 01.09

LOQ: 0.01 mg/kg.

Description Kidney, liver and muscle tissue. Tissues are frozen with liquid nitrogen and then ground or chopped with a hammer mill before homogenization with methanolic sodium hydroxide. The mixture is then centrifuged and an aliquot of the supernatant liquid is mixed with water + hydrochloric acid and cleaned up through a C_{18} SPE column. Haloxyfop residues are eluted from the column with an acetonitrile-ethyl acetate-acetic acid mixture. The solvent is evaporated and the residue is taken up in water-acetonitrile containing deuterium-labelled haloxyfop as an internal standard for LC-MS-MS analysis.

Eggs. Egg samples are shaken with ethanolic KOH + toluene and the mixture is centrifuged. An aliquot of the extract is then shaken with water and the mixture centrifuged. An aliquot of the aqueous layer, after addition of dilute hydrochloric, is ready for The SPE cleanup and remainder of the procedure described for kidney, liver and muscle tissues.

Fat tissue. The fat sample is mixed with ethanolic KOH and toluene and maintained at 50 °C for 2 hours to hydrolyse lipid conjugates. The extract is subject to partition cleanup followed by the SPE cleanup and remainder of the procedure described for kidney, liver and muscle tissues.

Milk. An aliquot of milk is mixed with ethanolic KOH and toluene and maintained at 50 °C for 30 minutes to hydrolyse lipid conjugates. The extract is subject to partition cleanup followed by the SPE cleanup and remainder of the procedure described for kidney, liver and muscle tissues.

Muscle, liver, kidney (Hoogenboom, 2001, NCRL 9901)

Analyte: haloxyfop GC-ECD Method NCRL-PHT-01

LOQ: 0.01 mg/kg.

Description Muscle, liver and kidney samples are homogenised and extracted with methanolic sodium hydroxide. The extract is cleaned up by solvent partition between ether and aqueous solutions (acid and alkaline). After methylation, the residue is further cleaned up by gel permeation chromatography. The eluate is analysed by GLC-ECD.

Fat (Hoogenboom, 2001, NCRL 9901)

Analyte: haloxyfop GC-ECD Method NCRL-PHF-01

LOQ: 0.01 mg/kg.

Description Rendered fat is dissolved in toluene. Acetonitrile is added and the mixture is cooled to precipitate the fat. The acetonitrile is evaporated and the residue is methylated and then further cleaned up by gel permeation chromatography (GPC). The fraction from GPC is then cleaned up using solid phase extraction with an amino cartridge. The solution is then ready for analysis by GLC-ECD.

Note: Method NCRL-PHF-01 has no hydrolysis step, so is unlikely to recover haloxyfop quantitatively from glyceride conjugates.

Milk (Hoogenboom, 2001, NCRL 9901)

Analyte: haloxyfop GC-ECD Method NCRL-PHM-01

LOQ: 0.01 mg/kg.

Description Milk is acidified and extracted with ether. The concentrated extract is dissolved in toluene, acetonitrile is added and the mixture is cooled to precipitate out the fat. The acetonitrile is evaporated and the residue is methylated and then further cleaned up by gel permeation chromatography (GPC). The fraction from GPC is then cleaned up using solid phase extraction with an amino cartridge. The solution is then ready for analysis by GLC-ECD.

Note: Method NCRL-PHM-01 has no hydrolysis step, so is unlikely to recover haloxyfop quantitatively from glyceride conjugates.

Crop commodities

Soya bean seed and processed commodities (Gardner, 1983, GH-C 1625)

Analyte: haloxyfop GLC-ECD Method ACR 83.1

LOQ: 0.05 mg/kg.

Description Residues are extracted from the substrate with methanolic NaOH by shaking overnight. The residue is extracted from aqueous solution into benzene and then adsorbed on to silica gel and eluted with aqueous NaOH. The eluate is acidified and the residue is partitioned into ether where it is methylated with diazomethane. The methylated residue is further cleaned up on an acidic alumina column ready for GLC-ECD analysis.

Oilseed rape seed (Yon *et al*., 1983, ERC 83.17)

Analyte: haloxyfop GLC-ECD Method ERC 83.17

LOQ: 0.05 mg/kg.

Description Homogenised rape seed is extracted with methanolic NaOH. The mixture is shaken for 2 hours and allowed to stand for at least 4 hours, or overnight. An aliquot of the extract is acidified and the residues are partitioned into diethyl ether. Residues are cleaned up by solvent partition between ether and aqueous solutions (acid and alkaline) before being butylated with a sulphuric acid *n*-butanol reagent. After further cleanup on a Florisil column, the residue is analysed by GLC-ECD.

Sunflower seed (Yon *et al.,* 1983, ERC 83.18)

Analyte: haloxyfop GLC-ECD Method ERC 83.18

LOQ: 0.05 mg/kg.

Description Homogenised sunflower seed is blended and extracted with methanolic NaOH. The mixture is shaken for 2 hours and allowed to stand for at least 4 hours, or preferably overnight. An aliquot of the supernatant from centrifuged extract is acidified and the residues are partitioned into diethyl ether. Residues are cleaned up by solvent partition between ether and aqueous solutions (acid and alkaline) before being butylated with a sulphuric acid *n*-butanol reagent. After further cleanup on an activated Florisil column, the residue is analysed by GLC-ECD.

Oilseed rape cake Yon, 1983, ERC 83.24)

Analyte: haloxyfop GLC-ECD Method ERC 83.23

LOQ: 0.05 mg/kg.

Description Similar to ERC 83.17

Oilseed rape oil Yon, 1983, ERC 83.20)

Analyte: haloxyfop GLC-ECD Method ERC 83.20

LOQ: 0.01 mg/kg.

Description Rape oil is dissolved in diethyl ether and washed with aqueous acid. The ether is evaporated and the oil is reacted with ethanolic KOH in toluene at 50 ˚C for 30 minutes. The aqueous mixture is acidified and residues are partitioned into dichloromethane. Residues are cleaned up by solvent partition between dichloromethane and aqueous solutions (acid and alkaline) before being butylated with a sulphuric acid *n*-butanol reagent. After further cleanup on a Florisil column, the residue is analysed by GLC-ECD.

Coffee beans, sunflower seeds (Doege, 1983, BRC 83.1)

Analyte: haloxyfop GLC-ECD Method BRC 83.1

LOQ: 0.02 mg/kg coffee beans, 0.05 mg/kg sunflower seed.

Description Ground coffee beans or sunflower seeds are homogenized and shaken with methanolic NaOH for at least 2 hours. An aliquot is then diluted with water and sodium chloride, acidified with sulphuric acid and extracted with benzene. The benzene solution is cleaned up on a silica Sep-Pak cartridge, with the haloxyfop residue eluted with aqueous NaOH. After acidification of the aqueous phase, the residues are extracted into diethyl ether. Evaporation of the ether leaves the residue, which is methylated with diazomethane. The residue of methyl ester is further cleaned up on a Florisil cartridge, ready for GLC-ECD analysis.

Sugar beet roots, tops and immature plants (Yon *et al.,* 1984, ERC 83.19)

Analyte: haloxyfop GLC-ECD Method ERC 83.19

LOQ: 0.01 mg/kg in roots, 0.02 mg/kg in tops and plants

Description Homogenised matrix is extracted with methanolic NaOH. The mixture is shaken for 2 hours. An aliquot of the extract is acidified and the residues are partitioned into diethyl ether. Residues are cleaned up by solvent partition between ether and aqueous solutions (acid and alkaline) before being butylated with a sulphuric acid *n*-butanol reagent. After further cleanup on a Florisil column, the residue is analysed by GLC-ECD.

Sugar beet processed commodities (Yon, 1984, GHE-P-1125)

Analyte: haloxyfop GLC-ECD Method ERC 84.02

LOQ: 0.01 mg/kg

Description White sugar is dissolved in water. Cossettes and pulp are macerated with methanolic NaOH.

The extract is acidified and the residue is extracted into dichloromethane. Residues are cleaned up by solvent partition between dichloromethane and aqueous solutions (acid and alkaline) before being butylated with a sulphuric acid *n*-butanol reagent. After further cleanup on a Florisil column, the residue is analysed by GLC-ECD.

Oilseed rape straw and immature plants (Yon *et al.,* 1984, ERC 84.03)

Analyte: haloxyfop GLC-ECD Method ERC 84.03

LOQ: 0.05 mg/kg in straw, 0.02 mg/kg in plants.

Description Similar to ERC 83.17

Grapes (Anon, 1985, ERC 84.05)

Analyte: haloxyfop GLC-ECD Method ERC 84.05

LOQ: 0.01 mg/kg.

Description Similar to method ERC 88.4

Soya beans and cotton seed (Gardner, 1985, ACR 83.1.S1)

Analyte: haloxyfop GLC-ECD Method ACR 83.1.S1

LOQ: 0.01 mg/kg

Description Sample matrix is homogenized with methanolic NaOH and then the mixture is shaken overnight. A 25 mL aliquot is evaporated to 5 mL at 60 ºC. Water, sodium chloride and sulphuric acid are added and the residue is extracted into benzene. The benzene solution is applied to a silica gel column and the residues are eluted with acetic acid in dichloromethane. The solvent is evaporated to leave a residue which is methylated with BF_3-methanol. The methylated residue is further cleaned up on a SPE silica gel column and eluted with benzene ready for GLC-ECD analysis.

Citrus fruits (Rodrigues, 1987, GHB-P 040)

Analyte: haloxyfop GLC-ECD Method BRC 85.1

LOQ: 0.1 mg/kg

Description Citrus fruits are macerated and shaken with methanolic NaOH. After acidification of the extract, the residue is extracted into benzene and the benzene solution passed through a silica gel column. The residue is eluted from the silica gel with aqueous NaOH. The eluant is acidified and the residue extracted into ether, which is evaporated leaving the residue ready for methylation with BF_3 methanol and subsequent GLC-ECD analysis.

Bulb onions (Anon, 1988, ERC 87.7)

Analyte: haloxyfop GLC-ECD Method ERC 87.7

LOQ: 0.02 mg/kg.

Description Similar to method ERC 88.4

Apples and processed fractions (Gardner, 1990, GH-C 2037)

Analyte: haloxyfop GLC Method ACR 83.1R.S4

LOQ: 0.05 mg/kg.

Description The method is a replacement for ACR 83.1R.S2, developed from ACR 83.1.S1. Benzene is replaced by toluene.

Sunflower seed (Perkins and Harrison, 1990, GHE-P-2059)

Analyte: haloxyfop GLC-MSD Method GHE-P-2059

LOQ: 0.05 mg/kg.

Description Sunflower seeds are homogenised for 30 seconds with methanolic NaOH. The mixture is then shaken for 2 hours and left overnight at ambient temperature. An aliquot of the clear methanol layer is taken after centrifuging. Acid and salt are added and the residue is extracted into dichloromethane. The residue is then partitioned into aqueous sodium bicarbonate where it is treated with potassium permanganate. After 10 minutes, excess permanganate is removed with sodium metabisulphite, the mixture is treated with salt and acid and the residue is partitioned into di-isopropyl ether. The solvent is then evaporated. After butylation with a sulphuric acid-butanol reagent, the butyl ester is ready for GLC-MSD analysis.

Crude oil, refined oil and soapstock (Phillips, 1991, 89026

Analyte: haloxyfop GLC-MSD Method ACR 86.6.S2

LOQ: 0.1 mg/kg.

Description Soapstock samples are first extracted with diethyl ether. Crude oil, refined oil and the soapstock extract are hydrolysed in 1.5% KOH in ethanol+toluene with heating. After cooling and dilution with water the toluene layer is discarded. The residue is then partitioned from aqueous acid into toluene, adsorbed on silica gel and eluted with acetic acid in dichloromethane. The remainder of the cleanup procedure follows ACR 83.1.S1.

Beans (Butcher, 1992, ERC 91.7)

Analyte: haloxyfop GLC-ECD Method ERC 91.7

LOQ: 0.02 mg/kg.

Description Field beans are macerated and extracted with methanolic NaOH. The mixture is shaken for 2 hours. An aliquot of the extract is then cleaned up by solvent partition between toluene and aqueous solutions (acid and alkaline). A sodium bicarbonate solution of the residue is treated with permanganate and metabisulphite solutions before the residue is extracted back into toluene, which is then evaporated. After butylation with a sulphuric acid-butanol reagent, the butyl ester is ready for GLC-ECD analysis.

Oranges (Long and Butcher, 1993, ERC 92.24)

Analyte: haloxyfop GLC-ECD Method ERC 92.24

LOQ: 0.02 mg/kg.

Description Similar to ERC 91.7

Cotton seed (Long and Butcher, 1993, ERC 92.25)

Analyte: haloxyfop GLC-ECD Method ERC 92.25

LOQ: 0.02 mg/kg.

Description Similar to ERC 91, but with an additional cartridge cleanup of the butyl ester before the GLC analysis. Also, the sample extraction step leaves the sample over night in contact with the extracting solvent, methanolic NaOH, which may hydrolyse conjugates.

Apples (Perkins, 1994, ERC 88.4)

Analyte: haloxyfop GLC-ECD Method ERC 88.4

LOQ:	0.02 mg/kg.		
Description	Apple matrix is extracted with methanolic sodium hydroxide and then the extraction mixture is mixed with water, sulphuric acid and dichloromethane. The residue in dichloromethane solution is then cleaned up by solvent partition between dichloromethane and aqueous solutions (acid and alkaline). The residue is then butylated with a sulphuric acid-butanol reagent and the butyl ester is further cleaned up on a small Florisil column before GLC-ECD analysis.		
Potatoes, sugar beet roots and peas (Perkins, 1994, ERC 89.3)			
Analyte:	haloxyfop	GLC-ECD	Method ERC 89.3
LOQ:	0.01 mg/kg potato, 0.02 mg/kg sugar beet, 0.05 mg/kg peas.		
Description	Sample matrix is homogenized and extracted with methanolic sodium hydroxide and then the extraction mixture is mixed with water, acid and dichloromethane. The residue in dichloromethane solution is then cleaned up by solvent partition between dichloromethane and aqueous solutions (acid and alkaline). Finally the residue is extracted from an acidified aqueous phase into di-isopropyl ether. After solvent evaporation, the residue is then butylated with a sulphuric acid-butanol reagent and the butyl ester is further cleaned up on a small Florisil column before GLC-ECD analysis.		
Soya beans (Perkins, 1994, ERC 88.3)			
Analyte:	haloxyfop	GLC-ECD	Method ERC 88.3
LOQ:	0.05 mg/kg		
Description	Similar to method ERC 83.17.		
Canola forage, fodder and grain (Cowles *et al.,* 1998, GHF-P 1700))			
Analyte:	haloxyfop-P	GLC-ECD	Method PA-RM-98-02
LOQ:	0.02 mg/kg canola forage and grain, 0.05 mg/kg for canola fodder		
Description	Haloxyfop-P is extracted from milled canola forage, fodder or grain with methanolic NaOH An aliquot of the mixture, after acidification with hydrochloric acid and addition of sodium chloride, is extracted with toluene. The residue is partitioned into aqueous sodium carbonate and, after addition of acid and sodium chloride, into ethyl acetate. The residue is then butylated and the butyl ester partitioned into hexane. The hexane is evaporated and the residue is further cleaned up on a silica solid phase extraction column ready for GLC-ECD analysis.		
Chickpea straw and grain (Cowles, 1998, GHF-P 1718)			
Analyte:	haloxyfop-P	GLC-ECD	Method PA-RM-98-05
LOQ:	0.02 mg/kg chickpea grain, 0.05 mg/kg for chickpea straw.		
Description	Similar to PA-RM-98-02, but using a capillary column instead of a megabore column to separate the analyte from an interfering peak in chickpea straw.		
Plant material – medic, clover, lucerne, spinach, lettuce, garlic, asparagus, flax, strawberries (Clements *et al.,* 2001, GRM 00.02)			
Analyte:	haloxyfop	LC-MS-MS	Method GRM 00.02
LOQ:	0.05 mg/kg for medic, clover, lucerne. 0.01 mg/kg for others.		
Description	Crop matrix samples are homogenised with methanolic NaOH. Water and sulphuric acid-sodium chloride are added to an aliquot of the extract and haloxyfop residues are partitioned into toluene. The toluene solution is then passed through an anion exchange solid phase extraction cartridge and then haloxyfop residues are eluted with formic acid. The eluate is evaporated to dryness and then taken up in mobile solvent (acetonitrile-water) for HPLC-MS-MS analysis.		
Dry beans, soya beans, cotton seed (Pinheiro, GHB-P 721)			
Analyte:	haloxyfop-P	GC-MSD	Method GRM 01.10
LOQ:	0.01 mg/kg		
Description	Crop matrix samples are extracted with methanolic NaOH. After the methanol is evaporated, the extract is diluted with water, saturated with sodium chloride, acidified with sulphuric acid and then extracted with diethyl ether. Haloxyfop residues are then extracted from the ether phase into aqueous sodium bicarbonate, which is then acidified with sulphuric acid and saturated with sodium chloride. The residues are next extracted into toluene, which is evaporated to dryness to leave a residue ready for diazomethane methylation. The methyl ester is further cleaned up on a silica solid phase column before GC-MSD analysis.		
Oilseed rape plant and seed (Balluff, 2007, GHE-P-11656)			
Analyte:	haloxyfop-P	LC-MS-MS	Method GRM 04.03
LOQ:	0.01 mg/kg		
Description	Crop matrix samples are homogenised and shaken with methanolic NaOH. Haloxyfop-P-methyl was hydrolysed during this process. An aliquot of the extract is acidified with hydrochloric acid and cleaned up on a solid phase extraction system. An internal standard (deuterated haloxyfop) is added to the eluate, which is then ready for analysis by HPLC with negative-ion electrospray ionization tandem mass spectrometry. A second ion transition is also monitored to assist in identification of the residue.		

Gardner (1983, GH-C 1625) tested the completeness of extraction of haloxyfop and its conjugates and of their conversion to parent acid using method ACR 83.1 to extract soya bean samples available from the previous metabolism study (Yackovich and Miller, 1983, GH-C 1618). Method ACR 83.1 uses overnight shaking of substrate with 0.1M NaOH in 98% methanol + 2% water for extraction, which extracted 93% of the ^{14}C from the soya beans. HPLC produced a single peak matching haloxyfop which accounted for 95% of the ^{14}C in the extract.

Gardner (1984, GH-C 1709) tested the completeness of extraction of haloxyfop present as the free acid, the methyl ester or as conjugates from milk using method ACR 84.6. The milk was goat milk from a dosing study with [^{14}C]haloxyfop-butyl. Haloxyfop was quantitatively (99–100%) extracted in the three ether extractions. Completeness of hydrolysis of haloxyfop-methyl and conjugates to haloxyfop acid was checked by measuring the ^{14}C in the benzene washes after hydrolysis (1.6% of ^{14}C remained) and the ^{14}C remaining in the acidified aqueous solution after benzene extraction (5% of ^{14}C, representing polar degradation products of hydrolysis). A high percentage of the ^{14}C (91%) was present in the benzene solution (as haloxyfop acid)ready for further cleanup.

Analytical method GRM 01 09 for animal commodities was successfully subjected to independent laboratory validation (Schuster, 2002, CCRL #990109). Recovery testing data are included in Table 9.

Analytical method ERC 83.17 for oilseed rape was successfully subjected to independent laboratory validation (Nicholson and Schultz, 2000, IF-100/18445-00). Recovery testing data are included in Table 9.

Analytical method ERC 89.3 for potatoes, sugar beet roots and peas was successfully subjected to independent laboratory validation (Rawle, 2000, CEMS-1240). Recovery testing data are included in Table 9.

Class (2002, B 579/1) examined the possibilities of including haloxyfop in a multiresidue method of analysis. Haloxyfop requires hydrolysis in the extraction step for release of conjugates and, for GLC analysis, it requires a derivatisation step. These two requirements mean that haloxyfop does not fit into existing multiresidue methods.

Gardner (1988, 2084-21) drew attention to the potential losses of haloxyfop that may occur during the hydrolysis step of some analytical methods. If haloxyfop is exposed to higher temperatures or longer hydrolysis times than ideal, low recoveries may occur. The condition of 35–40 minutes at 70 °C was satisfactory.

Table 9 Analytical recoveries for spiked haloxyfop, haloxyfop-P, haloxyfop-P-methyl, haloxyfop-methyl or haloxyfop-ethoxyethyl in various substrates

Commodity	Spiked compound	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
apple dry pomace	haloxyfop	0.05	3	95%	88–100%	ACR 83.1R	2084-24
apple juice	haloxyfop	0.05	4	88%	72–96%	ACR 83.1R	2084-24
apple wet pomace	haloxyfop	0.05	4	82%	76–86%	ACR 83.1R	2084-24
apples	haloxyfop	0.05–0.10	14	95%	60–134%	ACR 83.1R	2084-24
apples	haloxyfop	0.02–5	12	93%	76–112%	ERC 88.4	ERC 88.4+
asparagus	haloxyfop	0.01–15	3	90%	87–93%	GRM 00.02	GRM 00.02
bovine fat	haloxyfop	0.01–0.2	11	77%	71–90%	ACR 84.1	ACR 84.1
bovine fat	haloxyfop	0.01–0.25	12	97%	60–107%	NCRL-PHF-01	NCRL 9902
bovine kidney	haloxyfop	0.01–2.0	11	90%	75–110%	ACR 84.1	ACR 84.1
bovine kidney	haloxyfop	0.01–0.5	6	99%	96–101%	GRM 01.09	GRM 01.09
bovine kidney	haloxyfop	0.01–0.25	12	103%	92–110%	NCRL-PHT-01	GHF-P 1939
bovine liver	haloxyfop	0.01–1.0	11	90%	83–97%	ACR 84.1	ACR 84.1
bovine liver	haloxyfop	0.01	5	75%	72–76%	GRM 01.09 (ILV)	CCRL #990109
bovine liver	haloxyfop	0.5	5	89%	86–93%	GRM 01.09 (ILV)	CCRL #990109
bovine liver	haloxyfop	0.01–0.25	12	100%	83–118%	NCRL-PHT-01	NCRL 9902
bovine muscle	haloxyfop	0.01–0.1	10	91%	86–94%	ACR 84.1	ACR 84.1
bovine muscle	haloxyfop	0.01–0.5	7	99%	96–100%	GRM 01.09	GRM 01.09

Commodity	Spiked compound	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
bovine muscle	haloxyfop	0.01–0.25	12	101%	75–114%	NCRL-PHT-01	NCRL 9902
bulb onions	haloxyfop	0.02	6	109%	104–125%	ERC 87.7	ERC 87.7
bulb onions	haloxyfop	0.05–5.0	18	86%	70–97%	ERC 87.7	ERC 87.7
chicken fat	haloxyfop	0.01–0.5	20	99%	97–102%	GRM 01.09	GRM 01.09
chicken liver	haloxyfop	0.01–0.5	7	99%	96–102%	GRM 01.09	GRM 01.09
chickpea grain	haloxyfop-P	0.02–0.2	11	93%	81–109%	PA-RM-98-05	GHF-P 1718
chickpea straw	haloxyfop-P	0.05–0.5	10	100%	95–102%	PA-RM-98-05	GHF-P 1718
citrus fruits	haloxyfop	0.1–1.0	12	92%	88–94%	BRC 85.1	GHB-P 040
coffee beans	haloxyfop	0.02–0.20	23	95%	81–114%	BRC 83.1	BRC 83.1
cotton seed	haloxyfop	0.01–0.4	10	89%	79–105%	ACR 83.1	ACR 83.1.S1
cotton seed	haloxyfop	0.02–0.10	5	95%	88–103%	ERC 92.25	ERC 92.25
cream	haloxyfop	0.02	6	94%	89–99%	ACR 84.6R	ACR 84.6R
cream	haloxyfop	0.04	7	96%	89–106%	ACR 84.6R	ACR 84.6R
cream	haloxyfop	0.2	5	91%	75–99%	ACR 84.6R	ACR 84.6R
cream	haloxyfop	0.4	2		83, 84%	ACR 84.6R	ACR 84.6R
dried beans	haloxyfop	0.02–0.5	6	91%	81–99%	ERC 91.7	ERC 91.7+
eggs	haloxyfop	0.01–0.5	20	98%	95–101%	GRM 01.09	GRM 01.09
eggs	haloxyfop	0.01	5	110%	104–116%	GRM 01.09 (ILV)	CCRL #990109
eggs	haloxyfop	0.5	5	97%	95–98%	GRM 01.09 (ILV)	CCRL #990109
fennel	haloxyfop	0.02–0.10	6	92%	76–103%	ERC 92.24	ERC 92.24
flax grain	haloxyfop	0.01–15	3	83%	80–89%	GRM 00.02	GRM 00.02
flax straw	haloxyfop	0.01–15	3	88%	83–93%	GRM 00.02	GRM 00.02
garlic	haloxyfop	0.01–15	3	83%	73–89%	GRM 00.02	GRM 00.02
grapes	ethoxy ethyl haloxyfop	0.01–0.10	4	73%	66–78%	ERC 84.05	ERC 84.05
grapes	haloxyfop	0.01	8	96%	71–124%	ERC 84.05	ERC 84.05
grapes	haloxyfop	0.10–1.0	6	86%	68–98%	ERC 84.05	ERC 84.05
green bean pods	haloxyfop	0.02–0.5	6	99%	88–104%	ERC 91.7	ERC 91.7+
green beans	haloxyfop	0.02–0.5	6	99%	90–109%	ERC 91.7	ERC 91.7+
lettuce	haloxyfop	0.01–15	3	94%	90–100%	GRM 00.02	GRM 00.02
medic, clover, lucerne	haloxyfop	0.05	8	98%	94–109%	GRM 00.02	GRM 00.02
medic, clover, lucerne	haloxyfop	0.1	8	91%	75–106%	GRM 00.02	GRM 00.02
medic, clover, lucerne	haloxyfop	1.0	8	96%	86–110%	GRM 00.02	GRM 00.02
medic, clover, lucerne	haloxyfop	5	3	92%	89–96%	GRM 00.02	GRM 00.02
medic, clover, lucerne	haloxyfop	15	5	98%	85–108%	GRM 00.02	GRM 00.02
milk	haloxyfop	0.01	5	90%	86–93%	ACR 84.6R	ACR 84.6R
milk	haloxyfop	0.05–0.1	6	99%	92–104%	ACR 84.6R	ACR 84.6R
milk	haloxyfop	0.01–0.5 mg/L	20	98%	90–101%	GRM 01.09	GRM 01.09
milk	haloxyfop	0.01	5	90%	81–106%	GRM 01.09 (ILV)	CCRL #990109
milk	haloxyfop	0.5	5	98%	94–101%	GRM 01.09 (ILV)	CCRL #990109
milk	haloxyfop	0.01–0.25	10	98%	84–108%	NCRL-PHM-01	NCRL 9902
oilseed rape	haloxyfop	0.05	6	83%	73–97%	ERC 83.17	ERC 83.17
oilseed rape	haloxyfop	0.1–5.0	9	75%	57–104%	ERC 83.17	ERC 83.17
oilseed rape	haloxyfop	0.05	6	93%	83–102%	ERC 83.17 (ILV)	IF-100/18445-00
oilseed rape	haloxyfop	0.5	6	92%	86–99%	ERC 83.17 (ILV)	IF-100/18445-00
oilseed rape	haloxyfop-P-methyl	0.01–10	20	83%	72–101%	GRM 04.03	GHE-P-11656
oilseed rape cake	haloxyfop	0.05–0.5	11	90%	80–114%	ERC 83.24	ERC 83.24
oilseed rape oil	ethoxy ethyl haloxyfop	0.01	7	90%	70–100%	ERC 83.20	ERC 83.20
oilseed rape oil	ethoxy ethyl haloxyfop	0.05–1.0	11	89%	71–102%	ERC 83.20	ERC 83.20
oilseed rape oil	haloxyfop	0.02–0.20	4	88%	62–118%	ERC 83.20	ERC 83.20
oilseed rape plants	haloxyfop	0.02	7	100%	88–114%	ERC 84.03	ERC 84.03
oilseed rape plants	haloxyfop	0.05–10	12	91%	74–105%	ERC 84.03	ERC 84.03
oilseed rape plants	haloxyfop-P-methyl	0.01–2.0	12	85%	68–108%	GRM 04.03	GHE-P-11656
oilseed rape straw	haloxyfop	0.05	6	72%	62–87%	ERC 84.03	ERC 84.03
oilseed rape straw	haloxyfop	0.2–1.0	12	79%	69–93%	ERC 84.03	ERC 84.03
orange peel	haloxyfop	0.02–0.10	6	90%	76–108%	ERC 92.24	ERC 92.24
orange pulp	haloxyfop	0.02–0.10	6	103%	96–110%	ERC 92.24	ERC 92.24

Commodity	Spiked compound	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
peas	haloxyfop	0.05–5.0	8	93%	84–102%	ERC 89.3	ERC 89.3
peas	haloxyfop	0.05–0.50	7	85%	74–97%	ERC 89.3 (ILV)	CEMS-1240
potatoes	haloxyfop	0.01–5.0	13	89%	76–108%	ERC 89.3	ERC 89.3
potatoes	haloxyfop	0.01–0.10	7	89%	85–95%	ERC 89.3 (ILV)	CEMS-1240
soya beans	haloxyfop	0.05–2.0	20	86%	69–105%	ARC 83.1	GH-C 1625
soya bean hulls	haloxyfop	0.05–0.50	3	96%	91–100%	ARC 83.1	GH-C 1625
soya bean meal	haloxyfop	0.05–0.50	3	94%	83–100%	ARC 83.1	GH-C 1625
soya bean crude oil	haloxyfop	0.05–0.50	3	61%	57–67%	ARC 83.1	GH-C 1625
soya bean refined oil	haloxyfop	0.05–0.50	3	73%	67–77%	ARC 83.1	GH-C 1625
soya beans	haloxyfop-methyl	0.05	2	94%	90%, 98%	ARC 83.1	GH-C 1625
soya beans	haloxyfop	0.01–0.10	9	101%	93–108%	ACR 83.1	ACR 83.1.S1
soya beans	haloxyfop	0.05–5.0	11	91%	78–104%	ERC 88.3	ERC 88.3
soya beans	haloxyfop-methyl	0.10	1	98%		ACR 83.1	ACR 83.1.S1
spinach	haloxyfop	0.01–15	6	92%	85–98%	GRM 00.02	GRM 00.02
strawberries	haloxyfop	0.01–15	3	75%	68–81%	GRM 00.02	GRM 00.02
sugar beet	haloxyfop	0.02–5.0	13	87%	80–94%	ERC 89.3	ERC 89.3
sugar beet	haloxyfop	0.02–0.20	4	86%	74–101%	ERC 89.3 (ILV)	CEMS-1240
sugar beet cossettes	haloxyfop	0.01–1.0	4	87%	84–91%	ERC 84.02	GHE-P-1125
sugar beet cossettes	haloxyfop-ethoxyethyl	0.01	1	120%		ERC 84.02	GHE-P-1125
sugar beet juice	haloxyfop	0.01–1.0	3	83%	73–96%	ERC 84.02	GHE-P-1125
sugar beet juice	haloxyfop-ethoxyethyl	0.01	1	96%		ERC 84.02	GHE-P-1125
sugar beet plants	haloxyfop	0.02	4	95%	81–115%	ERC 83.19	ERC 83.19
sugar beet plants	haloxyfop	0.05–5.0	12	82%	70–90%	ERC 83.19	ERC 83.19
sugar beet pulp	haloxyfop	0.025–0.05	3	85%	80–92%	ERC 84.02	GHE-P-1125
sugar beet pulp	haloxyfop-ethoxyethyl	0.025	1	93%		ERC 84.02	GHE-P-1125
sugar beet roots	haloxyfop	0.01	8	76%	64–88%	ERC 83.19	ERC 83.19
sugar beet roots	haloxyfop	0.02–1.0	8	71%	59–80%	ERC 83.19	ERC 83.19
sugar beet roots	haloxyfop	0.02–0.20	10	91%	77–111%	ERC 91.7	CEMS-185
sugar beet tops	haloxyfop	0.02	8	100%	79–114%	ERC 83.19	ERC 83.19
sugar beet tops	haloxyfop	0.10–10	11	83%	77–89%	ERC 83.19	ERC 83.19
sugar beet tops	haloxyfop	0.02–0.20	10	95%	85–104%	ERC 91.7	CEMS-185
sugar beet white sugar	haloxyfop	0.01, 0.1	2	81%	73%, 89%	ERC 84.02	GHE-P-1125
sugar beet white sugar	haloxyfop-ethoxyethyl	0.01, 0.1	2	85%	72%, 97%	ERC 84.02	GHE-P-1125
sunflower seeds	haloxyfop	0.05–0.20	16	97%	86–108%	BRC 83.1	BRC 83.1
sunflower seeds	haloxyfop	0.05	4	75%	73–80%	ERC 83.18	ERC 83.18
sunflower seeds	haloxyfop	0.1–5.0	8	80%	70–85%	ERC 83.18	ERC 83.18

ILV: independent laboratory validation

Stability of residues in stored analytical samples

Information was received on the freezer storage stability of haloxyfop in plant commodities.

Gardner (1990, GH-C 2037) reported on the frozen storage of haloxyfop fortified into apples, juice and pomace at 1 mg/kg levels. The data had been adjusted for procedural recoveries, which were not available, so the data could not be used.

Table 10 Freezer storage stability data for haloxyfop spiked into matrices of: green peas, cabbage and rice

Storage interval	Procedural recov %	haloxyfop, mg/kg or % remaining	Storage interval	Procedural recov %	haloxyfop, mg/kg or % remaining
GREEN PEAS, (homogenized matrix) fortified with haloxyfop at 0.2 mg/kg in small glass jars, storage temperature below -16 °C (Hastings and Butcher, 1993, GHE-P-3157).			CABBAGE, (chopped matrix) fortified with haloxyfop at 0.2 mg/kg in small glass jars, storage temperature below -16 °C (Hastings and Butcher, 1993, GHE-P-3158). Analytical method ERC 91.7.		
0 days	105% 110%	0.22 0.23	0 days	102% 99%	0.205 0.206

Storage interval	Procedural recov %	haloxyfop, mg/kg or % remaining	Storage interval	Procedural recov %	haloxyfop, mg/kg or % remaining
92 days	98% 99%	0.22 0.21	91 days	101% 96%	0.216 0.204
483 days	108% 110%	0.22 0.22	484 days	108% 106%	0.204 0.203
residues apparently stable			residues apparently stable		
RICE, fortified with haloxyfop at 1 mg/kg and stored in a freezer, temperature not reported (Gardner, 1989, GH-C 2175). Procedural recoveries not reported.					
4 months		0.91 0.99 1.09			
7 months		0.97 1.1			

Gardner (1983, GH-C 1625) tested the freezer storage stability of haloxyfop residues in soya beans matrix. Seventeen months after fortification with 1 mg/kg haloxyfop and storage in a freezer at -20 ºC, 93% remained.

Gardner (1983, GH-C 1623) tested the freezer storage stability of haloxyfop fortified into a control sample of cotton seed matrix at 1 mg/kg. Seventeen months after storage in a freezer at -20 °C, 87% of the haloxyfop remained.

Phillips (1991, 89026) reported that haloxyfop residues in soya beans were stable under frozen conditions for 43 months, the period of the test.

No data are available on the freezer storage stability of haloxyfop residues in animal commodities.

USE PATTERN

Haloxyfop-P is a systemic, post-emergent, selective herbicide used for the control of many annual and perennial grass weeds in a number of crops. Because of its penetration and translocation capacity, it acts quickly on competing weeds immediately after its application. Haloxyfop-P products are diluted with water and normally applied with a spraying oil or wetter, either as a directed spray to the ground at the base of the plant, or broadcast treatment using conventional spray equipment.

Haloxyfop-P is usually prepared as the methyl ester and formulated as an emulsifiable concentrate (EC). In some countries, the label concentration is expressed as the ester and in others as the acid equivalent. For the purposes of JMPR the active ingredient is expressed as haloxyfop-P acid.

The label directions for use make it clear that application rates depend on the nature of the weeds to be controlled rather than the crop. Timing is also somewhat dependent on the weeds to be controlled and their growth stages. Some labels rely on crop growth stages for directions on application timing, while others rely on pre-harvest intervals or give very little direction on timing.

The target for haloxyfop application is the weeds, not the crop. This is a different situation from the use of insecticides or fungicides where the crop is the target. For field crops, the amount of applied haloxyfop reaching the crop will depend very much on the degree of area cover provided by the crop. Only small percentages of the applied haloxyfop (on a kg ai/ha basis) will contact the crop in its early growth stages.

The label directions often suggest the addition of crop oil to the spray and sometimes mandate such an addition. Its purpose is to increase efficacy. Labels provide specific instructions on crops and weeds where crop oil should be used and on others where it should not. The complexity of when and when not to use crop oils is not included in Table 11.

Table 11 Registered uses of haloxyfop-P-methyl in horticultural and field crops. The table is based on information from labels and translated labels available to JMPR. The active ingredient (ai) is expressed as haloxyfop-P acid.

Crop	Country	Highest application rate, kg ai/ha	PHI	Use instructions
Alfalfa	Australia	0.078		Apply from second trifoliate leaf onwards. Grazing or cutting for livestock feed interval, 21 days.
Alfalfa	Chile	0.06		Grazing or cutting for livestock feed interval, 30 days.
Alfalfa	Mexico	0.18		Grazing or cutting for livestock feed interval, 21 days.
Alfalfa	South Africa	0.42		The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to. Grazing or cutting for livestock feed interval, 28 days.
Apple	Romania	0.10	60–90	
Apple	Saudi Arabia	0.06	90	
Apple orchards	Serbia	0.16		Before flowering of apple
Apple trees	Chile	0.09	14	Maximum 3 times per season.
Apple, pear	Algeria	0.40		
Apricot trees	Chile	0.09	14	Maximum 3 times per season.
Apricot trees	Saudi Arabia	0.06	90	
Avocado trees	Chile	0.09	14	Maximum 3 times per season.
Bean, field	Switzerland	0.16	56	
Bean, field	Tunisia	0.10		
Beans	Argentina	0.15	65	
Beans	Brazil	0.048	66	Usually apply between 20 and 30 days after sowing.
Beans	Chile	0.09	30	
Beans	Costa Rica	0.046		Apply between 15 and 30 days after sowing, depending upon infestation level.
Beans	Guatemala	0.046		Apply between 15 and 30 days after sowing, depending upon infestation level.
Beans	Morocco	0.05		
Beans, dry	South Africa	0.42	60	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Beans, Faba	Australia	0.039		Apply from second leaf, second node or second branch to full flowering. Grazing or cutting for livestock feed interval, 28 days.
Beans, green	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Beetroot	Syria	0.13		
Beets	Algeria	0.40		
Beets	Iraq	0.12		
Beets	Morocco	0.05		
Beets	Romania	0.16	60–90	
Berries	Switzerland	0.16		
Bush beans	Switzerland	0.16	28	
Cabbage	China	0.062		
Canola	Argentina	0.15		
Canola	Australia	0.052		Apply from second leaf to prior to bud formation and stem elongation. Grazing or cutting for livestock feed interval, 28 days.
Canola	Paraguay	0.15		
Canola	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Cherry trees	Chile	0.09	14	Maximum 3 times per season.

Crop	Country	Highest application rate, kg ai/ha	PHI	Use instructions
Chickpeas	Australia	0.052		Apply from second leaf, second node or second branch prior to flowering. Grazing or cutting for livestock feed interval, 28 days.
Chickpeas	Israel	0.1		
Chickpeas	Tunisia	0.10		
Citrus fruits	Saudi Arabia	0.06	90	
Citrus fruits	Tunisia	0.10		
Citrus fruits	Turkey	0.25	15	
Citrus trees	New Zealand	0.15		Apply as a directed spray around the base of the trees, to avoid direct contact with the fruit.
Coffee	Colombia	0.36		Apply between the furrows of trees.
Cotton	Argentina	0.15		
Cotton	Australia	0.078		Apply from second leaf to before the onset of flowering. Grazing or cutting for livestock feed interval, 28 days.
Cotton	Brazil	0.060	123	Usually apply between 20 and 45 days after sowing.
Cotton	China	0.14		
Cotton	Iraq	0.12		
Cotton	Israel	0.1		
Cotton	Paraguay	0.15		
Cotton	Russian Fed	0.16		1 treatment
Cotton	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Cotton	Turkey	0.060	120	
Cotton	Turkey	0.062	15	
Forage legumes	Uruguay	0.060		
Fruit	Switzerland	0.16		
Fruit trees	Romania	0.052	60–90	
Fruit trees	Syria	0.13		
Fruit trees	Uruguay	0.15		
Fruit, deciduous	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Fruit, orchard crops	Uruguay	0.18		Apply before blooming
Grape vine	Lebanon	0.13		
Grapefruit trees	Chile	0.09	14	Maximum 3 times per season.
Grapes	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Grapevine	Romania	0.16	60–90	
Grapevine	Switzerland	0.16		
Grapevines	Chile	0.09	14	Maximum 3 times per season.
Lemon trees	Chile	0.09	14	Maximum 3 times per season.
Mandarin trees	Chile	0.09	14	Maximum 3 times per season.
Nectarine trees	Chile	0.09	14	Maximum 3 times per season. Apply directing to ground level in peach and nectarine trees, since defoliation might take place in leaves that contact herbicide.
Oilseed rape	Belarus	0.10		
Oilseed rape	Chile	0.06	30	Apply with crop in phenological stage of 2–3 leaves, until before floral stem shooting.
Oilseed rape	China	0.043		
Oilseed rape	Moldova	0.10		
Oilseed rape	Romania	0.10	60–90	
Oilseed rape	Russian Fed	0.10		1 treatment
Oilseed rape	Switzerland	0.16		Application: autumn

Crop	Country	Highest application rate, kg ai/ha	PHI	Use instructions
Oilseed rape	Ukraine	0.10		
Oilseed rape	Uruguay	0.075		
Onion, transplanted	Philippines	0.05		Spray at 10–14 days after planting
Onions	Chile	0.09	30	
Onions	Iran	0.12	28	
Onions	Iraq	0.12		
Onions	Israel	0.1		
Onions	Lebanon	0.13		
Onions	Moldova	0.10		
Onions	New Zealand	0.15	35	
Onions	Romania	0.10	60–90	
Onions	Saudi Arabia	0.06	30	
Onions	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Onions	Switzerland	0.16	56	
Onions	Tunisia	0.10		
Onions	Turkey	0.047	52	
Orange trees	Chile	0.09	14	Maximum 3 times per season.
Oranges	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Orchard, vine and plantation	Australia	0.42		Includes: apples, avocado, banana, blueberry, citrus, custard apple, feijoa, grapevines, guava, kiwifruit, litchi, longan, mango, nashi, nut trees, passionfruit, papaya, pear, persimmon, pineapple, rambutan and stone fruit. Spray should be directed to base of tree or vine. Avoid contact with fruit and foliage.
Peach trees	Chile	0.09	14	Maximum 3 times per season. Apply directing to ground level in peach and nectarine trees, since defoliation might take place in leaves that contact herbicide.
Peach trees	Saudi Arabia	0.06	90	
Peanuts	Argentina	0.15		
Peanuts	Australia	0.078		Apply from second leaf to pegging. Grazing or cutting for livestock feed interval, 28 days.
Peanuts	China	0.047		
Peanuts	Israel	0.1		
Peanuts	Paraguay	0.15		
Peanuts	Saudi Arabia	0.06	80	
Peanuts	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Peanuts	Turkey	0.062	98	
Pear trees	Chile	0.09	14	Maximum 3 times per season.
Peas	Chile	0.09	30	
Peas	Israel	0.1		
Peas	Morocco	0.05		
Peas	New Zealand	0.15		Do not apply later than flowering
Peas	Saudi Arabia	0.06	60	
Peas	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Peas	Switzerland	0.16	56	
Peas	Tunisia	0.10		
Peas (grain)	Belarus	0.10		
Peas (grain)	Ukraine	0.10		

Crop	Country	Highest application rate, kg ai/ha	PHI	Use instructions
Peas, field	Australia	0.052		Apply from second leaf, second node or second branch prior to flowering. Grazing or cutting for livestock feed interval, 28 days.
Peas, field	Switzerland	0.16	28	
Pineapples	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Plum trees	Chile	0.09	14	Maximum 3 times per season.
Pome fruit trees	New Zealand	0.15		Apply as a directed spray around the base of the trees, to avoid direct contact with the fruit.
Potatoes	China	0.078		
Potatoes	Saudi Arabia	0.06	90	
Potatoes	Ukraine	0.10		
Soya beans	Argentina	0.15		
Soya beans	Australia	0.078		Apply from second leaf to flowering. Grazing or cutting for livestock feed interval, 28 days.
Soya beans	Bolivia	0.062		
Soya beans	Brazil	0.060	98	Usually apply between 20 and 45 days after sowing.
Soya beans	China	0.14		
Soya beans	Moldova	0.10		
Soya beans	Paraguay	0.15		
Soya beans	Romania	0.16	60–90	
Soya beans	Russian Fed	0.10		1 treatment
Soya beans	Serbia	0.16		Single application
Soya beans	South Africa	0.42	60	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Soya beans	Switzerland	0.16	56	
Soya beans	Thailand	0.078		Apply when soya beans are 25–28 days old.
Soya beans	Turkey	0.062	15	
Soya beans	Ukraine	0.05		
Soya beans	Uruguay	0.075		
Sugar beet	Belarus	0.10		
Sugar beet	Chile	0.09	30	
Sugar beet	Iran	0.12	28	
Sugar beet	Lebanon	0.13		
Sugar beet	Moldova	0.10		
Sugar beet	Russian Fed	0.10		1 treatment
Sugar beet	Serbia	0.16		Single application
Sugar beet	Switzerland	0.16	56	
Sugar beet	Ukraine	0.10		
Sunflowers	Argentina	0.15		
Sunflowers	Australia	0.078		Apply from second leaf to head initiation. Grazing or cutting for livestock feed interval, 28 days.
Sunflowers	Israel	0.1		
Sunflowers	Moldova	0.10		
Sunflowers	Paraguay	0.15		
Sunflowers	Romania	0.10	60–90	
Sunflowers	Russian Fed	0.10		1 treatment
Sunflowers	Serbia	0.16		Single application
Sunflowers	South Africa	0.42	40	The crop may be treated at any time when the weeds are in the correct stage, providing that the withholding periods are adhered to.
Sunflowers	Switzerland	0.16		
Sunflowers	Tunisia	0.10		
Sunflowers	Ukraine	0.05		

Crop	Country	Highest application rate, kg ai/ha	PHI	Use instructions
Sunflowers	Uruguay	0.075		
Tangerine trees	Chile	0.09	14	Maximum 3 times per season.
Vines	Algeria	0.40		
Vines	Israel	0.1		
Vines	Saudi Arabia	0.06	90	
Vines	Syria	0.13		
Vines	Tunisia	0.10		
Vineyards	Serbia	0.16		Before flowering of grape vine
Vineyards	Turkey	0.25	52	

The Netherlands provided information that products based on haloxyfop or haloxyfop-P are not authorised for use on agricultural crops in the Netherlands.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for haloxyfop uses on crops that produced residue data. The commodities are grouped into Codex commodity groups.

Commodities	Crop group	Table
Oranges, grapefruit, lemons: *Australia, Brazil, Italy, New Zealand.* Haloxyfop-P-methyl, haloxyfop-methyl, haloxyfop-ethoxyethyl.	Citrus fruits	Table 13
Apples: *Australia, Italy, New Zealand, USA.* Haloxyfop-ethoxyethyl, haloxyfop-methyl.	Pome fruits	Table 14
Peaches: *Australia*. Haloxyfop-ethoxyethyl.	Stone fruits	Table 15
Grapes: *Australia, France, Italy*. Haloxyfop-ethoxyethyl, haloxyfop-P-methyl.	Berries and other small fruits	Table 16
Bananas: *Australia*. Haloxyfop-ethoxyethyl.	Tropical fruits, inedible peel	Table 17
Onions: *Belgium, France, Germany, New Zealand.* Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Bulb vegetables	Table 18
Field beans: Belgium, France, Germany, Greece, Spain. Haloxyfop-P-methyl.	Legume vegetables	Table 19
Peas: Belgium, France, Italy, Spain. Haloxyfop-P-methyl.	Legume vegetables	Table 20
Pigeon peas: *Australia*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Pulses	Table 21
Beans: Argentina, Brazil, Costa Rica, Germany. Haloxyfop-P-methyl.	Pulses	Table 22

Commodities	Crop group	Table
Chickpeas: *Australia*. Haloxyfop-P-methyl.	Pulses	Table 23
Peas, pulses: *Australia, France*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Pulses	Table 24
Soya beans: Argentina, Brazil, France, Germany, Hungary, Italy, Spain, USA. Haloxyfop-P-methyl, haloxyfop-methyl.	Pulses	Table 25
Sugar beet: *Belgium, France, Germany, Italy, Spain*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Root and tuber vegetables	Table 26
Rice: *USA*. Haloxyfop-methyl.	Cereal grains	Table 27
Cotton: *Brazil, Greece, Spain, USA*. Haloxyfop-P-methyl, haloxyfop-methyl.	Oilseed	Table 28
Cotton: *USA*. Haloxyfop-methyl	Oilseed	Table 29
Oilseed rape: *Australia, France, Germany, Greece, Italy, Poland, Spain*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Oilseed	Table 30
Peanuts: *Argentina, Australia*. Haloxyfop-P-methyl.	Oilseed	Table 31
Sunflowers: *Argentina, France, Germany, Greece, Spain*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Oilseed	Table 32
Coffee: *Brazil, Colombia*. Haloxyfop-methyl.	Seed for beverages	Table 33
Alfalfa: *Australia, France, Germany, Poland*. haloxyfop-ethoxyethyl, haloxyfop-P-methyl.	Legume animal feeds	Table 34
Chickpeas, peanuts, peas and soya beans: *Australia, France, Germany, Hungary, Italy, Spain*. Haloxyfop-P-methyl.	Legume animal feeds	Table 35
Sugar beet: *Belgium, Germany, Italy, Spain*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Miscellaneous fodder and forage	Table 36
Oilseed rape: *Australia, France, Germany, Greece, Italy, Poland, Spain*. Haloxyfop-P-methyl, haloxyfop-ethoxyethyl.	Miscellaneous fodder and forage	Table 37

Trials were generally well documented with laboratory and field reports; trials from the 1980s followed the standards of those times. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Control samples are indicated in the summary tables with a "c". Residue data are recorded unadjusted for recovery.

Label directions for use of haloxyfop often suggest the addition of crop oil to the spray and crop oils were used in many of the supervised trials. In the tables of residue data, the use of crop oil is indicated in the formulation column by "+ oil".

Various compounds were used in the supervised trials: haloxyfop-P-methyl, haloxyfop-methyl and haloxyfop-ethoxyethyl. The compound used in each trial is indicated in the formulations column. The application rate, kg ai/ha, is expressed in terms of haloxyfop or haloxyfop-P, i.e. the active ingredient is taken as haloxyfop or haloxyfop-P, not the esters.

Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Growth stage of the crop at the time of application may be an important determinant of residues in the harvested commodity. For haloxyfop, growth stage at time of application may be more important than PHI. Growth stage data have been included in the trial data summaries where available.

Because of the importance of growth stage at time of treatment and the nature of the field crops being tested, in some residue trials separate plots were treated at various growth stages and then all were harvested at crop maturity on the same day.

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

Table 12 Summary of sprayers, plot sizes and field sample sizes in the supervised trials

Crop	Place	Year	Sprayer	Plot size	Sample size
Alfalfa	Australia	1988–89	precision plot sprayer	60–240 m^2	?
Alfalfa	Australia	1998	precision plot sprayer, LPG precision sprayer	108–120 m^2	?
Alfalfa	Europe	2004	backpack with boom, plot boom sprayer	90–120 m^2	1 kg
Apples	Australia	1991	LPG powered, hand lance	2 trees	?
Apples	Italy	1987	pressure sprayers	?	?
Apples	New Zealand	1986	precision plot sprayer	9 m^2	2 kg, 15 fruit
Apples	USA	1987	CO_2 powered backpack, tractor mounted CO_2 sprayer	1 tree, 37–46 m^2	?
Banana	Australia	1991	precision plot sprayer	?	25 fruit
Beans	Argentina	1992–93	CO_2 powered backpack	13–45 m^2	2 kg
Beans	Brazil	2001–2002	CO_2 powered backpack	72–180 m^2	2 kg
Beans	Brazil	1997–98	CO_2 powered backpack	20–60 m^2	2 kg
Beans	Costa Rica	1998	CO_2 powered backpack	32 m^2	2 kg
Beans	Europe	1999	hand carried boom sprayer	45 m^2	> 1 kg
Beans	Europe	2000–01	3-metre boom, knapsack sprayer, compressed air sprayer	45–90 m^2	1–2 kg
Beans	Germany	1990	plot sprayer with teejet	50–100 m^2	?
Chickpeas	Australia	1997	small plot sprayer, all terrain vehicle and precision gas powered sprayer	120–132 m^2	?
Citrus	Brazil	1986	CO_2 powered backpack	?	?
Coffee	Brazil	1983–84	CO_2 powered backpack, knapsack	450 m^2	?
Coffee	Colombia	1984	CO_2 powered backpack	3 trees	?
Cotton	Brazil	1984–85	CO_2 powered backpack, knapsack	18–72 m^2	?
Cotton	Brazil	1997	CO_2 powered backpack	24 m^2	1–2 kg

Crop	Place	Year	Sprayer	Plot size	Sample size
Cotton	Brazil	2001	CO_2 powered backpack	60–500 m^2	2 kg
Cotton	Greece	1999–2000	A20 sprayer	40 m^2	?
Cotton	Spain	1999–2000	backpack with boom, conventional boom sprayer	60–275 m^2	1 kg, 5 kg bolls
Cotton	Spain	1991	CO_2 powered boom spray, flat fan	60 m^2	?
Cotton	USA	1982	backpack, tractor mounted sprayer, hand-held CO_2 sprayer, compressed air sprayer	19–465 m^2	?
Grapefruit	New Zealand	1985	precision backpack	10 m^2	2 kg, > 8 fruit
Grapes	Australia	1990	precision plot sprayer	1 vine	?
Grapes	France	1982	knapsack sprayer with boom	?	?
Grapes	France	1985	Crystal sprayer with boom and teejet	?	1 kg
Grapes	Italy	1989	Crystal sprayer, motor pump and teejet	10–40 m^2	1 kg
Lemons	Australia	1990	precision plot sprayer	1 tree	?
Lemons	New Zealand	1991	precision backpack	1 tree	?
Oilseed rape	Australia	1997	precision plot sprayer, all terrain vehicle	32–240 m^2	?
Oilseed rape	Europe	1988–89	knapsack sprayer, plot sprayer with teejet, Crystal sprayer	20–15 m^2	2–5 kg?
Oilseed rape	Europe	2000	conventional boom sprayer, precision plot sprayer	60–100 m^2	1 kg from 12 plants
Oilseed rape	Europe	2006	conventional boom sprayer	30–120 m^2	0.2–2.6 kg
Oilseed rape	France	1983	?	?	?
Oilseed rape	France	2002	conventional boom sprayer	45 m^2	> 1 kg
Oilseed rape	France	2002	conventional boom sprayer	45 m^2	> 1 kg
Onions	Europe	1999–2000	3 metre boom, boom sprayers	42–93 m^2	1–2.3 kg
Onions	New Zealand	1987	precision plot sprayer	12 m^2	2 kg, 12 onions
Orange	Italy	1991	knapsack	3 trees	24 fruits
Peach	Australia	1991	LPG powered hand lance	2 trees	?
Peanuts	Argentina	1992	CO_2 powered backpack	11 m^2	?
Peanuts	Australia	1997	precision plot sprayers	120 m^2	?
Peas	Australia	1989	precision plot sprayers	84–90 m^2	?
Peas	Europe	1999–2000	hand carried boom sprayer, motorised backpack, plot sprayer, 3 metre boom, knapsack	44–90 m^2	1–6 kg
Peas	Europe	1988–89	broadcast backpack, knapsack, Cristal sprayer	13–180 m^2	?
Peas	Europe	2004	AUK plot sprayer	39–60 m^2	0.3–1 kg
Pigeon peas	Australia	1989	precision plot sprayer	32 m^2	?
Rice	USA	1987–88	aircraft, CO_2 powered backpack, small plot sprayers, 4-wheeled CO_2 powered sprayer	37–3300 m^2	?
Soya beans	Argentina	1991	CO_2 powered backpack	25 m^2	?
Soya beans	Brazil	1984	CO_2 powered backpack	15–30 m^2	2 kg
Soya beans	Brazil	1990	CO_2 powered backpack	20 m^2	?
Soya beans	Brazil	1992	CO_2 powered backpack	10–12 m^2	?
Soya beans	Brazil	1995	CO_2 powered backpack	32–72 m^2	?
Soya beans	Brazil	2000–02	CO_2 powered handboom	64–184 m^2	2 kg
Soya beans	Europe	2000–01	AUK plot sprayer, plot boom sprayer	30–60 m^2	1–3 kg
Soya beans	USA	1982	tractor mounted boom, CO_2 powered backpack, plot sprayers	20 m^2 to 3 rows of 45 m	?
Soya beans	USA	1982	tractor mounted boom, CO_2 powered backpack, plot sprayers	19 m^2 to 8 rows of 75 m	?
Soya beans	USA	1989	plot sprayer, hand boom sprayer	37–139 m^2	?

Crop	Place	Year	Sprayer	Plot size	Sample size
Sugar beet	Europe	1988	knapsack with teejet, flat nozzle sprayer	22–144 m^2	5 plants
Sugar beet	Europe	2000	boom sprayers	42–45 m^2	1–6 kg
Sugar beet	Italy	1992	Crystal sprayer with teejet	20 m^2	?
Sugar beet	UK	1983	precision plot sprayer	?	?
Sunflower	Argentina	1992	CO_2 backpack sprayer	27 m^2	?
Sunflower	Europe	2000	boom sprayers	42–60 m^2	1–1.3 kg
Sunflower	Europe	2001	boom sprayers, compressed air sprayers	45–60 m^2	1 kg
Sunflower	France	1988–89	Crystal sprayer, knapsack sprayer	24–36 m^2	?
Sunflower	France	1999	hand carried boom sprayer	45–60 m^2	?

Table 1 Haloxyfop residues in citrus fruits resulting from supervised trials in Australia, Brazil, Italy and New Zealand.

CITRUS FRUITS	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
CITRUS Brazil (SP), 1986 (Baiana)	EC + oil haloxyfop-methyl	0.24 directed [a]			1	67	fruit	< 0.1 (3)	GHB-P 040
CITRUS Brazil (SP), 1986 (Baiana)	EC + oil haloxyfop-methyl	0.48 directed [a]			1	67	fruit	< 0.1 (3)	GHB-P 040
CITRUS Brazil (SP), 1986 (Baiana)	EC + oil haloxyfop-methyl	0.72 directed [a]			1	67	fruit	< 0.1 (3)	GHB-P 040
CITRUS Brazil (SP), 1986 (Baiana)	EC + oil haloxyfop-methyl	0.96 directed [a]			1	67	fruit	< 0.1 (3)	GHB-P 040
CITRUS Brazil (SP), 1986 (Baiana)	EC + oil haloxyfop-methyl	1.44 directed [a]			1	67	fruit	< 0.1 (3)	GHB-P 040
CITRUS Brazil (SP), 1986 (Baiana)	EC + oil haloxyfop-methyl	1.9 directed [a]			1	67	fruit	< 0.1 (3)	GHB-P 040
GRAPEFRUIT New Zealand, 1985 (Morrison Seedless)	EC + oil haloxyfop-ethoxyethyl	0.21 directed [a]		200	1	29	fruits	< 0.01 (2)	GHF-P-515 011-209
GRAPEFRUIT New Zealand, 1985 (Morrison Seedless)	EC + oil haloxyfop-ethoxyethyl	0.42 directed [a]		200	1	29	fruits	< 0.01 (2)	GHF-P-515 011-209
LEMONS Australia (NSW), 1990 (Lisbon)	EC haloxyfop-ethoxyethyl	0.21		100	1	28 58	fruits	< 0.05 (2) < 0.05 (2)	GHF-P 1110
LEMONS Australia (NSW), 1990 (Lisbon)	EC haloxyfop-ethoxyethyl	0.42		100	1	28 58	fruits	< 0.05 (2) < 0.05 (2)	GHF-P 1110
LEMONS Australia (NSW), 1990 (Lisbon)	EC haloxyfop-ethoxyethyl	0.83		100	1	28 58 87	fruits	< 0.05 (2) < 0.05 (2) < 0.05	GHF-P 1110
LEMONS New Zealand, 1991 (Meyer)	EC haloxyfop-ethoxyethyl	0.42 directed [a]		1000	1	28	fruits	< 0.05	GHF-P 1147
LEMONS New Zealand, 1991 (Meyer)	EC haloxyfop-ethoxyethyl	0.83 directed [a]		1000	1	28	fruits	< 0.05	GHF-P 1147

CITRUS FRUITS	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
LEMONS New Zealand, 1991 (Meyer)	EC + oil haloxyfop-P-methyl	0.21 directed [a]		1000	1	28	fruits	< 0.05	GHF-P 1147
LEMONS New Zealand, 1991 (Meyer)	EC + oil haloxyfop-P-methyl	0.42 directed [a]		1000	1	28	fruits	< 0.05	GHF-P 1147
LEMONS New Zealand, 1991 (Meyer)	WG + oil haloxyfop-P-methyl	0.21 directed [a]		1000	1	28	fruits	< 0.05	GHF-P 1147
LEMONS New Zealand, 1991 (Meyer)	WG+ oil haloxyfop-P-methyl	0.42 directed [a]		1000	1	28	fruits	< 0.05	GHF-P 1147
ORANGE Brazil (SP), 1985–1986 (Pera Rio)	EC + oil haloxyfop-methyl	0.24 directed [a]			1	206	fruit	< 0.1	GHB-P 040
ORANGE Brazil (SP), 1985–1986 (Pera Rio)	EC + oil haloxyfop-methyl	0.48 directed [a]			1	206	fruit	< 0.1	GHB-P 040
ORANGE Brazil (SP), 1985–1986 (Pera Rio)	EC + oil haloxyfop-methyl	0.72 directed [a]			1	206	fruit	< 0.1	GHB-P 040
ORANGE Brazil (SP), 1985–1986 (Pera Rio)	EC + oil haloxyfop-methyl	0.96 directed [a]			1	206	fruit	< 0.1	GHB-P 040
ORANGE Brazil (SP), 1985–1986 (Pera Rio)	EC + oil haloxyfop-methyl	1.44 directed [a]			1	206	fruit	< 0.1	GHB-P 040
ORANGE Brazil (SP), 1985–1986 (Pera Rio)	EC + oil haloxyfop-methyl	1.94 directed [a]			1	206	fruit	< 0.1	GHB-P 040
ORANGES Italy, 1991 (Moro)	EC haloxyfop-P-methyl	0.16 directed [a]			1	56	orange peel orange pulp	< 0.02 (2) < 0.02 (2)	GHE-P-2771 R91-28B
ORANGES Italy, 1991 (Navelina)	EC haloxyfop-P-methyl	0.16 directed [a]			1	56	orange peel orange pulp	< 0.02 (2) < 0.02 (2)a6	GHE-P-2771 R91-28A

[a] Directed application for grass control.

Table 14 Haloxyfop residues in apples resulting from supervised trials in Australia, Italy, New Zealand and USA

APPLES	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
Australia (Vic), 1991 (Granny Smith)	EC haloxyfop-ethoxyethyl	0.42 directed [a]		220	1	24 56	apples	< 0.05 < 0.05	GHF-P 1121
Australia (Vic), 1991 (Granny Smith)	EC haloxyfop-ethoxyethyl	0.83 directed [a]		220	1	24 56	apples	< 0.05 < 0.05	GHF-P 1121
Italy, 1987 (Early)	EC haloxyfop-ethoxyethyl	0.10 directed [a]			1	132	apples	< 0.02	GHE-P-1965 trial RT 12^{a}87
Italy, 1987 (Early)	EC haloxyfop-ethoxyethyl	0.21 directed [a]/			1	132	apples	< 0.02	GHE-P-1965 trial RT 12^{a}87

APPLES	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
Italy, 1987 (Early)	EC haloxyfop-ethoxyethyl	0.42 directed [a]			1	132	apples	< 0.02	GHE-P-1965 trial RT 12^{a}87
Italy, 1987 (Golden Delicious)	EC haloxyfop-ethoxyethyl	0.10 directed [a]			1	126	apples	< 0.02	GHE-P-1965 trial RT 12^{b}87
Italy, 1987 (Golden Delicious)	EC haloxyfop-ethoxyethyl	0.21 directed [a]			1	126	apples	< 0.02	GHE-P-1965 trial RT 12^{b}87
Italy, 1987 (Golden Delicious)	EC haloxyfop-ethoxyethyl	0.42 directed [a]			1	126	apples	< 0.02	GHE-P-1965 trial RT 12^{b}87
New Zealand, 1986 (Granny Smith)	EC haloxyfop-ethoxyethyl + crop oil	0.21 directed [a]			1	29	apples	< 0.01	GHF-O-584
New Zealand, 1986 (Granny Smith)	EC haloxyfop-ethoxyethyl + crop oil	0.21 directed [a]			1	29	apples	< 0.01	GHF-O-584
USA (CA), 1987 (Golden Delicious)	WG haloxyfop-methyl	0.56 directed [a]		190	1	60	apples	< 0.05	GH-C 2016
USA (CA), 1987 (Golden Delicious)	EC haloxyfop-methyl	0.28 directed [a]		190	1	60	apples	< 0.05	GH-C 2016
USA (CA), 1987 (Rome)	WG haloxyfop-methyl	0.56 directed [a]		190	1	59	apples	< 0.05	GH-C 2016
USA (CA), 1987 (Rome)	EC haloxyfop-methyl	0.28 directed [a]		190	1	59	apples	< 0.05	GH-C 2016
USA (MI), 1987 (Golden Delicious)	WG haloxyfop-methyl	0.28 directed [a]		190	1	60	apples	< 0.05	GH-C 2016
USA (MI), 1987 (Golden Delicious)	EC haloxyfop-methyl	0.28 directed [a]		190	1	60	apples	< 0.05	GH-C 2016
USA (MI), 1987 (Macintosh)	WG haloxyfop-methyl	0.28 directed [a]		190	1	60	apples	< 0.05	GH-C 2016
USA (MI), 1987 (Macintosh)	EC haloxyfop-methyl	0.28 directed [a]		190	1	60	apples	< 0.05	GH-C 2016

[a] Directed application for grass control.

Table 15 Haloxyfop residues in peaches resulting from supervised trials in Australia

PEACH	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
Australia (Vic), 1991 (Golden Queen)	EC haloxyfop-ethoxyethyl	0.42 directed [a]		220	1	24 56	peaches	< 0.05 < 0.05	GHF-P 1122

PEACH	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
Australia (Vic), 1991 (Golden Queen)	EC haloxyfop-ethoxyethyl	0.83 directed [a]		220	1	24 56	peaches	< 0.05 < 0.05	GHF-P 1122

[a] Directed application for grass control.

Table 16 Haloxyfop residues in grapes resulting from supervised trials in Australia, France and Italy.

GRAPES	Application					PHI	Commodity [a]	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
Australia (NSW), 1990 (Burger)	EC haloxyfop-ethoxyethyl	0.21 directed [b]		100	1	21 49	grapes grapes	< 0.05 < 0.05	GHF-P 1150
Australia (NSW), 1990 (Burger)	EC haloxyfop-ethoxyethyl	0.42 directed [b]		100	1	21 49	grapes grapes	< 0.05 < 0.05	GHF-P 1150
Australia (NSW), 1990 (Burger)	EC haloxyfop-ethoxyethyl	0.83 directed [b]		100	1	21 49	grapes grapes	< 0.05 < 0.05	GHF-P 1150
Australia (NSW), 1990 (Cabernet Sauvignon)	EC haloxyfop-ethoxyethyl	0.21 directed [b]		100	1	29 56	grapes grapes	< 0.05 < 0.05	GHF-P 1150
Australia (NSW), 1990 (Cabernet Sauvignon)	EC haloxyfop-ethoxyethyl	0.42 directed [b]		100	1	29 56	grapes grapes	< 0.05 < 0.05	GHF-P 1150
Australia (NSW), 1990 (Cabernet Sauvignon)	EC haloxyfop-ethoxyethyl	0.83 directed [b]		100	1	29 56	grapes grapes	< 0.05 < 0.05	GHF-P 1150
France, 1983 (Cabernay)	EC haloxyfop-ethoxyethyl	0.21 directed [b]		650	1	93	grapes	< 0.01 (4)	GHE-P-1148 RT 277-7/83
France, 1983 (Cabernay)	EC haloxyfop-ethoxyethyl	0.42 directed [b]		650	1	93	grapes	< 0.01 (4)	GHE-P-1148 RT 277-7/83
France, 1983 (Cabernay)	EC haloxyfop-ethoxyethyl	0.83 directed [b]		650	1	93	grapes	< 0.01 (4)	GHE-P-1148 RT 277-7/83
France, 1983 (Cabernay)	EC haloxyfop-ethoxyethyl	1.7 directed [b]		650	1	93	grapes	< 0.01 (4)	GHE-P-1148 RT 277-7/83
France, 1983 (Cabernay)	EC haloxyfop-ethoxyethyl	0.10 directed [b]		650	1	93	grapes	< 0.01 (4)	GHE-P-1148 RT 277-7/83
France, 1985 (variety not reported)	EC haloxyfop-ethoxyethyl	0.42 directed [b]			1	119	grapes	< 0.01 (4) [c]	GHE-P-1523 RT 30[d]85
France, 1985 (variety not reported)	EC haloxyfop-ethoxyethyl	0.83 directed [b]			1	119	grapes	< 0.01 (4) [c]	GHE-P-1523 RT 30[d]85
France, 1985 (variety not reported)	EC haloxyfop-ethoxyethyl	0.42 directed [b]		400	1	115	grapes	< 0.01 (4) [c]	GHE-P-1523 RT 310/85
France, 1985 (variety not reported)	EC haloxyfop-ethoxyethyl	0.83 directed [b]		400	1	115	grapes	< 0.01 (4) [c]	GHE-P-1523 RT 310/85

GRAPES	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	[a]	mg/kg	
France, 1985 (variety not reported)	EC haloxyfop-ethoxyethyl	0.42 directed [b]		300	1	86	grapes	< 0.01 (4) [c]	GHE-P-1523 RT 31^{a}85
France, 1985 (variety not reported)	EC haloxyfop-ethoxyethyl	0.83 directed [b]		300	1	86	grapes	< 0.01 (4) [c]	GHE-P-1523 RT 31^{a}85
Italy, 1989 (Dolcetto)	EC haloxyfop-P-methyl	0.21 directed [b]		400	1	51	grapes	< 0.05 [d]	GHE-P-2115 R89-31C
Italy, 1989 (Malvasia)	EC haloxyfop-P-methyl	0.21 directed [b]		400	1	67	grapes	< 0.05 [d]	GHE-P-2115 R89-31A
Italy, 1989 (Trebbiano)	EC haloxyfop-P-methyl	0.21 directed [b]		400	1	63	grapes	< 0.05 [d]	GHE-P-2115 R89-31B

[a] GHF-P 1150: Grape berries, stalks removed.

[b] Directed application for grass control.

[c] GHE-P-1523: 4 replicate samples.

[d] GHE-P-2115. Method ERC 89.3 was modified. The methanolic NaOH extraction step was replaced by a solvent extraction step. Conjugates, if present, may not be included.

Table 17 Haloxyfop residues in bananas resulting from supervised trials in Australia

BANANAS	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha [b]	kg ai/hL	water (L/ha)	no.	days	[a]	mg/kg	
Australia (Qld), 1990 (Cavendish)	EC haloxyfop-ethoxyethyl	0.83 directed [c]		167	1	14	bananas	< 0.05	GHF-P 1149
Australia (Qld), 1990 (Cavendish)	WG haloxyfop-P-methyl	0.42+oil directed [c]		167	1	7 14	bananas bananas	< 0.05 < 0.05	GHF-P 1149

[a] Banana bunches were bagged.

[b] GHF-P 1149: A QA audit could not verify the application rate from the data supplied. The QA audit report stated that application rates may be 30.7% higher than stated in the report.

[c] Directed application for grass control.

Table 18 Haloxyfop residues in onions resulting from supervised trials in Belgium, France, Germany and New Zealand

ONIONS	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		[a]	
Belgium, 2000 (Hygro)	EC haloxyfop-P-methyl	0.10	BBCH 17-41	310	1	28	onions (whole plant)	0.06	GHE-P-8870 CEMS-1269B
Belgium, 2000 (Sumith)	EC haloxyfop-P-methyl	0.10	BBCH 17-41	300	1	28	onions (whole plant)	0.12	GHE-P-8870 CEMS-1269A
France, 2000 (Doré de Parme)	EC haloxyfop-P-methyl	0.16	BBCH 13	320	1	90	onions (whole plant)	< 0.02	GHE-P-8871 CEMS-1270A

ONIONS country, year (variety)	Application					PHI days	Commodity	haloxyfop, mg/kg [a]	Ref
	Form	kg ai/ha	growth stage	water (L/ha)	no.				
France, 2000 (Doré de Parme)	EC haloxyfop-P-methyl	0.16	BBCH 13	320	1	90	onions (whole plant)	< 0.02	GHE-P-8871 CEMS-1270B
France, 2000 (RF 18)	EC haloxyfop-P-methyl	0.10	BBCH 45	300	1	28	onions (whole plant)	< 0.02	GHE-P-8870 CEMS-1269C
France, 2000 (RF 18)	EC haloxyfop-P-methyl	0.10	BBCH 45	280	1	28	onions (whole plant)	0.03	GHE-P-8870 CEMS-1269D
Germany, 1999 (Cardas)	EC haloxyfop-P-methyl	0.10	BBCH 45	400	1	28	onions	0.04	GHE-P-8309 R99-122A
Germany, 1999 (Duralo)	EC haloxyfop-P-methyl	0.10	BBCH 47	400	1	27	onions	0.09	GHE-P-8309 R99-122D
Germany, 1999 (Elsa)	EC haloxyfop-P-methyl	0.10	BBCH 43	400	1	26	onions	0.02	GHE-P-8309 R99-122B
Germany, 1999 (Hydon)	EC haloxyfop-P-methyl	0.10	BBCH 45	400	1	26	onions	0.03	GHE-P-8309 R99-122C
New Zealand, 1987 (Pukehoe Long Keeper)	EC + oil haloxyfop-ethoxyethyl	0.052	semi-mature bulb with green leaves	200	1	30	onion bulb	0.01	GHF-P-633
New Zealand, 1987 (Pukehoe Long Keeper)	EC + oil haloxyfop-ethoxyethyl	0.21	semi-mature bulb with green leaves	200	1	30	onion bulb	0.06	GHF-P-633
New Zealand, 1987 (Pukehoe Long Keeper)	EC + oil haloxyfop-ethoxyethyl	0.052	mature bulbs with tops beginning to collapse	200	1	15	onion bulb	< 0.01	GHF-P-633
New Zealand, 1987 (Pukehoe Long Keeper)	EC + oil haloxyfop-ethoxyethyl	0.10	mature bulbs with tops beginning to collapse	200	1	15	onion bulb	0.01	GHF-P-633
New Zealand, 1987 (Pukehoe Long Keeper)	EC + oil haloxyfop-ethoxyethyl	0.21	mature bulbs with tops beginning to collapse	200	1	15	onion bulb	0.03	GHF-P-633
New Zealand, 1987 (Pukehoe Long Keeper)	EC + oil haloxyfop-ethoxyethyl	0.10	semi-mature bulb with green leaves	200	1	30	onion bulb	0.02	GHF-P-633
Spain, 2000 (Babosa)	EC haloxyfop-P-methyl	0.15	BBCH 13	310	1	50	onions (whole plant)	< 0.02	GHE-P-8871 CEMS-1270D
Spain, 2000 (Tardia de Lerida)	EC haloxyfop-P-methyl	0.15	BBCH 13	320	1	58	onions (whole plant)	< 0.02	GHE-P-8871 CEMS-1270C

[a] GHF-P-633: Bulb after removal of roots, tops and loose parchment skin.

GHE-P-8309: Roots and leaves discarded.

Bulb vegetables, BBCH growth stages (Meier, 2001)

13 3rd leaf (> 3cm)

17 7th leaf

41 Leaf bases begin to thicken or extend.

43 30% of the expected bulb or shaft diameter reached.

45 50% of the expected bulb or shaft diameter reached.

47 Bolting begins; in 10% of the plants leaves bent over.

Table 19 Haloxyfop residues in field beans resulting from supervised trials in Belgium, France, Germany, Greece and Spain

FIELD BEANS	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]	mg/kg	
Belgium, 2000 (Kylian)	EC haloxyfop-P-methyl	0.10	BBCH 50-51	320	1	28	whole pod	0.09	GHE-P-8849 CEMS-1246C
Belgium, 2000 (Proton)	EC haloxyfop-P-methyl	0.10	BBCH 50-51	300	1	29	whole pod	0.09	GHE-P-8849 CEMS-1246D
France, 1999 (Booster)	EC haloxyfop-P-methyl	0.10	BBCH 51	350	1	28	whole pod	0.03	GHE-P-8577 R99-167A
France, 1999 (Filao)	EC haloxyfop-P-methyl	0.10	BBCH 55	336	1	28	whole pod	0.06	GHE-P-8559 99 H CL DO P21 (B)
France, 1999 (Firato)	EC haloxyfop-P-methyl	0.099	BBCH 14	258	1	27	whole pod	< 0.02	GHE-P-8559 99 H CL DO P22 (C)
France, 1999 (Lipsos)	EC haloxyfop-P-methyl	0.098	BBCH 59	344	1	26	whole pod	0.26	GHE-P-8559 99 H CL DO P20 (A)
France, 1999 (Tasman)	EC haloxyfop-P-methyl	0.10	BBCH 24	236	1	25	whole pod	0.07	GHE-P-8559 99 H CL DO P23 (D)
France, 2000 (Autant)	EC haloxyfop-P-methyl	0.10	BBCH 51	270	1	28	whole pod	0.10	GHE-P-8849 CEMS-1246B
France, 2000 (Sonore)	EC haloxyfop-P-methyl	0.10	BBCH 59	200	1	29	whole pod	0.19	GHE-P-8849 CEMS-1246A
France, 2001 (Booster)	EC haloxyfop-P-methyl	0.10	BBCH 14	290	1	29	whole pod	< 0.02	GHE-P-9783 CEMS-1570A
Germany, 1990 (Alfred)	EC haloxyfop-P-methyl	0.052	EC 29, 6 leaves	400	1	76 76	green beans green pods	< 0.02 < 0.02	GHE-P-2444 R90-8A
Germany, 1990 (Alfred)	EC haloxyfop-P-methyl	0.10	EC 29, 6 leaves	400	1	76 76	green beans green pods	< 0.02 < 0.02	GHE-P-2444 R90-8A
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.052	EC 26, 4-5 leaves	400	1	70 70	green beans green pods	< 0.02 < 0.02	GHE-P-2444 R90-8B
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.10	EC 26, 4-5 leaves	400	1	70 70	green beans green pods	< 0.02 < 0.02	GHE-P-2444 R90-8B
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.052	EC 25, 4 leaves	400	1	80 80	green beans green pods	< 0.02 < 0.02	GHE-P-2444 R90-8C
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.10	EC 25, 4 leaves	400	1	80 80	green beans green pods	< 0.02 < 0.02	GHE-P-2444 R90-8C
Germany, 1990 (Treu)	EC haloxyfop-P-methyl	0.052	EC 61, begin flowering	400	1	62 62	green beans green pods	0.06 < 0.02	GHE-P-2444 R90-8D

FIELD BEANS	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]	mg/kg	
Germany, 1990 (Treu)	EC haloxyfop-P-methyl	0.10	EC 61, begin flowering	400	1	62 62	green beans green pods	0.16 0.02	GHE-P-2444 R90-8D
Greece, 2001 (Green Crop)	EC haloxyfop-P-methyl	0.10	BBCH 50	300	1	28	whole pod	0.18	GHE-P-9783 CEMS-1570B
Spain, 1999 (Tabella)	EC haloxyfop-P-methyl	0.10	BBCH 65	400	1	28	whole pod	0.22	GHE-P-8577 R99-167B

[a] Green beans: fresh bean seed without pods.

Beans, BBCH growth stages (Meier, 2001)

14 Fourth true leaf (second trifoliate leaf) unfolded.

24 Fourth side shoot visible .

51 First flower buds visible.

55 First flower buds enlarged.

59 First petals visible, flowers still closed.

65 Full flowering: 50% of flowers open.

Table 20 Haloxyfop residues in peas (legume vegetables) resulting from supervised trials in Belgium, France, Italy and Spain

PEAS	Application [a]					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		mg/kg	
Belgium, 2000 (Vegetable peas, Sigra)	EC haloxyfop-P-methyl	0.10	BBCH 50	300	1	34	peas empty pod peas in pod	0.09 0.12 (0.11) [d]	GHE-P-8851 CEMS-1248D
Belgium, 2000 (Vegetable peas, Style)	EC haloxyfop-P-methyl	0.10	BBCH 50	300	1	31	peas empty pod peas in pod	0.04 0.09 (0.07) [d]	GHE-P-8851 CEMS-1248C
France, 1999 (Vegetable peas, Augusto)	EC haloxyfop-P-methyl	0.10	BBCH 61	290	1	28	peas pod peas in pod	< 0.01 0.38 0.14 [b]	GHE-P-8573 R99-133A
France, 1999 (Vegetable peas, Koka)	EC haloxyfop-P-methyl	0.10	BBCH 61	310	1	25	peas pod peas in pod	0.29 0.36 0.32 [b]	GHE-P-8573 R99-133D
France, 1999 (Vegetable peas, Nina)	EC haloxyfop-P-methyl	0.10	BBCH 61	300	1	27	peas pod peas in pod	0.32 0.32 0.32 [b]	GHE-P-8573 R99-133B
France, 1999 (Vegetable peas, Nina)	EC haloxyfop-P-methyl	0.10	BBCH 61	280	1	27	peas pod peas in pod	< 0.05 0.09 0.07 [b]	GHE-P-8573 R99-133C
France, 1999 (Vegetable peas, Salsado)	EC haloxyfop-P-methyl	0.10	BBCH 65	300	1	25	peas pod peas in pod	0.26 0.09 0.21 [b]	GHE-P-8578 R99-168A
France, 2000 (Vegetable peas, Bonette)	EC haloxyfop-P-methyl	0.10	BBCH 55-59	200	1	31	peas empty pod peas in pod	0.07 0.08 (0.08) [d]	GHE-P-8851 CEMS-1248A
France, 2000 (Vegetable peas, Bonette)	EC haloxyfop-P-methyl	0.10	BBCH 61	250	1	22	peas empty pod peas in pod	0.44 0.42 (0.43) [d]	GHE-P-8851 CEMS-1248B

PEAS country, year (variety)	Application [a]					PHI days	Commodity	haloxyfop mg/kg	Ref
	Form	kg ai/ha	growth stage	water (L/ha)	no.				
France, 2000 (Vegetable peas, variety not recorded)	EC haloxyfop-P-methyl	0.10	BBCH 51-60	410	1	29	peas empty pod	0.15 < 0.05	GHE-P-8852 CEMS-1249A
France, 2004 (Vegetable peas, Austin)	EC haloxyfop-P-methyl	0.10	BBCH 50	300	1	60	peas empty pod peas in pod	0.07 0.09 (0.08) [d]	GHE-P-11073 CEMS-2339B Note [c]
Italy, 2000 (Vegetable peas, Lambado)	EC haloxyfop-P-methyl	0.10	BBCH 50	420	1	28	peas empty pod peas in pod	< 0.05 < 0.05 (< 0.05) [d]	GHE-P-8852 CEMS-1249B
Italy, 2004 (Vegetable peas, Corallo)	EC haloxyfop-P-methyl	0.10	BBCH 50	300	1	36	peas empty pod peas in pod	0.05 0.05 (0.05) [d]	GHE-P-11073 CEMS-2339A Note [c]
Italy, 2004 (Vegetable peas, Milor)	EC haloxyfop-P-methyl	0.10	BBCH 49-50	310	1	31	peas empty pod peas in pod	0.05 0.09 (0.07) [d]	GHE-P-11073 CEMS-2339C Note [c]
Spain, 1999 (Vegetable peas, Mastin)	EC haloxyfop-P-methyl	0.10	BBCH 59-60	400	1	28	peas pod peas in pod	0.75 0.17 0.53 [b]	GHE-P-8578 R99-168B
Spain, 2004 (Vegetable peas, Globo)	EC haloxyfop-P-methyl	0.10	BBCH 50	300	1	28	peas empty pod peas in pod	0.12 0.12 (0.12) [d]	GHE-P-11073 CEMS-2339D Note [c]

[a] Spray application.

GHE-P-8578, GHE-P-8573: broadcast application. Interpretation: the spray was applied over the whole area, including the crop.

GHE-P-8851, GHE-P-8852, GHE-P-11073: foliar application.

[b] Residue in peas-in-pod calculated from measured residues in peas and pods and relative weights.

[c] GHE-P-11073. Interval of freezer storage before analysis: 22–24 months.

[d] Residue in peas-in-pod estimated as average of measured residues in peas and pods.

Peas, BBCH growth stages (Meier, 2001)

51 First flower buds visible outside leaves.

55 First separated flower buds visible outside leaves, but still closed.

59 First petals visible, flowers still closed.

60 First flowers open (sporadically within the population).

61 Beginning of flowering: 10% of flowers open.

65 Full flowering: 50% of flowers open.

Table 21 Haloxyfop residues in pigeon peas resulting from supervised trials in Australia

PIGEON PEAS country, year (variety)	Application					PHI days	Commodity	haloxyfop mg/kg	Ref
	Form	kg ai/ha	growth stage	water (L/ha)	no.				
Australia (Qld), 1989 (Hunt)	EC haloxyfop-P-methyl	0.038	43 days after sowing	110	1	85	grain	< 0.01	GHF-P 895
Australia (Qld), 1989 (Hunt)	EC haloxyfop-P-methyl	0.075	43 days after sowing	110	1	85	grain	0.01	GHF-P 895
Australia (Qld), 1989 (Hunt)	EC haloxyfop-ethoxyethyl	0.16	43 days after sowing	110	1	85	grain	0.03	GHF-P 895

PIGEON PEAS country, year (variety)	Application					PHI	Commodity	haloxyfop	Ref
	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		mg/kg	
Australia (Qld), 1989 (Hunt)	EC haloxyfop-ethoxyethyl	0.31	43 days after sowing	110	1	85	grain	0.05	GHF-P 895

Table 22 Haloxyfop residues in beans (pulses) resulting from supervised trials in Argentina, Brazil, Costa Rica and Germany.

BEANS (PULSES) country, year (variety)	Application [c]					PHI	Commodity	haloxyfop, mg/kg	Ref
	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]	reps, field sample	
Argentina (Salta), 1992 (White bean, Alubia/Inta Cerrilos) Note [b] Note [e]	EC haloxyfop-P-methyl	0.06	PE 55 days	200	1	30	beans	0.09 0.10 0.12	GHB-P 283
		0.06	PE 45 days	200	1	43	beans	0.27 0.22 0.14	
		0.06	PE 35 days	200	1	52	beans	0.07 0.06 0.08 c 0.29 < 0.01 (2)	
Argentina (Salta), 1992 (White bean, Alubia/Inta Cerrilos) Note [b] Note [e]	EC haloxyfop-P-methyl	0.12	PE 55 days	200	1	30	beans	0.30 0.30 0.31	GHB-P 283
		0.12	PE 45 days	200	1	43	beans	0.39 0.42 0.30	
		0.12	PE 35 days	200	1	52	beans	0.09 0.10 0.21 c 0.29 < 0.01 (2)	
Argentina (Salta), 1993 (Black bean, JEO 8702 N) Note [b] Note [e]	EC haloxyfop-P-methyl	0.06	PE 55 days	180	1	89	beans	0.42 0.58	GHB-P 283
		0.06	PE 45 days	180	1	98	beans	0.33 0.76 0.24	
		0.06	PE 35 days	180	1	110	beans	0.22 0.26 0.24 c 0.02 < 0.01 (2)	
Argentina (Salta), 1993 (Black bean, JEO 8702 N) Note [b] Note [e]	EC haloxyfop-P-methyl	0.12	PE 55 days	180	1	89	beans	0.87 0.84	GHB-P 283
		0.12	PE 45 days	180	1	98	beans	0.84 0.73 0.84	
		0.12	PE 35 days	180	1	110	beans	0.26 0.26 0.24 c 0.02 < 0.01 (2)	
Argentina (Salta), 1998 (White beans, alubia cerrillos)	EC haloxyfop-P-methyl	0.040	BBCH 13-14 and 16 [d]	200	1	63 72	beans	0.26 0.24 0.19 0.09 0.10 0.16 c 0.02 < 0.01 0.05	GHB-P 402
Argentina (Salta), 1998 (White beans, alubia cerrillos)	EC haloxyfop-P-methyl	0.060	BBCH 13-14 and 16 [d]	200	1	63 72	beans	0.41 0.27 0.24 0.24 0.18 0.20 c 0.02 < 0.01 0.05	GHB-P 402
Argentina (Salta), 1998 (White beans, alubia cerrillos)	EC haloxyfop-P-methyl	0.080	BBCH 13-14 and 16 [d]	200	1	63 72	beans	0.65 0.45 0.42 0.30 0.29 0.22 c 0.02 < 0.01 0.05	GHB-P 402
Argentina (Salta), 1998 (White beans, alubia cerrillos)	EC haloxyfop-P-methyl	0.12	BBCH 13-14 and 16 [d]	200	1	63 72	beans	1.1 1.1 1.5 0.62 0.57 0.40 c 0.02 < 0.01 0.05	GHB-P 402
Argentina (Salta), 1998 (White beans, alubia cerrillos)	EC haloxyfop-P-methyl	0.16	BBCH 13-14 and 16 [d]	200	1	63 72	beans	0.88 1.5 0.98 0.67 0.64 0.46 c 0.02 < 0.01 0.05	GHB-P 402
Argentina (Santiago del Estero), 1992 (Black bean, BAT 304) Note [b] Note [e]	EC haloxyfop-P-methyl	0.06	PE 55 days	200	1	42	beans	1.6 1.1 0.87	GHB-P 283
		0.06	PE 45 days	200	1	50	beans	0.91 1.2 0.98	
		0.06	PE 35 days	200	1	56	beans	0.29 0.31 0.33 c 0.02 < 0.01 (2)	
Argentina (Santiago del Estero), 1992 (Black bean, BAT 304) Note [b] Note [e]	EC haloxyfop-P-methyl	0.12	PE 55 days	200	1	42	beans	1.7 2.2 1.8)	GHB-P 283
		0.12	PE 45 days	200	1	50	beans	1.8 2.0 1.7	
		0.12	PE 35 days	200	1	56	beans	0.60 0.22 1.6 c 0.02 < 0.01 (2)	

BEANS (PULSES) country, year (variety)	Application [c] Form	 kg ai/ha	 growth stage	 water (L/ha)	 no.	PHI days	Commodity Note [a]	haloxyfop, mg/kg reps, field sample	Ref
Argentina (Santiago del Estero), 1993 (Black bean, XAN 112) Note [b] Note [e]	EC haloxyfop-P-methyl	0.06	PE 55 days	180	1	62	beans	0.80 0.58 0.49	GHB-P 283
		0.06	PE 45 days	180	1	70	beans	0.43 0.54 0.44	
		0.06	PE 35 days	180	1	82	beans	0.12 0.14 0.18 c 0.03 < 0.01 (2)	
Argentina (Santiago del Estero), 1993 (Black bean, XAN 112) Note [b] Note [e]	EC haloxyfop-P-methyl	0.12	PE 55 days	180	1	62	beans	1.8 1.1	GHB-P 283
		0.12	PE 45 days	180	1	70	beans	0.71 0.38 1.2	
		0.12	PE 35 days	180	1	82	beans	0.44 0.27 0.27 c 0.03 < 0.01 (2)	
Argentina (Tucumán), 1992 (White bean, Alubia/Inta Cerrilos) Note [b] Note [e]	EC haloxyfop-P-methyl	0.06	PE 55 days	200	1	31	beans	0.36 0.35 0.27	GHB-P 283
		0.06	PE 45 days	200	1	41	beans	0.52 0.51 0.31	
		0.06	PE 35 days	200	1	52	beans	0.27 0.14 0.23 c 1.6 0.03 0.01	
Argentina (Tucumán), 1992 (White bean, Alubia/Inta Cerrilos) Note [b] Note [e]	EC haloxyfop-P-methyl	0.12	PE 55 days	200	1	31	beans	0.25 0.27 0.33	GHB-P 283
		0.12	PE 45 days	200	1	41	beans	0.53 0.56 0.69	
		0.12	PE 35 days	200	1	52	beans	0.39 0.26 0.27 c 1.6 0.03 0.01	
Argentina (Tucumán), 1993 (Black bean, NAG 12) Note [b] Note [e]	EC haloxyfop-P-methyl	0.06	PE 55 days	180	1	46	beans	0.53 0.59 0.60	GHB-P 283
		0.06	PE 45 days	180	1	61	beans	0.66 0.70 0.22	
		0.06	PE 35 days	180	1	67	beans	0.04 0.02 0.03	
Argentina (Tucumán), 1993 (Black bean, NAG 12) Note [b] Note [e]	EC haloxyfop-P-methyl	0.12	PE 55 days	180	1	46	beans	1.0 1.4 0.84	GHB-P 283
		0.12	PE 45 days	180	1	61	beans	0.75 0.86 0.58	
		0.12	PE 35 days	180	1	67	beans	0.07 0.06 0.07	
Brazil (Parana), 1997 (Dry beans, Iapar-44)	EC haloxyfop-P-methyl	0.048	BBCH 12-14 [b]	200	1	77 88 97	beans beans beans	0.07 0.06 0.08 0.03 0.03 0.02 < 0.01 (2) 0.01	GHB-P 378
Brazil (Parana), 1997 (Dry beans, Iapar-44)	EC haloxyfop-P-methyl	0.096	BBCH 12-14 [b]	200	1	77 88 97	beans beans beans	0.16 0.36 0.14 0.08 0.14 0.04 0.03 0.02 0.02	GHB-P 378
Brazil (Parana), 1998 (Dry beans, Carioca)	EC haloxyfop-P-methyl	0.048	BBCH 12-14 [b]	200	1	59 66 77	beans	0.09 0.08 0.09 0.03 0.02 0.02 < 0.01 (3)	GHB-P 391
Brazil (Parana), 1998 (Dry beans, Carioca)	EC haloxyfop-P-methyl	0.096	BBCH 12-14 [b]	200	1	59 66 77	beans	0.12 0.12 0.16 0.04 0.04 0.04 < 0.01 (2) 0.01	GHB-P 391
Brazil (Parana), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.06	PE: 25 days	200	1	61	beans	0.03 0.03	GHB-P 713
Brazil (Parana), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.12	PE: 25 days	200	1	61	beans	0.06 0.06	GHB-P 713
Brazil (Parana), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.06	PE: 32 days	200	1	64	beans	0.11 0.23	GHB-P 713
Brazil (Parana), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.12	PE: 32 days	200	1	64	beans	0.08 0.06	GHB-P 713
Brazil (Parana), 2000 Dry beans, Pérola)	EC + oil haloxyfop-P-methyl	0.06	PE: 25 days	200	1	55	beans	0.49 0.20	GHB-P 732

BEANS (PULSES)	Application [c]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]	reps, field sample	
Brazil (Parana), 2000 Dry beans, Pérola)	EC + oil haloxyfop-P-methyl	0.12	PE: 25 days	200	1	55	beans	0.30 0.42	GHB-P 732
Brazil (Parana), 2000 Dry beans, Pérola)	EC + oil haloxyfop-P-methyl	0.06	PE: 32 days	200	1	48	beans	0.34 0.52	GHB-P 732
Brazil (Parana), 2000 Dry beans, Pérola)	EC + oil haloxyfop-P-methyl	0.12	PE: 32 days	200	1	48	beans	0.76 0.92	GHB-P 732
Brazil (SP), 1996–1997 (Dry beans, IAC Carioca)	EC haloxyfop-P-methyl	0.048	BBCH 12-14 [b]	200	1	50 57	beans beans	0.02 0.04 0.04 0.06 0.03 0.03 0.03 0.02	GHB-P 377
Brazil (SP), 1996–1997 (Dry beans, IAC Carioca)	EC haloxyfop-P-methyl	0.096	BBCH 12-14 [b]	200	1	50 57	beans beans	0.13 0.13 0.10 0.14 0.02 0.03 0.03 0.02	GHB-P 377
Brazil (SP), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.06	PE: 25 days	200	1	36 43 50 57 64	beans	0.01 0.02 < 0.01 0.01 0.03 0.04 0.03 0.04 0.03 0.02	GHB-P 713
Brazil (SP), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.12	PE: 25 days	200	1	36 43 50 57 64	beans	0.03 0.04 0.03 0.07 0.04 0.05 0.06 0.03 0.05 0.07	GHB-P 713
Brazil (SP), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.06	PE: 32 days	200	1	43 50 57 64 71	beans	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	GHB-P 713
Brazil (SP), 2000 Dry beans, Carioquinha)	EC + oil haloxyfop-P-methyl	0.12	PE: 32 days	200	1	43 50 57 64 71	beans	< 0.01 (2) < 0.01 (2) 0.04 0.01 < 0.01 0.01 0.01 < 0.01	GHB-P 713
Brazil (SP), 2001–2002 Dry beans, Carioca pérola)	EC + oil haloxyfop-P-methyl	0.06	PE: 25 days	200	1	57	beans	0.04 0.04	GHB-P 786
Brazil (SP), 2001–2002 Dry beans, Carioca pérola)	EC + oil haloxyfop-P-methyl	0.12	PE: 25 days	200	1	57	beans	0.08 0.07	GHB-P 786
Brazil (SP), 2001–2002 Dry beans, Carioca pérola)	EC + oil haloxyfop-P-methyl	0.06	PE: 32 days	200	1	57	beans	0.06 0.06	GHB-P 786
Brazil (SP), 2001–2002 Dry beans, Carioca pérola)	EC + oil haloxyfop-P-methyl	0.12	PE: 32 days	200	1	57	beans	0.32 0.32	GHB-P 786
Costa Rica, 1998 (Dry beans, Brunca)	EC haloxyfop-P-methyl	0.060	BBCH 12-14 PS: 20 days	201	1	48	beans	0.03 0.02 0.03 0.02	GHB-P 384
Costa Rica, 1998 (Dry beans, Brunca)	EC haloxyfop-P-methyl	0.12	BBCH 12-14 PS: 20 days	201	1	48	beans	0.03 0.04 0.04 0.05	GHB-P 384
Germany, 1990 (Alfred)	EC haloxyfop-P-methyl	0.052	EC 29, 6 leaves	400	1	91	dried beans	< 0.02	GHE-P-2444 R90-8A

BEANS (PULSES)	Application [c]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]	reps, field sample	
Germany, 1990 (Alfred)	EC haloxyfop-P-methyl	0.10	EC 29, 6 leaves	400	1	91	dried beans	0.02	GHE-P-2444 R90-8A
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.052	EC 26, 4-5 leaves	400	1	98	dried beans	< 0.02	GHE-P-2444 R90-8B
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.10	EC 26, 4-5 leaves	400	1	98	dried beans	< 0.02	GHE-P-2444 R90-8B
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.052	EC 25, 4 leaves	400	1	106	dried beans	< 0.02	GHE-P-2444 R90-8C
Germany, 1990 (Herz Freya)	EC haloxyfop-P-methyl	0.10	EC 25, 4 leaves	400	1	106	dried beans	0.02	GHE-P-2444 R90-8C
Germany, 1990 (Treu)	EC haloxyfop-P-methyl	0.052	EC 61, begin flowering	400	1	72	dried beans	0.11	GHE-P-2444 R90-8D
Germany, 1990 (Treu)	EC haloxyfop-P-methyl	0.10	EC 61, begin flowering	400	1	72	dried beans	0.30	GHE-P-2444 R90-8D

[a] Beans: bean seed separated from pod by threshing.

[b] Separate plots were treated on 3 different occasions and sampled on the same date at crop maturity.

[c] PE: post emergence. PS: post sowing.

[d] Separate plots were treated on 2 different occasions and sampled on the same date at crop maturity.

[e] GHB-P 283. Interval of freezer storage before analysis: 26–40 months.

Beans, BBCH growth stages (Meier, 2001)

12 Two full leaves (first leaf pair unfolded).

13 Third true leaf (first trifoliate leaf) unfolded.

14 Fourth true leaf (second trifoliate leaf) unfolded.

16 Sixth true leaf unfolded.

Table 23 Haloxyfop residues in chickpeas resulting from supervised trials in Australia

CHICKPEAS	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		mg/kg	
Australia (WA), 1997 (Koniva)	EC + oil haloxyfop-P-methyl	0.052	3–7 branch	100	2	118	grain	< 0.02	GHF-P 1718 97355.01
Australia (WA), 1997 (Koniva)	EC + oil haloxyfop-P-methyl	0.10	3–7 branch	100	2	118	grain	< 0.02	GHF-P 1718 97355.01
Australia (SA), 1997 (Desavic)	EC + oil haloxyfop-P-methyl	0.052	some bloom	100	2	109	grain	0.02	GHF-P 1718 97355.02
Australia (SA), 1997 (Desavic)	EC + oil haloxyfop-P-methyl	0.10	some bloom	100	2	109	grain	0.04	GHF-P 1718 97355.02

Table 24 Haloxyfop residues in peas (pulses) resulting from supervised trials in Australia and France

PEAS (PULSES)	Application [a]					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [b]	mg/kg	
Australia (SA), 1989 (Field peas, Alma)	EC haloxyfop-P-methyl	0.052	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (SA), 1989 (Field peas, Alma)	EC + oil haloxyfop-P-methyl	0.052	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (SA), 1989 (Field peas, Alma)	EC haloxyfop-P-methyl	0.10	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (SA), 1989 (Field peas, Alma)	EC + oil haloxyfop-P-methyl	0.10	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (SA), 1989 (Field peas, Alma)	EC haloxyfop-ethoxyethyl	0.10	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (SA), 1989 (Field peas, Alma)	EC + oil haloxyfop-ethoxyethyl	0.10	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (SA), 1989 (Field peas, Alma)	EC haloxyfop-ethoxyethyl	0.21	2 cm and 14 cm	100	2	94	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC haloxyfop-P-methyl	0.052	3 cm, 6–10 cm and 50–100% bloom	100	3	64	peas (grain)	0.26	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC haloxyfop-P-methyl	0.052	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC + oil haloxyfop-P-methyl	0.052	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC haloxyfop-P-methyl	0.10	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC + oil haloxyfop-P-methyl	0.10	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC haloxyfop-ethoxyethyl	0.10	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC + oil haloxyfop-ethoxyethyl	0.10	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC haloxyfop-ethoxyethyl	0.21	3 cm and 6–10 cm	100	2	93	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC haloxyfop-P-methyl	0.052	3 cm	100	1	125	peas (grain)	< 0.01	GHF-P 1392
Australia (Vic), 1989 (Field peas, Dunn)	EC + oil haloxyfop-P-methyl	0.052	3 cm	100	1	125	peas (grain)	< 0.01	GHF-P 1392
France, 1988 (Field peas, Final)	EC haloxyfop-P-methyl	0.052	4 leaf		1	109	dry peas	< 0.05	GHE-P-1966 R88-39 RT 48/88

PEAS (PULSES)	Application [a]					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [b]	mg/kg	
France, 1988 (Field peas, Final)	EC haloxyfop-P-methyl	0.10	4 leaf		1	109	dry peas	< 0.05	GHE-P-1966 R88-39 RT 48/88
France, 1988 (Field peas, Final)	EC haloxyfop-ethoxyethyl	0.10	4 leaf		1	109	dry peas	< 0.05	GHE-P-1966 R88-39 RT 48/88
France, 1988 (Field peas, Final)	EC haloxyfop-ethoxyethyl	0.21	4 leaf		1	109	dry peas	< 0.05	GHE-P-1966 R88-39 RT 48/88
France, 1988 (Field peas, Solara)	EC haloxyfop-P-methyl	0.052	9–10 leaves completely open	300	1	92	dry peas	< 0.05	GHE-P-1966 R88-39 RT 4[d]88
France, 1988 (Field peas, Solara)	EC haloxyfop-P-methyl	0.10	9–10 leaves completely open	300	1	92	dry peas	0.10	GHE-P-1966 R88-39 RT 4[d]88
France, 1988 (Field peas, Solara)	EC haloxyfop-ethoxyethyl	0.10	9–10 leaves completely open	300	1	92	dry peas	0.07	GHE-P-1966 R88-39 RT 4[d]88
France, 1988 (Field peas, Solara)	EC haloxyfop-ethoxyethyl	0.21	9–10 leaves completely open	300	1	92	dry peas	0.14	GHE-P-1966 R88-39 RT 4[d]88
France, 1988 (Kapuciner)	EC haloxyfop-P-methyl	0.052	8–9 leaf		1	39	peas (grain)	< 0.05	GHE-P-1956 R88-40
France, 1988 (Kapuciner)	EC haloxyfop-P-methyl	0.10	8–9 leaf		1	39	peas (grain)	0.06	GHE-P-1956 R88-40
France, 1988 (Kapuciner)	EC haloxyfop-ethoxyethyl	0.10	8–9 leaf		1	39	peas (grain)	< 0.05	GHE-P-1956 R88-40
France, 1988 (Kapuciner)	EC haloxyfop-ethoxyethyl	0.21	8–9 leaf		1	39	peas (grain)	0.11	GHE-P-1956 R88-40
France, 1989 (Atisem)	EC haloxyfop-P-methyl	0.052	flower buds hidden by top foliage	250	1	36	peas (grain)	0.03	GHE-P-2057 R89-7B
France, 1989 (Atisem)	EC haloxyfop-P-methyl	0.10	flower buds hidden by top foliage	250	1	36	peas (grain)	0.05	GHE-P-2057 R89-7B
France, 1989 (Atisem)	EC haloxyfop-ethoxyethyl	0.10	flower buds hidden by top foliage	250	1	36	peas (grain)	0.04	GHE-P-2057 R89-7B
France, 1989 (Atisem)	EC haloxyfop-ethoxyethyl	0.21	flower buds hidden by top foliage	250	1	36	peas (grain)	0.07	GHE-P-2057 R89-7B
France, 1989 (Cador)	EC haloxyfop-ethoxyethyl	0.10	5–6 open leaves	500	1	36	peas (grain)	0.03	GHE-P-2057 R89-7A
France, 1989 (Cador)	EC haloxyfop-ethoxyethyl	0.21	5–6 open leaves	500	1	36	peas (grain)	0.06	GHE-P-2057 R89-7A
France, 1989 (Cador)	EC haloxyfop-P-methyl	0.052	5–6 open leaves	500	1	36	peas (grain)	0.03	GHE-P-2057 R89-7A
France, 1989 (Cador)	EC haloxyfop-P-methyl	0.10	5–6 open leaves	500	1	36	peas (grain)	0.04	GHE-P-2057 R89-7A
France, 1989 (Field peas, Amac)	EC haloxyfop-P-methyl	0.052	6-leaf	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4C

PEAS (PULSES)	Application [a]					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [b]	mg/kg	
France, 1989 (Field peas, Amac)	EC haloxyfop-P-methyl	0.10	6-leaf	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4C
France, 1989 (Field peas, Amac)	EC haloxyfop-ethoxyethyl	0.10	6-leaf	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4C
France, 1989 (Field peas, Amac)	EC haloxyfop-ethoxyethyl	0.21	6-leaf	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4C
France, 1989 (Field peas, Final)	EC haloxyfop-P-methyl	0.052	6 spread out leaves	400	1	80	dry peas	< 0.02	GHE-P-2055 R89-6
France, 1989 (Field peas, Final)	EC haloxyfop-P-methyl	0.10	6 spread out leaves	400	1	80	dry peas	< 0.02	GHE-P-2055 R89-6
France, 1989 (Field peas, Final)	EC haloxyfop-ethoxyethyl	0.10	6 spread out leaves	400	1	80	dry peas	< 0.02	GHE-P-2055 R89-6
France, 1989 (Field peas, Final)	EC haloxyfop-ethoxyethyl	0.21	6 spread out leaves	400	1	80	dry peas	< 0.02	GHE-P-2055 R89-6
France, 1989 (Field peas, Frijaune)	EC haloxyfop-P-methyl	0.052	6–7 spread out leaves	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4B
France, 1989 (Field peas, Frijaune)	EC haloxyfop-P-methyl	0.10	6–7 spread out leaves	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4B
France, 1989 (Field peas, Frijaune)	EC haloxyfop-ethoxyethyl	0.10	6–7 spread out leaves	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4B
France, 1989 (Field peas, Frijaune)	EC haloxyfop-ethoxyethyl	0.21	6–7 spread out leaves	330	1	99	dry peas	< 0.02	GHE-P-2058 R89-4B
France, 1989 (Field peas, Frilene)	EC haloxyfop-P-methyl	0.052	5–6 spread out leaves	330	1	68	dry peas	0.04	GHE-P-2058 R89-4A
France, 1989 (Field peas, Frilene)	EC haloxyfop-P-methyl	0.10	5–6 spread out leaves	330	1	68	dry peas	0.06	GHE-P-2058 R89-4A
France, 1989 (Field peas, Frilene)	EC haloxyfop-ethoxyethyl	0.10	5–6 spread out leaves	330	1	68	dry peas	0.04	GHE-P-2058 R89-4A
France, 1989 (Field peas, Frilene)	EC haloxyfop-ethoxyethyl	0.21	5–6 spread out leaves	330	1	68	dry peas	0.06	GHE-P-2058 R89-4A

[a] Spray application.

GHE-P-2058, GHE-P-2057, GHE-P-2055: Sprayer directed at ground. Interpretation: the spray was directed at the ground beside the row, not over the top of the row.

[b] Commodity analysed.

GHE-P-2058, GHE-P-2055, GHE-P-1966: The field peas were allowed to dry on the plant (in accordance with agricultural practice) and samples of peas were taken for residue analysis at normal harvest. Interpretation: samples analysed were dry peas.

GHE-P-2057: Pod samples were shelled and the peas were homogenized. Interpretation: samples analysed were peas without pod.

GHE-P-1956: Pods shelled by machine. . Interpretation: samples analysed were peas without pod.

Table 25 Haloxyfop residues in soya beans resulting from supervised trials in Argentina, Brazil, France, Germany, Hungary, Italy, Spain and USA

SOYA BEANS	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		[b]	
Argentina, 1990–1991 (Hood 75) El Socorro	EC haloxyfop-P-methyl	0.09	PE 25 days	190	1	134	soya beans	< 0.01	GHB-P 139
		0.09	PE 35days	190	1	121	soya beans	< 0.01	
		0.09	PE 45 days	190	1	108	soya beans	0.03	
Argentina, 1990–1991 (Hood 75) El Socorro	EC haloxyfop-P-methyl	0.18	PE 25 days	190	1	134	soya beans	0.01	GHB-P 139
		0.18	PE 35 days	190	1	121	soya beans	0.01	
		0.18	PE 45 days	190	1	108	soya beans	0.11	
Argentina, 1990–1991 (Hood 75) Las Rosas	EC haloxyfop-P-methyl	0.09	PE 25 days	200	1	133	soya beans	< 0.01	GHB-P 139
		0.09	PE 35days	200	1	118	soya beans	0.01	
		0.09	PE 45 days	200	1	106	soya beans	0.03	
Argentina, 1990–1991 (Hood 75) Las Rosas	EC haloxyfop-P-methyl	0.18	PE 25 days	200	1	133	soya beans	< 0.01	GHB-P 139
		0.18	PE 35days	200	1	118	soya beans	0.02	
		0.18	PE 45 days	200	1	106	soya beans	0.03	
Brazil (GO), 1995 (Doko-RC)	WP haloxyfop-P-methyl	0.060	PE 38 days	200	1	85	soya beans	< 0.01	GHB-P 301
		0.060	PE 43 days	200	1	80	soya beans	< 0.01	
Brazil (GO), 1995 (Doko-RC)	WP haloxyfop-P-methyl	0.12	PE 38 days	200	1	85	soya beans	< 0.01	GHB-P 301
		0.12	PE 43 days	200	1	80	soya beans	< 0.01	
Brazil (PR), 1983–1984 (Bossier) Mandaguari	EC + oil haloxyfop-methyl	0.12	pre-bloom 20–30 cm	300	1	98	soya beans	0.06 0.05 0.05 0.06	GHB-P-024 RT/2^{e}84
Brazil (PR), 1983–1984 (Bossier) Mandaguari	EC + oil haloxyfop-methyl	0.24	pre-bloom 20–30 cm	300	1	98	soya beans	0.12 0.09 0.10 0.11	GHB-P-024 RT/26/84
Brazil (PR), 1983–1984 (Bossier) Mandaguari	EC + oil haloxyfop-methyl	0.12	in bloom 50–60 cm	300	1	78	soya beans	0.90 0.30 0.22 0.23	GHB-P-024 RT/28/84
Brazil (PR), 1983–1984 (Bossier) Mandaguari	EC + oil haloxyfop-methyl	0.24	in bloom 50–60 cm	300	1	78	soya beans	0.62 0.50 0.74 0.71	GHB-P-024 RT/29/84
Brazil (PR), 1983–1984 (Bossier) Marialva	EC + oil haloxyfop-methyl	0.12	pre-bloom ~30 cm	300	1	102	soya beans	< 0.05 (4)	GHB-P-024 RT/3^{b}84
Brazil (PR), 1983–1984 (Bossier) Marialva	EC + oil haloxyfop-methyl	0.24	pre-bloom ~30 cm	300	1	102	soya beans	< 0.05 (4)	GHB-P-024 RT/3^{c}84
Brazil (PR), 1983–84 (Bossier) Marialva	EC +oil haloxyfop-methyl	0.36	pre-bloom ~30 cm	300	1	102	soya beans	0.05 < 0.05 (3)	GHB-P-024 RT/3^{d}84
Brazil (PR), 1983–1984 (Bossier) Marialva	EC + oil haloxyfop-methyl	0.12	in bloom	300	1	77	soya beans	0.16 0.19 0.14 0.12	GHB-P-024 RT/3^{e}84
Brazil (PR), 1983–1984 (Bossier) Marialva	EC + oil haloxyfop-methyl	0.24	in bloom	300	1	77	soya beans	0.33 0.38 0.31 0.39	GHB-P-024 RT/36/84

SOYA BEANS	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		[b]	
Brazil (PR), 1983–1984 (Bossier) Marialva	EC + oil haloxyfop-methyl	0.36	in bloom	300	1	77	soya beans	0.74 0.51 0.45 0.38	GHB-P-024 RT/37/84
Brazil (PR), 1984 (IAC-4) Ponta Grossa	EC + oil haloxyfop-methyl	0.24	pre-bloom 3-leaf	240	1	105	soya beans	< 0.05 (4)	GHB-P-024 RT/1^{c}84
Brazil (PR), 1984 (IAC-4) Ponta Grossa	EC + oil haloxyfop-methyl	0.36	pre-bloom 3-leaf	240	1	105	soya beans	< 0.05 0.05 0.28 0.15	GHB-P-024 RT/1^{d}84
Brazil (PR), 1984 (IAC-4) Ponta Grossa	EC + oil haloxyfop-methyl	0.12	in bloom	240	1	60	soya beans	0.78 0.76 1.8 0.75	GHB-P-024 RT/1^{e}84
Brazil (PR), 1984 (IAC-4) Ponta Grossa	EC + oil haloxyfop-methyl	0.24	in bloom	240	1	60	soya beans	1.0 1.1 0.82 0.22	GHB-P-024 RT/16/84
Brazil (PR), 1984 (IAC-4) Ponta Grossa	EC + oil haloxyfop-methyl	0.36	in bloom	240	1	60	soya beans	1.9 1.4 1.1	GHB-P-024 RT/17/84
Brazil (PR), 1984 (IAC-4) Ponta Grossa	EC + oil haloxyfop-methyl	0.12	pre-bloom 3-leaf	240	1	105	soya beans	< 0.05 (2) 0.06	GHB-P-024 RT/1^{b}84
Brazil (PR), 1991–1992 (Primavera)	EC haloxyfop-P-methyl	0.06	PE	200	1 1 1	112 102 91	soya beans soya beans soya beans	< 0.01 0.01 0.03	GHB-P 144
Brazil (PR), 1991–1992 (Primavera)	EC haloxyfop-P-methyl	0.12	PE	200	1 1 1	112 102 91	soya beans soya beans soya beans	< 0.01 0.02 0.08	GHB-P 144
Brazil (PR), 1994–1995 (BR-4)	WP haloxyfop-P-methyl	0.060	PS 20 days	200	1	105	soya beans	< 0.01	GHB-P 277
		0.060	PS 30 days	200	1	95	soya beans	< 0.01	
		0.060	PS 46 days	200	1	79	soya beans	0.02	
Brazil (PR), 1994–1995 (BR-4)	WP haloxyfop-P-methyl	0.12	PS 20 days	200	1	105	soya beans	< 0.01	GHB-P 277
		0.12	PS 30 days	200	1	95	soya beans	< 0.01	
		0.12	PS 46 days	200	1	79	soya beans	0.03	
Brazil (PR), 2000–2001 (Coodetec 205)	EC + oil haloxyfop-P-methyl	0.060	PE 35 days (5 leaves)	200	1	73 80	soya beans	< 0.01 (2) < 0.01 (2)	GHB-P 714
Brazil (PR), 2000–2001 (Coodetec 205)	EC + oil haloxyfop-P-methyl	0.12	PE 35 days (5 leaves)	200	1	73 80	soya beans	< 0.01 (2) 0.01 0.02	GHB-P 714
Brazil (RS), 2000–2001 (BRS-137)	EC + oil haloxyfop-P-methyl	0.060	PE 35 days (5 leaves)	200	1	73 80	soya beans	< 0.01 (2) < 0.01 (2)	GHB-P 714
Brazil (RS), 2000–2001 (BRS-137)	EC + oil haloxyfop-P-methyl	0.12	PE 35 days (5 leaves)	200	1	73 80	soya beans	< 0.01 (2) < 0.01 (2)	GHB-P 714
Brazil (RS), 2000–2001 (BRS-137)	EC + oil haloxyfop-P-methyl	0.060	PE 35 days (5 leaves)	200	1	70 80 90	soya beans	< 0.01 (2) < 0.01 (2) < 0.01 (2)	GHB-P 717
Brazil (RS), 2000–2001 (BRS-137)	EC + oil haloxyfop-P-methyl	0.12	PE 35 days (5 leaves)	200	1	70 80 90	soya beans	< 0.01 (2) < 0.01 (2) < 0.01 (2)	GHB-P 717
Brazil (SP), 1990 (IAS-5)	EC haloxyfop-P-methyl	0.060	PE 20 days	210	1	88	soya beans	< 0.01	GHB-P 117
		0.060	PE 30days	210	1	78	soya beans	< 0.01	
		0.060	PE 40 days	210	1	68	soya beans	0.01	
Brazil (SP), 1990 (IAS-5)	EC haloxyfop-P-methyl	0.12	PE 20 days	210	1	88	soya beans	< 0.01	GHB-P 117
		0.12	PE 30days	210	1	79	soya beans	0.01	
		0.12	PE 40 days	210	1	69	soya beans	0.02	

SOYA BEANS	Application [a]					PHI	Commodity	haloxyfop, mg/kg [b]	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Brazil (SP), 1991–1992 (Paranaiba)	EC haloxyfop-P-methyl	0.060	PE	200	1	115	soya beans	< 0.01	GHB-P 144
					1	105	soya beans	< 0.01	
					1	95	soya beans	< 0.01	
Brazil (SP), 1991–1992 (Paranaiba)	EC haloxyfop-P-methyl	0.12	PE	200	1	115	soya beans	< 0.01	GHB-P 144
					1	105	soya beans	< 0.01	
					1	95	soya beans	< 0.01	
Brazil (SP), 2000–2001 (Cometa)	EC + oil haloxyfop-P-methyl	0.060	PE 35 days (4–5 leaves)	200	1	52	soya beans	0.05 0.06	GHB-P 721
						59		0.18 0.09	
						66		0.13 0.06	
						73		0.23 0.13	
						80		0.13 0.16	
						87		0.16 0.09	
Brazil (SP), 2000–2001 (Cometa)	EC + oil haloxyfop-P-methyl	0.12	PE 35 days (4–5 leaves)	200	1	52	soya beans	0.11 0.12	GHB-P 721
						59		0.37 0.30	
						66		0.32 0.28	
						73		0.38 0.40	
						80		0.41 0.45	
						87		0.40 0.39	
Brazil (SP), 2001–2002 (FT-Cometa)	EC + oil haloxyfop-P-methyl	0.060	first bloom	200	1	56	soya beans	0.03	GHB-P 790
						63		0.03	
						70		0.05	
						77		0.07	
						84		0.05	
						91		0.06	
Brazil (SP), 2001–2002 (FT-Cometa)	EC + oil haloxyfop-P-methyl	0.12	first bloom	200	1	56	soya beans	0.07	GHB-P 790
						63		0.11	
						70		0.15	
						77		0.12	
						84		0.10	
						91		0.09	
France, 2000 (Queen)	EC haloxyfop-P-methyl	0.10	BBCH 12-13	290	1	90	soya beans	< 0.05	GHE-P-8859 CEMS-1257B
France, 2000 (Queen)	EC haloxyfop-P-methyl	0.10	BBCH 67-69	300	1	60	soya beans	0.31	GHE-P-8859 CEMS-1257B
France, 2000 (Sapporo)	EC haloxyfop-P-methyl	0.10	BBCH 13-14	300	1	90	soya beans	< 0.05	GHE-P-8859 CEMS-1257C
France, 2000 (Sapporo)	EC haloxyfop-P-methyl	0.10	BBCH 65-71	320	1	57	soya beans	0.99	GHE-P-8859 CEMS-1257C
Germany, 2001 (Dorena)	EC haloxyfop-P-methyl	0.11	BBCH 12	310	1	94	soya beans	< 0.05	GHE-P-9782 CEMS-1569B
Germany, 2001 (Dorena)	EC haloxyfop-P-methyl	0.11	BBCH 23	310	1	61	soya beans	0.23	GHE-P-9782 CEMS-1569B
Hungary, 2001 (Borostyan)	EC haloxyfop-P-methyl	0.10	BBCH 12	250	1	91	soya beans	< 0.05	GHE-P-9782 CEMS-1569A
Hungary, 2001 (Borostyan)	EC haloxyfop-P-methyl	0.10	BBCH 33	250	1	60	soya beans	0.11	GHE-P-9782 CEMS-1569A
Italy, 2000 (Silia)	EC haloxyfop-P-methyl	0.10	BBCH 11-19	300	1	86	soya beans	< 0.05	GHE-P-8859 CEMS-1257D
Italy, 2000 (Silia)	EC haloxyfop-P-methyl	0.10	BBCH 67-71	290	1	56	soya beans	0.96	GHE-P-8859 CEMS-1257D

SOYA BEANS	Application [a]					PHI	Commodity	haloxyfop, mg/kg [b]	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Spain, 2000 (Osumi)	EC haloxyfop-P-methyl	0.10	BBCH 19	300	1	90	soya beans	< 0.05	GHE-P-8859 CEMS-1257A
Spain, 2000 (Osumi)	EC haloxyfop-P-methyl	0.10	BBCH 55	300	1	60	soya beans	0.10	GHE-P-8859 CEMS-1257A
USA (AK), 1982 (Davis)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	118 77	soya beans	< 0.05 0.25	GH-C 1625
USA (AL), 1982 (DPL506)	EC+surfactant haloxyfop-methyl	0.28	pre-bloom		1	98	soya beans	0.060	GH-C 1625
USA (AL), 1982 (Essex)	EC+surfactant haloxyfop-methyl	0.28	in bloom		1	90	soya beans	0.40	GH-C 1625
USA (GA), 1982 (Wright)	EC+surfactant haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	104 76	soya beans	0.075 0.82	GH-C 1625
USA (IA), 1982 (Corsoy 79)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	96 75	soya beans	0.081 0.60	GH-C 1625
USA (IL), 1982 (Century)	EC + oil haloxyfop-methyl	0.28	in bloom		1	73	soya beans	2.3	GH-C 1625
USA (IL), 1982 (Northrup King 1492)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	91 69	soya beans	< 0.05 0.49	GH-C 1625
USA (IL), 1982 (Williams 79)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	98 86	soya beans	0.30 0.96	GH-C 1625
USA (IN), 1982 (Pella)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	167 157	soya beans	0.10 0.16	GH-C 1625
USA (KS), 1982 (Essex)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	91 69	soya beans	0.21 1.4	GH-C 1625
USA (LA), 1982 (Davis)	EC haloxyfop-methyl	0.28	pre-bloom full bloom		1 1	125 106	soya beans	0.05 0.43	GH-C 1625
USA (MI), 1982 (Corsoy)	EC + oil haloxyfop-methyl	0.28	in bloom in bloom		1 1	84 82	soya beans	1.8 2.4 2.2 1.9 1.6 2.4 2.6 2.3	GH-C 1625
USA (MN), 1982 (Hodgson 78)	EC + oil haloxyfop-methyl	0.28	in bloom in bloom		1 1	86 74	soya beans	2.0 3.1	GH-C 1625
USA (MS), 1982 (Forrest)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	94 84	soya beans	0.07 0.22 c 0.04	GH-C 1625
USA (MS), 1982 (Forrest)	EC+surfactant haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	94 84	soya beans	0.07 0.19 c 0.04	GH-C 1625
USA (MS), 1982 (Forrest)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	95 86	soya beans	0.11 0.37	GH-C 1625
USA (MS), 1982 (Forrest)	EC+surfactant haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	95 86	soya beans	0.11 0.43	GH-C 1625
USA (MS), 1982 (Forrest)	EC + oil haloxyfop-methyl	0.28	pre-bloom		1	109	soya beans	0.15 0.25	GH-C 1625

SOYA BEANS	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days		[b]	
USA (NC), 1982 (Forrest)	EC haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	122 89	soya beans	0.044 < 0.05 c 0.10	GH-C 1625
USA (NE), 1982 (Century)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	96 82	soya beans	0.22 1.2	GH-C 1625
USA (OH), 1982 (Williams 79)	EC + oil haloxyfop-methyl	0.28	pre-bloom in bloom		1 1	127 99	soya beans	0.05 0.34	GH-C 1625

[a] PE: post emergence. PS: post sowing ~ = appromimately

[b] GHB-P-024: replicated field samples

Soya beans, BBCH growth stages (Meier, 2001)

11 First pair of true leaves unfolded (unifoliate leaves on the first node)

12 Trifoliate leaf on the second node unfolded.

13 Trifoliate leaf on the third node unfolded.

14 Trifoliate leaf on the fourth node unfolded.

19 Trifoliate leaf on the ninth node unfolded. No side shoots visible.

23 Third side shoot of first order visible.

55 First flower buds enlarged.

65 Full flowering: about 50% of flowers open.

67 Flowering declining.

69 End of flowering: first pods visible.

71 About 10% of pods have reached final length.

Table 26 Haloxyfop residues in sugar beet resulting from supervised trials in Belgium, France, Germany, Italy and Spain

SUGAR BEET	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Belgium, 2000 (Semper)	EC haloxyfop-P-methyl	0.10	BBCH 39	310	1	116	beet roots	0.03	GHE-P-8867 CEMAS-1266C
France, 1988 (Apachee)	EC haloxyfop-ethoxyethyl	0.10	2 leaf		1	134	beet roots	< 0.02	GHE-P-1972 R88-36C
France, 1988 (Apachee)	EC haloxyfop-ethoxyethyl	0.21	2 leaf		1	134	beet roots	< 0.02	GHE-P-1972 R88-36C
France, 1988 (Apachee)	EC haloxyfop-P-methyl	0.052	2 leaf		1	134	beet roots	< 0.02	GHE-P-1972 R88-36C
France, 1988 (Apachee)	EC haloxyfop-P-methyl	0.10	2 leaf		1	134	beet roots	< 0.02	GHE-P-1972 R88-36C

SUGAR BEET	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
France, 1988 (Aramis)	EC haloxyfop-ethoxyethyl	0.10	2 leaf		1	165	beet roots	< 0.02	GHE-P-1972 R88-36B
France, 1988 (Aramis)	EC haloxyfop-ethoxyethyl	0.21	2 leaf		1	165	beet roots	< 0.02	GHE-P-1972 R88-36B
France, 1988 (Aramis)	EC haloxyfop-P-methyl	0.052	2 leaf		1	165	beet roots	< 0.02	GHE-P-1972 R88-36B
France, 1988 (Aramis)	EC haloxyfop-P-methyl	0.10	2 leaf		1	165	beet roots	< 0.02	GHE-P-1972 R88-36B
France, 1988 (Perfo)	EC haloxyfop-ethoxyethyl	0.10	2–3 leaf	250	1	131	beet roots	< 0.02	GHE-P-1972 R88-36A
France, 1988 (Perfo)	EC haloxyfop-ethoxyethyl	0.21	2–3 leaf	250	1	131	beet roots	< 0.02	GHE-P-1972 R88-36A
France, 1988 (Perfo)	EC haloxyfop-P-methyl	0.052	2–3 leaf	250	1	131	beet roots	< 0.02	GHE-P-1972 R88-36A
France, 1988 (Perfo)	EC haloxyfop-P-methyl	0.10	2–3 leaf	250	1	131	beet roots	< 0.02	GHE-P-1972 R88-36A
Germany, 1988 (Kaweduka)	EC haloxyfop-P-methyl	0.10	6 leaf (EC23)	400	1	15 98 125	beet roots beet roots beet roots	0.11 0.01 < 0.01	GHE-P-2036 RT 108/88
Germany, 1988 (Kaweduka)	EC haloxyfop-ethoxyethyl	0.21	6 leaf (EC23)	400	1	15 98 125	beet roots beet roots beet roots	0.25 0.01 < 0.01	GHE-P-2036 RT 108/88
Germany, 1988 (Kawemono)	EC haloxyfop-P-methyl	0.10	crop cover complete (EC45)	400	1	24 76 108 128	beet roots beet roots beet roots beet roots	0.22 [b] 0.03 c 0.02 0.04 0.03	GHE-P-2036 RT 85&86/88
Germany, 1988 (Kawemono)	EC haloxyfop-ethoxyethyl	0.21	crop cover complete (EC45)	400	1	24 76 108 128	beet roots beet roots beet roots beet roots	0.08 [b] 0.14 c 0.02 0.08 0.05	GHE-P-2036 RT 85&86/88
Germany, 1988 (Nowadima)	EC haloxyfop-P-methyl	0.10	6–8 leaf (EC 23-25)	400	1	13 76 104 118	beet roots beet roots beet roots beet roots	0.30 0.07 c 0.02 0.05 c 0.02 0.04	GHE-P-2036 RT 76/88
Germany, 1988 (Nowadima)	EC haloxyfop-ethoxyethyl	0.21	6–8 leaf (EC 23-25)	400	1	13 76 104 118	beet roots beet roots beet roots beet roots	0.38 0.16 c 0.02 0.06 c 0.02 0.06	GHE-P-2036 RT 76/88
Germany, 2000 (Helix)	EC haloxyfop-P-methyl	0.10	BBCH 39	220	1	92	beet root	0.09	GHE-P-8867 CEMAS-1266A
Germany, 2000 (Kontrast)	EC haloxyfop-P-methyl	0.10	BBCH 39	310	1	116	beet root	0.02	GHE-P-8867 CEMAS-1266B
Italy, 1992 (Break)	EC haloxyfop-P-methyl	0.10	8 leaf	400	1	65	beet roots	0.02	GHE-P-3078 R92-45B
Italy, 1992 (Mirto)	EC haloxyfop-P-methyl	0.10	8–9 leaf	400	1	67	beet roots	0.03	GHE-P-3078 R92-45A

SUGAR BEET	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Spain, 2000 (Lola)	EC haloxyfop-P-methyl	0.10	BBCH 39	370	1	99	beet roots	< 0.02	GHE-P-8868 CEMS-1267A

[a] GHE-P-1972 (R88-36A), GHE-P-2036, GHE-P-8867, GHE-P-8868: broadcast application. GHE-P-1972 (R88-36B and R88-36C): directed at ground.

[b] RT 85&86/88. The study author suggests these two root samples (0.22 and 0.08 mg/kg) have been incorrectly labelled.

Beet, BBCH growth stages (Meier, 2001)

39 Crop cover complete: leaves covering 90% of ground.

Table 27 Haloxyfop residues in rice resulting from supervised trials in the USA

RICE	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
USA (LA), 1987 (Mercury)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	94	1	99	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Newbonnet)	WG + surfactant haloxyfop-methyl	0.10	4 leaf	94	1	109	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Newbonnet)	WG + surfactant haloxyfop-methyl	0.20	4 leaf	94	1	109	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Newbonnet)	WG + surfactant haloxyfop-methyl	0.10	4 leaf	94	1 ✈	113	rice grain	< 0.01	GH-C 2175
USA (TX), 1987 (Skybonnet)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	94	1	93	rice grain	< 0.01	GH-C 2175
USA (TX), 1987 (Skybonnet)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	94	1 ✈	93	rice grain	< 0.01	GH-C 2175
USA (LA), 1987 (Lemont)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	94	1	93	rice grain	< 0.01	GH-C 2175
USA (LA), 1987 (Lemont)	WG + surfactant haloxyfop-methyl	0.20	4–5 leaf	94	1	93	rice grain	< 0.01	GH-C 2175
USA (LA), 1987 (Lemont)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	94	1 ✈	86	rice grain	< 0.01	GH-C 2175
USA (MS), 1987 (Lemont)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	94	1	105	rice grain	< 0.01	GH-C 2175

RICE	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
USA (MS), 1987 (Lemont)	WG + surfactant haloxyfop-methyl	0.20	4–5 leaf	94	1	105	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Tebonnet)	WG + surfactant haloxyfop-methyl	0.10	4 leaf	94	1	104	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Tebonnet)	WG + surfactant haloxyfop-methyl	0.20	4 leaf	94	1	104	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Tebonnet)	WG + surfactant haloxyfop-methyl	0.10	4 leaf	75	1 ✈	101	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Newbonnet)	WG + surfactant haloxyfop-methyl	0.10	4 leaf	94	1 ✈	107	rice grain	< 0.01	GH-C 2175
USA (AR), 1987 (Rexmont)	WG + surfactant haloxyfop-methyl	0.10	4 leaf	94	1 ✈	99	rice grain	< 0.01	GH-C 2175
USA (CA), 1988 (M201)	WG + surfactant haloxyfop-methyl	0.10	4–5 leaf	110	1	106	rice grain	< 0.01	GH-C 2175

Table 28 Haloxyfop residues in cotton seed resulting from supervised trials in Brazil, Greece, Spain and the USA

COTTON	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Brazil (Mato Grosso), 2000–2001 (CD-404)	EC haloxyfop-P-methyl	0.12	first bloom	200	1	98	cotton seed	0.47 0.52	GHB-P 733
Brazil (Mato Grosso), 2000–2001 (CD-404)	EC haloxyfop-P-methyl	0.060	first bloom	200	1	98	cotton seed	0.23 0.30	GHB-P 733
Brazil (MG), 2000–2001 (Delta Opal)	EC haloxyfop-P-methyl	0.060	Pre-bloom	200	1	107	cotton seed	< 0.01 0.01	GHB-P 730
Brazil (MG), 2000–2001 (Delta Opal)	EC haloxyfop-P-methyl	0.12	Pre-bloom	200	1	107	cotton seed	0.03 0.03	GHB-P 730
Brazil (Parana), 1996–1997 (IAC-22)	EC haloxyfop-P-methyl	0.060		200	1	122 133 142	cotton seed cotton seed cotton seed	< 0.01 (3) < 0.01 (3) < 0.01 (3)	GHB-P 372
Brazil (Parana), 1996–1997 (IAC-22)	EC haloxyfop-P-methyl	0.12		200	1	122 133 142	cotton seed cotton seed cotton seed	< 0.01 (3) < 0.01 (3) < 0.01 (3)	GHB-P 372
Brazil (PR), 2000–2001 (IAPAR-71)	EC haloxyfop-P-methyl	0.060	Pre-bloom	200	1	105 116 125 134 145	cotton seed	0.03 0.03 0.04 0.04 0.08 0.06 0.06 0.08 0.03 0.05	GHB-P 730

COTTON	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Brazil (PR), 2000–2001 (IAPAR-71)	EC haloxyfop-P-methyl	0.12	Pre-bloom	200	1	105 116 125 134 145	cotton seed	0.06 0.06 0.06 0.08 0.08 0.08 0.09 0.07 0.05 0.06	GHB-P 730
Brazil (SP), 1983–1984 (IAC-17)	EC + oil haloxyfop-methyl	0.12		360	1	111	cotton seed	< 0.1 (4)	GHB-P-034 Araras Note [a]
Brazil (SP), 1983–1984 (IAC-17)	EC + oil haloxyfop-methyl	0.24		360	1	111	cotton seed	< 0.1 (4)	GHB-P-034 Araras Note [a]
Brazil (SP), 1983–1984 (IAC-17)	EC + oil haloxyfop-methyl	0.36		360	1	111	cotton seed	< 0.1 (4)	GHB-P-034 Araras Note [a]
Brazil (SP), 1983–1984 (IAC-19)	EC + oil haloxyfop-methyl	0.24		370	1	112	cotton seed	< 0.1 (3)	GHB-P-034 Miguelopolis Note [a]
Brazil (SP), 1983–1984 (IAC-19)	EC + oil haloxyfop-methyl	0.36		370	1	112	cotton seed	< 0.1 (3)	GHB-P-034 Miguelopolis Note [a]
Brazil (SP), 1983–1984 (IAC-19)	EC + oil haloxyfop-methyl	0.12		370	1	112	cotton seed	< 0.1 (3)	GHB-P-034 Miguelopolis Note [a]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.12		300	1	93	cotton seed	0.10 0.12 0.15 < 0.10	GHB-P-034 Guira Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.24		300	1	93	cotton seed	0.14 0.19 0.19 0.20	GHB-P-034 Guira Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.36		300	1	93	cotton seed	0.24 0.23 0.26 0.20	GHB-P-034 Guira Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.36		300	1	93	cotton seed	0.29 0.21 0.27 0.20	GHB-P-034 Guira Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.12		300	1	101	cotton seed	< 0.1 (4)	GHB-P-034 Miguelopolis Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.24		300	1	101	cotton seed	0.13 0.12 0.12 < 0.1	GHB-P-034 Miguelopolis Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.36		300	1	101	cotton seed	0.15 0.11 0.10 < 0.1	GHB-P-034 Miguelopolis Note [b]
Brazil (SP), 1984–1985 (IAC-19)	EC + oil haloxyfop-methyl	0.48		300	1	101	cotton seed	< 0.1 (3) 0.11	GHB-P-034 Miguelopolis Note [b]
Brazil (SP), 1996–1997 (IAC-22)	EC haloxyfop-P-methyl	0.060		200	1	102 113 123	cotton seed cotton seed cotton seed	0.02 0.01 0.01 < 0.01 < 0.01 (4) < 0.01 (4)	GHB-P 372
Brazil (SP), 1996–1997 (IAC-22)	EC haloxyfop-P-methyl	0.12		200	1	102 113 123	cotton seed cotton seed cotton seed	0.02 0.01 < 0.01 0.01 < 0.01 (4) < 0.01 (4)	GHB-P 372
Greece, 1999 (McNair)	EC haloxyfop-P-methyl	0.16	BBCH 28	200	1	90	cotton seed	0.29	GHE-P-9337 R99-134D
Greece, 1999 (T-16)	EC haloxyfop-P-methyl	0.16	BBCH 28-29	200	1	91	cotton seed	0.22	GHE-P-9337 R99-134C

COTTON	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Greece, 2000 (T-16)	EC haloxyfop-P-methyl	0.16	BBCH 23	200	1	96	cotton seed	< 0.02	GHE-P-8854 CEMS-1251A
Greece, 2000 (T-16)	EC haloxyfop-P-methyl	0.16	BBCH 23	200	1	96	cotton seed	0.03	GHE-P-8854 CEMS-1251B
Spain, 1991 (Stoneville 506)	EC haloxyfop-P-methyl	0.16	8 leaves	300	1	123	cotton seed	< 0.02 (2)	GHE-P-2802
Spain, 1999 (Corona)	EC haloxyfop-P-methyl	0.16	BBCH 52	310	1	98	cotton seed	0.14	GHE-P-9337 R99-134B
Spain, 1999 (La Chata)	EC haloxyfop-P-methyl	0.16	BBCH 52	300	1	90	cotton seed	0.23	GHE-P-9337 R99-134A
Spain, 2000 (Lachata)	EC haloxyfop-P-methyl	0.16	BBCH 51	200	1	90	cotton seed	0.04	GHE-P-8854 CEMS-1251D
Spain, 2000 (Nata)	EC haloxyfop-P-methyl	0.16	BBCH 51	200	1	99	cotton seed	0.03	GHE-P-8854 CEMS-1251C
USA (AL), 1982 (Stoneville 825)	EC+oil haloxyfop-methyl	0.56	early bloom	190	1	99	cotton seed	0.19	GH-C 1623
USA (AL), 1982 (Stoneville 825)	EC haloxyfop-methyl	0.56	early bloom	190	1	99	cotton seed	0.14	GH-C 1623
USA (AR), 1982 (Delta Pine 41)	EC + oil haloxyfop-methyl	0.56	early bloom	190	1	72	cotton seed	0.84	GH-C 1623
USA (CA), 1982 (Acala SJ-2)	EC haloxyfop-methyl	0.56	early bloom		1	109	cotton seed	< 0.05	GH-C 1623
USA (CA), 1982 (Acala SJ-5)	EC + oil haloxyfop-methyl	0.56	3 weeks beyond first bloom	190	1	96	cotton seed	0.40	GH-C 1623
USA (LA), 1982 (Delta Pine 41)	EC haloxyfop-methyl	0.56	early bloom	90	1	89	cotton seed	0.18	GH-C 1623
USA (MS), 1982 (D+PL-55)	EC haloxyfop-methyl	0.56	early bloom	190	1	126	cotton seed	< 0.05	GH-C 1623
USA (MS), 1982 (D+PL-55)	EC [c] haloxyfop-methyl	0.56 0.56	early bloom	190 190	1 1	84 84	cotton seed cotton seed	0.82 0.93	GH-C 1623
USA (MS), 1982 (D+PL-55)	EC [c] haloxyfop-methyl	0.56 0.56	early bloom	190 190	1 1	77 77	cotton seed cotton seed	0.81 0.80	GH-C 1623
USA (MS), 1982 (Stoneville 825)	EC haloxyfop-methyl	0.56	early bloom	190	1	98	cotton seed	0.67	GH-C 1623
USA (NC), 1982 (McNair 235)	EC haloxyfop-methyl	0.56	early bloom	280	1	103	cotton seed	< 0.05	GH-C 1623
USA (TX), 1982 (GSA-71)	EC haloxyfop-methyl	0.56	early bloom	140	1	90	cotton seed	0.64	GH-C 1623

COTTON country, year (variety)	Application					PHI days	Commodity	haloxyfop, mg/kg	Ref
	Form	kg ai/ha	growth stage	water (L/ha)	no.				
USA (TX), 1982 (SP 21S)	EC haloxyfop-methyl	0.56	early bloom	220	1	96	cotton seed	< 0.05	GH-C 1623

[a] GHB-P-034: Interval of freezer storage before analysis: 29 months.

[b] GHB-P-034. Interval of freezer storage before analysis: 17 months.

[c] GH-C 1623. Comparison of 2 surfactants as adjuvants.

Cotton, BBCH growth stages (Meier, 2001)

23 Three vegetative side shoots (2nd order) visible.

28 Eight vegetative side shots visible.

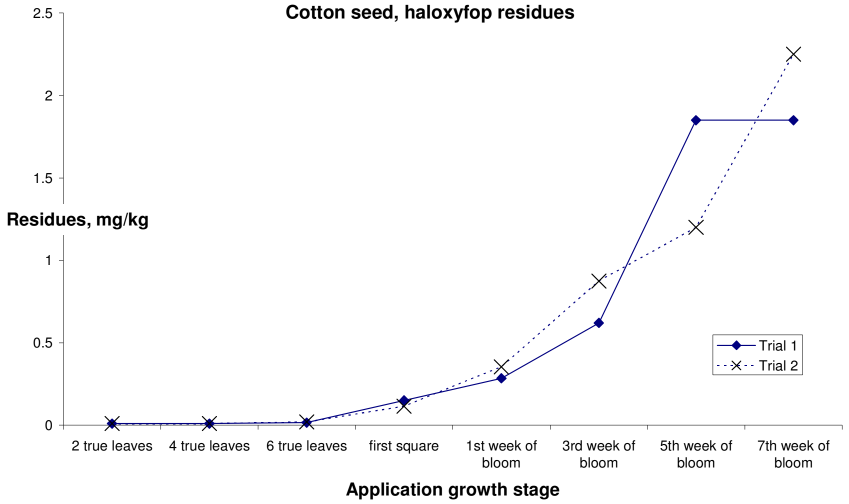
29 Nine or more vegetative side shoots visible.

51 First floral buds detectable ("pin-head square").

52 First floral buds visible("match-head square").

Gardner (1985, GH-C 1749) examined the effect of growth stage at time of application on the haloxyfop residue levels appearing in cotton seed at harvest. In two sets of trials haloxyfop-methyl was applied once to cotton crops at eight different growth stages from 'two true leaves' up to 'seventh week of bloom.' The residue data are summarised in Table 29.

When the residue levels are expressed as a function of growth stage and of interval between treatment and harvest (PHI) as in Figure 8, the growth stage appears to be a better predictor of likely residues.



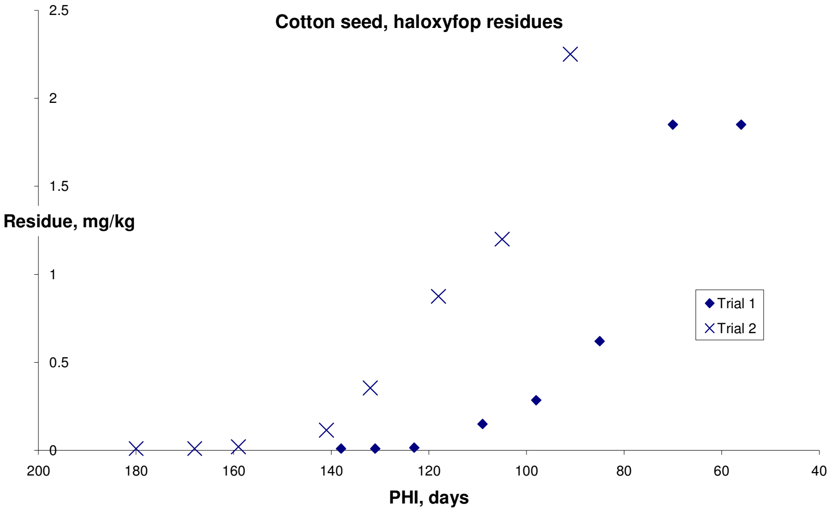


Figure 8 Effect of application growth stage and PHI on residue levels in cotton seed (Gardner, 1985, GH-C 1749)

Table 29 Investigation of the effect of growth stage at time of application on the magnitude of haloxyfop residues in cotton seed (Gardner, 1985, GH-C 1749)

COTTON country, year (variety)	Application				PHI days	Commodity	haloxyfop, mg/kg	Ref
	Form	kg ai/ha	growth stage	no.				
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	2 true leaves	1	138	cotton seed	< 0.01 (2)	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	4 true leaves	1	131	cotton seed	< 0.01 (2)	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	6 true leaves	1	123	cotton seed	0.01 0.02	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	first square	1	109	cotton seed	0.16 0.14	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	first week of bloom	1	98	cotton seed	0.30 0.27	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	third week of bloom	1	85	cotton seed	0.79 0.45	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	fifth week of bloom	1	70	cotton seed	1.7 2.0	GH-C 1749
USA (MS), 1983 (D + PL55)	EC + oil haloxyfop-methyl	0.56	seventh week of bloom	1	56	cotton seed	2.0 1.7	GH-C 1749
USA (MS), 1983 (D + PL55)	control plot	0		0		cotton seed	0.02 < 0.01	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	2 true leaves	1	180	cotton seed	< 0.01 (2)	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	4 true leaves	1	168	cotton seed	< 0.01 (2)	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	6 true leaves	1	159	cotton seed	0.02 0.02	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	first square	1	141	cotton seed	0.11 0.12	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	first week of bloom	1	132	cotton seed	0.35 0.36	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	third week of bloom	1	118	cotton seed	0.90 0.85	GH-C 1749

COTTON	Application				PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	no.	days			
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	fifth week of bloom	1	105	cotton seed	1.1 1.3	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	EC + oil haloxyfop-methyl	0.56	seventh week of bloom	1	91	cotton seed	2.3 2.2	GH-C 1749
USA (CA), 1983 (Acola SJ-2)	control plot	0		0		cotton seed	0.035 0.039	GH-C 1749

Table 30 Haloxyfop residues in oilseed rape resulting from supervised trials in Australia, France, Germany, Greece, Italy, Poland and Spain

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
Australia (NSW), 1997 (Canola, Rainbow)	EC + oil haloxyfop-P-methyl	0.052	1% flowering	100	1	96	canola grain	0.55	GHF-P 1700 97358.04
Australia (NSW), 1997 (Canola, Rainbow)	EC + oil haloxyfop-P-methyl	0.10	1% flowering	100	1	96	canola grain	0.96	GHF-P 1700 97358.04
Australia (NSW), 1997–1998 (Canola, Oscar)	EC + oil haloxyfop-P-methyl	0.052	early flower	100	1	64	canola grain	0.77	GHF-P 1700 97358.03
Australia (NSW), 1997–1998 (Canola, Oscar)	EC + oil haloxyfop-P-methyl	0.10	early flower	100	1	64	canola grain	1.2	GHF-P 1700 97358.03
Australia (SA), 1997–1998 (Canola, Siren)	EC + oil haloxyfop-P-methyl	0.052	budding, pre-flower	100	1	94	canola grain	0.22	GHF-P 1700 97358.02
Australia (SA), 1997–1998 (Canola, Siren)	EC + oil haloxyfop-P-methyl	0.10	budding, pre-flower	100	1	94	canola grain	0.49	GHF-P 1700 97358.02
Australia (WA), 1997–1998 (Canola, Karoo)	EC + oil haloxyfop-P-methyl	0.052	pre-flower, bud emergence	100	1	98	canola grain	0.86	GHF-P 1700 97358.01
Australia (WA), 1997–1998 (Canola, Karoo)	EC + oil haloxyfop-P-methyl	0.10	pre-flower, bud emergence	100	1	98	canola grain	1.7	GHF-P 1700 97358.01
France, 1988–1989 (Bienvenu)	EC haloxyfop-ethoxyethyl	0.10 directed	4–5 leaf		1	248	rape seed	< 0.05 (2)	GHE-P-1973 R89-5B
France, 1988–1989 (Bienvenu)	EC haloxyfop-ethoxyethyl	0.21 directed	4–5 leaf		1	248	rape seed	< 0.05 (2)	GHE-P-1973 R89-5B
France, 1988–1989 (Bienvenu)	EC haloxyfop-P-methyl	0.052 directed	4–5 leaf		1	248	rape seed	< 0.05 (2)	GHE-P-1973 R89-5B
France, 1988–1989 (Bienvenu)	EC haloxyfop-P-methyl	0.10 directed	4–5 leaf		1	248	rape seed	< 0.05 (2)	GHE-P-1973 R89-5B
France, 1988–1989 (Bienvenu)	EC haloxyfop-ethoxyethyl	0.10 directed	4–5 leaf		1	261	rape seed	< 0.05 (2)	GHE-P-1973 R89-5C
France, 1988–1989 (Bienvenu)	EC haloxyfop-ethoxyethyl	0.21 directed	4–5 leaf		1	261	rape seed	< 0.05 (2)	GHE-P-1973 R89-5C

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
France, 1988–1989 (Bienvenu)	EC haloxyfop-P-methyl	0.052 directed	4–5 leaf		1	261	rape seed	< 0.05 (2)	GHE-P-1973 R89-5C
France, 1988–1989 (Bienvenu)	EC haloxyfop-P-methyl	0.10 directed	4–5 leaf		1	261	rape seed	< 0.05 (2)	GHE-P-1973 R89-5C
France, 1988–1989 (Darmor)	EC haloxyfop-ethoxyethyl	0.10 directed	8 leaf	250	1	268	rape seed	< 0.05 (2)	GHE-P-1973 R89-5A
France, 1988–1989 (Darmor)	EC haloxyfop-ethoxyethyl	0.21 directed	8 leaf	250	1	268	rape seed	< 0.05 (2)	GHE-P-1973 R89-5A
France, 1988–1989 (Darmor)	EC haloxyfop-P-methyl	0.052 directed	8 leaf	250	1	268	rape seed	< 0.05 (2)	GHE-P-1973 R89-5A
France, 1988–1989 (Darmor)	EC haloxyfop-P-methyl	0.10 directed	8 leaf	250	1	268	rape seed	< 0.05 (2)	GHE-P-1973 R89-5A
France, 2000 (Capitol)	EC haloxyfop-P-methyl	0.055	BBCH 57	200	1	104	rape seed	0.35	GHE-P-8857 CEMS-1254D
France, 2000 (Carolus)	EC haloxyfop-P-methyl	0.055	BBCH 60	215	1	102	rape seed	0.51	GHE-P-8857 CEMS-1254C
France, 2000 (Pronto)	EC haloxyfop-P-methyl	0.052	BBCH 50	255	1	102	rape seed	0.33	GHE-P-8858 CEMS-1255
France, 2001–2002 (Synergy)	EC haloxyfop-P-methyl	0.097	BBCH 16	190	1	233	rape seed	< 0.01	GHE-P-10092 CEMS-1572A
France, 2001–2002 (Zenith)	EC haloxyfop-P-methyl	0.10	BBCH 16	200	1	204	rape seed	< 0.01	GHE-P-10092 CEMS-1572B
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.079	BBCH 32	300	1	63	rape seed	1.9	GHE-P-11656 F06W028R T2
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.083	BBCH 39	320	1	63	rape seed	1.5	GHE-P-11656 F06W028R T3
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.085	BBCH 50	330	1	61	rape seed	1.1	GHE-P-11656 F06W028R T4
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.056	BBCH 32	320	1	63	rape seed	1.2	GHE-P-11656 F06W028R T5
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.052	BBCH 39	300	1	63	rape seed	0.78	GHE-P-11656 F06W028R T6
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.052	BBCH 50	310	1	63	rape seed	0.75	GHE-P-11656 F06W028R T7
France, 2006 (Prima)	EC + adjuvant haloxyfop-P-methyl	0.053	BBCH 32	310	1	61	rape seed	1.3	GHE-P-11656 F06W028R T8
Germany, 1988–1989 (Arabella)	EC haloxyfop-P-methyl	0.10	6 leaf (EC 23)	400	1	259	rape seed	< 0.05 (2)	GHE-P-2144 R88-21B

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
Germany, 1988–1989 (Arabella)	EC haloxyfop-ethoxyethyl	0.21	6 leaf (EC 23)	400	1	259	rape seed	< 0.05 (2)	GHE-P-2144 R88-21B
Germany, 1988–1989 (Arabella)		0 control plot			0	259	rape seed	< 0.05 (2)	GHE-P-2144 R88-21B
Germany, 1988–1989 (Ceres)	EC haloxyfop-P-methyl	0.10	6 leaf (EC 23)	400	1	272	rape seed	0.07 0.07	GHE-P-2144 R88-21A
Germany, 1988–1989 (Ceres)	EC haloxyfop-ethoxyethyl	0.21	6 leaf (EC 23)	400	1	272	rape seed	0.13 0.13	GHE-P-2144 R88-21A
Germany, 1988–1989 (Ceres)		0 control plot			0	272	rape seed	< 0.05 (2)	GHE-P-2144 R88-21A
Germany, 2000 (Mohican)	EC haloxyfop-P-methyl	0.051	BBCH 50	255	1	113	rape seed	0.42	GHE-P-8857 CEMS-1254A
Germany, 2000 (Zenith)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	113	rape seed	0.35	GHE-P-8857 CEMS-1254B
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.081	BBCH 32	310	1	97	rape seed	0.11	GHE-P-11657 G06W018R T2
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.084	BBCH 39	320	1	92	rape seed	0.10	GHE-P-11657 G06W018R T3
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.075	BBCH 50	290	1	91	rape seed	0.37	GHE-P-11657 G06W018R T4
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.055	BBCH 32	320	1	97	rape seed	0.065	GHE-P-11657 G06W018R T5
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.054	BBCH 39	310	1	92	rape seed	0.055	GHE-P-11657 G06W018R T6
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	91	rape seed	0.17	GHE-P-11657 G06W018R T7
Germany, 2006 (Heros)	EC + adjuvant haloxyfop-P-methyl	0.055	BBCH 32	320	1	97	rape seed	0.065	GHE-P-11657 G06W018R T8
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.082	BBCH 32	310	1	88	rape seed	< 0.01	GHE-P-11657 G06W017R T2
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.078	BBCH 39	300	1	85	rape seed	0.57	GHE-P-11657 G06W017R T3
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.085	BBCH 50	330	1	82	rape seed	0.43	GHE-P-11657 G06W017R T4
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.053	BBCH 32	310	1	88	rape seed	< 0.01	GHE-P-11657 G06W017R T5
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.056	BBCH 39	320	1	85	rape seed	0.49	GHE-P-11657 G06W017R T6

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.055	BBCH 50	310	1	82	rape seed	0.52	GHE-P-11657 G06W017R T7
Germany, 2006 (Oase)	EC + adjuvant haloxyfop-P-methyl	0.054	BBCH 32	310	1	88	rape seed	0.43	GHE-P-11657 G06W017R T8
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.081	BBCH 32	210	1	80	rape seed	1.8	GHE-P-11656 GR06W013R T2
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.076	BBCH 39	290	1	69	rape seed	2.7	GHE-P-11656 GR06W013R T3
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.079	BBCH 50-63	300	1	65	rape seed	2.4	GHE-P-11656 GR06W013R T4
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.052	BBCH 32	200	1	80	rape seed	0.37	GHE-P-11656 GR06W013R T5
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.052	BBCH 39	300	1	69	rape seed	1.5	GHE-P-11656 GR06W013R T6
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.051	BBCH 50-63	290	1	65	rape seed	2.9	GHE-P-11656 GR06W013R T7
Greece, 2006 (Licosmos)	EC + adjuvant haloxyfop-P-methyl	0.054	BBCH 32	210	1	80	rape seed	1.4	GHE-P-11656 GR06W013R T8
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.082	BBCH 32	319	1	60	rape seed	0.68	GHE-P-11656 I06W023R T2
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.078	BBCH 39	300	1	57	rape seed	0.72	GHE-P-11656 I06W023R T3
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.078	BBCH 50	300	1	53	rape seed	1.4	GHE-P-11656 I06W023R T4
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.051	BBCH 32	290	1	60	rape seed	0.56	GHE-P-11656 I06W023R T5
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.052	BBCH 39	300	1	57	rape seed	0.46	GHE-P-11656 I06W023R T6
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	53	rape seed	0.79	GHE-P-11656 I06W023R T7
Italy, 2006 (Heros)	EC + adjuvant haloxyfop-P-methyl	0.054	BBCH 32	310	1	60	rape seed	0.70	GHE-P-11656 I06W023R T8
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.082	BBCH 32	320	1	92	rape seed	0.33	GHE-P-11657 PL06W009R T2
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.080	BBCH 39	310	1	89	rape seed	0.42	GHE-P-11657 PL06W009R T3
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.084	BBCH 50	320	1	87	rape seed	0.62	GHE-P-11657 PL06W009R T4

OILSEED RAPE country, year (variety)	Application [a]					PHI days	Commodity	haloxyfop, mg/kg	Ref
	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.				
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.051	BBCH 32	300	1	92	rape seed	0.21	GHE-P-11657 PL06W009R T5
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.055	BBCH 39	320	1	89	rape seed	0.27	GHE-P-11657 PL06W009R T6
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	87	rape seed	0.35	GHE-P-11657 PL06W009R T7
Poland, 2006 (Californium)	EC + adjuvant haloxyfop-P-methyl	0.052	BBCH 32	300	1	87	rape seed	0.24	GHE-P-11657 PL06W009R T8
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.079	BBCH 32	300	1	147	rape seed [b]	0.02	GHE-P-11656 S06W027R T2
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.077	BBCH 39	300	1	141	rape seed [b]	0.03	GHE-P-11656 S06W027R T3
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.077	BBCH 50	290	1	134	rape seed [b]	0.087	GHE-P-11656 S06W027R T4
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.056	BBCH 32	320	1	147	rape seed [b]	0.02	GHE-P-11656 S06W027R T5
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.053	BBCH 39	300	1	141	rape seed [b]	0.02	GHE-P-11656 S06W027R T6
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.053	BBCH 50	300	1	134	rape seed [b]	0.03	GHE-P-11656 S06W027R T7
Spain, 2006 (Tracia)	EC + adjuvant haloxyfop-P-methyl	0.052	BBCH 32	300	1	147	rape seed [b]	0.01	GHE-P-11656 S06W027R T8

[a] Directed: spray directed at ground.

[b] Plants were dried at 30 °C in a drying oven for 40 hours and separated afterwards by threshing.

[c] Adjuvant used in European trials: rapeseed oil.

Oilseed rape, BBCH growth stages (Meier, 2001)

16	Six leaves unfolded.
32	Two visibly extended internodes.
39	Nine or more visibly extended internodes.
50	Flower buds present, still enclosed by leaves.
57	Individual flower buds (secondary inflorescences) visible but still closed.
60	First flowers open.
63	30% of flowers on main raceme open.

Table 31 Haloxyfop residues in peanuts resulting from supervised trials in Argentina and Australia

PEANUTS	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 days after emergence	200	1	153	peanuts	< 0.02 (3)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	45 days after emergence	200	1	143	peanuts	< 0.02 (3)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	53 days after emergence	200	1	135	peanuts	< 0.02 (3)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	35 days after emergence	200	1	153	peanuts	< 0.02 (3)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	45 days after emergence	200	1	143	peanuts	< 0.02 (3)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	53 days after emergence	200	1	135	peanuts	0.02 < 0.02 (2)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 and 81 days after emergence	200	2	107	peanuts	0.02 < 0.02 (2)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	45 and 91 days after emergence	200	2	97	peanuts	< 0.02 (3)	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	53 and 103 days after emergence	200	2	85	peanuts	< 0.02 (2) 0.02	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 and 81 days after emergence	200	2	107	peanuts	0.03 0.03 0.03	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	45 and 91 days after emergence	200	2	97	peanuts	0.03 0.02 0.03	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	53 and 103 days after emergence	200	2	85	peanuts	0.02 0.02 0.03	GHB-P 293 Acequias Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 days after emergence	200	1	163	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	45 days after emergence	200	1	153	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	57 days after emergence	200	1	141	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	35 days after emergence	200	1	163	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	45 days after emergence	200	1	153	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]

PEANUTS	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	57 days after emergence	200	1	141	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 and 83 days after emergence	200	2	115	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	45 and 97 days after emergence	200	2	101	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	57 and 107 days after emergence	200	2	91	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	35 and 83 days after emergence	200	2	115	peanuts	0.03 0.03 0.02	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	45 and 97 days after emergence	200	2	101	peanuts	< 0.02 0.02 < 0.02	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	57 and 107 days after emergence	200	2	91	peanuts	< 0.02 (3)	GHB-P 293 Coronel Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	48 days after emergence	200	1	131	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	54 days after emergence	200	1	125	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	35 days after emergence	200	1	144	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	48 days after emergence	200	1	131	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	54 days after emergence	200	1	125	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 and 85 days after emergence	200	2	94	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–92 (Florunner)	EC haloxyfop-P-methyl	0.045	48 and 95 days after emergence	200	2	84	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	54 and 105 days after emergence	200	2	74	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	35 and 85 days after emergence	200	2	94	peanuts	< 0.02 (2) 0.03	GHB-P 293 Espinillo Note [a]

PEANUTS	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	48 and 95 days after emergence	200	2	84	peanuts	0.02 < 0.02 (2)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.090	54 and 105 days after emergence	200	2	74	peanuts	0.02 < 0.02 (2)	GHB-P 293 Espinillo Note [a]
Argentina (Cordoba), 1991–1992 (Florunner)	EC haloxyfop-P-methyl	0.045	35 days after emergence	200	1	144	peanuts	< 0.02 (3)	GHB-P 293 Espinillo Note [a]
Australia (Qld), 1997–1998 (A46)	EC + oil haloxyfop-P-methyl	0.078	4–6 leaf pegging	100	2	56	peanuts	0.02	GHF-P 1773 974040GW
Australia (Qld), 1997–1998 (A46)	EC + oil haloxyfop-P-methyl	0.16	4–6 leaf pegging	100	2	56	peanuts	0.03	GHF-P 1773 974040GW
Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.078	5–6 leaf pegging	100	2	56	peanuts	0.02	GHF-P 1773 974039GW
Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.16	5–6 leaf pegging	100	2	56	peanuts	0.05	GHF-P 1773 974039GW
Australia (Qld), 1997–98 (Streeton)	EC + oil haloxyfop-P-methyl	0.078	6–8 leaf late pegging	100	2	56	peanuts	< 0.02	GHF-P 1773 974042GW
Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.16	6–8 leaf late pegging	100	2	56	peanuts	0.03	GHF-P 1773 974042GW
Australia (Qld), 1997–1998 (White Spanish)	EC + oil haloxyfop-P-methyl	0.078	6–7 leaf late pegging	100	2	56	peanuts	< 0.02	GHF-P 1773 974043GW
Australia (Qld), 1997–1998 (White Spanish)	EC + oil haloxyfop-P-methyl	0.16	6–7 leaf late pegging	100	2	56	peanuts	< 0.02	GHF-P 1773 974043GW

[a] GHB-P 293. Interval of freezer storage before analysis: 42 months.

Table 32 Haloxyfop residues in sunflower seed resulting from supervised trials in Argentina, France, Germany, Greece and Spain

SUNFLOWER	Application [a b]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Argentina, 1991–1992 (Cargill 407)	EC haloxyfop-P-methyl	0.18	PS 26 days	200	1	100	sunflower seed	0.01	GHB-P 199 Note [c]
		0.18	PS 37 days	200	1	89	sunflower seed	0.01	
		0.18	PS 47 days	200	1	79	sunflower seed	0.14	
Argentina, 1991–1992 (Cargill 407)	EC haloxyfop-P-methyl	0.090	PS 26 days	200	1	100	sunflower seed	0.01	GHB-P 199 Note [c]
		0.090	PS 37 days	200	1	89	sunflower seed	0.01	
		0.090	PS 47 days	200	1	79	sunflower seed	0.05	
France, 1988 (Cargisol)	EC haloxyfop-P-methyl	0.050	3 pairs of leaves	330	1	118	sunflower seed	< 0.05	GHE-P-2059 R88-37
France, 1988 (Cargisol)	EC haloxyfop-P-methyl	0.10	3 pairs of leaves	330	1	118	sunflower seed	< 0.05	GHE-P-2059 R88-37

SUNFLOWER	Application [a][b]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
France, 1988 (Cargisol)	EC haloxyfop-ethoxyethyl	0.10	3 pairs of leaves	330	1	118	sunflower seed	< 0.05	GHE-P-2059 R88-37
France, 1988 (Cargisol)	EC haloxyfop-ethoxyethyl	0.20	3 pairs of leaves	330	1	118	sunflower seed	0.09	GHE-P-2059 R88-37
France, 1989 (Agrisol)	EC haloxyfop-P-methyl	0.050	6 pairs of leaves	330	1	105	sunflower seed	< 0.05	GHE-P-2059 R89-8A
France, 1989 (Agrisol)	EC haloxyfop-P-methyl	0.10	6 pairs of leaves	330	1	105	sunflower seed	< 0.05	GHE-P-2059 R89-8A
France, 1989 (Agrisol)	EC haloxyfop-ethoxyethyl	0.10	6 pairs of leaves	330	1	105	sunflower seed	< 0.05	GHE-P-2059 R89-8A
France, 1989 (Agrisol)	EC haloxyfop-ethoxyethyl	0.20	6 pairs of leaves	330	1	105	sunflower seed	0.07	GHE-P-2059 R89-8A
France, 1989 (Albena)	EC haloxyfop-P-methyl	0.050	8 pairs of leaves	330	1	89	sunflower seed	< 0.05	GHE-P-2059 R89-8B
France, 1989 (Albena)	EC haloxyfop-P-methyl	0.10	8 pairs of leaves	330	1	89	sunflower seed	0.07	GHE-P-2059 R89-8B
France, 1989 (Albena)	EC haloxyfop-ethoxyethyl	0.10	8 pairs of leaves	330	1	89	sunflower seed	0.05	GHE-P-2059 R89-8B
France, 1989 (Albena)	EC haloxyfop-ethoxyethyl	0.20	8 pairs of leaves	330	1	89	sunflower seed	0.16	GHE-P-2059 R89-8B
France, 1999 (Albena)	EC haloxyfop-P-methyl	0.10	BBCH 32	300	1	98	sunflower seed	0.10	GHE-P 8999 R99-136B
France, 1999 (Sanluca)	EC haloxyfop-P-methyl	0.10	BBCH 31	300	1	114	sunflower seed	0.06 Note	GHE-P 8999 R99-136A
France, 2000 (Allstar RM)	EC haloxyfop-P-methyl	0.15	BBCH 20	290	1	81	sunflower seed	0.14	GHE-P 8861 CEMS-1259A
France, 2000 (Melody)	EC haloxyfop-P-methyl	0.15	BBCH 20	290	1	89	sunflower seed	< 0.05	GHE-P 8861 CEMS-1259B
France, 2001 (Albena)	EC haloxyfop-P-methyl	0.15	BBCH 19-20	290	1	75	sunflower seed	0.05	GHE-P-9784 CEMS-1571A
Germany, 2000 (Alnika)	EC haloxyfop-P-methyl	0.10	BBCH 30	290	1	123	sunflower plant	< 0.05	GHE-P 8860 CEMS-1258B
Germany, 2000 (Alnika)	EC haloxyfop-P-methyl	0.10	BBCH 30	290	1	123	sunflower seed	< 0.05	GHE-P 8860 CEMS-1258B
Germany, 2000 (Coril)	EC haloxyfop-P-methyl	0.10	BBCH 33	370	1	84	sunflower seed	0.17	GHE-P 8860 CEMS-1258A
Germany, 2000 (Coril)	EC haloxyfop-P-methyl	0.10	BBCH 33	370	1	84	sunflower plant	0.17	GHE-P 8860 CEMS-1258A
Greece, 2001 (Sena)	EC haloxyfop-P-methyl	0.15	BBCH 20	300	1	107	sunflower seed	< 0.05	GHE-P-9784 CEMS-1571C

SUNFLOWER	Application [a b]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Greece, 2001 (Sena)	EC haloxyfop-P-methyl	0.15	BBCH 20	290	1	76	sunflower seed	< 0.05	GHE-P-9784 CEMS-1571C
Spain, 2000 (Florasol)	EC haloxyfop-P-methyl	0.16	BBCH 20	370	1	79	sunflower seed	< 0.05	GHE-P 8861 CEMS-1259D
Spain, 2000 (Sambro)	EC haloxyfop-P-methyl	0.16	BBCH 20	390	1	82	sunflower seed	< 0.05	GHE-P 8861 CEMS-1259C
Spain, 2001 (Almazon)	EC haloxyfop-P-methyl	0.16	BBCH 20	370	1	76	sunflower seed	0.17	GHE-P-9784 CEMS-1571B

[a] GHE-P-2059: spray directed at the ground.
GHB-P 199: ground broadcast
GHE-P-8999, GHE-P 8861, GHB-P 8860, GHE-P-9784: foliar application—broadcast

[b] PS: post-sowing.

[c] GHB-P 199. Interval of freezer storage before analysis: 20 months.

Sunflowers, BBCH growth stages (Meier, 2001)

19 Nine or more leaves unfolded.

30 Beginning of stem elongation.

31 One visibly extended internode.

32 Two visibly extended internodes.

33 Three visibly extended internodes.

Table 33 Haloxyfop residues in coffee resulting from supervised trials in Brazil and Colombia

COFFEE	Application				PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days	[a]	mg/kg	
Brazil (SP), 1983 (Mundo Novo)	EC haloxyfop-methyl	0.12 directed [b]		1	110	coffee beans	< 0.02 (4)	GHB-P 019
Brazil (SP), 1983 (Mundo Novo)	EC haloxyfop-methyl	0.24 directed [b]		1	110	coffee beans	< 0.02 (4)	GHB-P 019
Brazil (SP), 1983 (Mundo Novo)	EC haloxyfop-methyl	0.48 directed [b]		1	110	coffee beans	< 0.02 (4)	GHB-P 019
Brazil (SP), 1983 (Mundo Novo)	EC haloxyfop-methyl	0.96 directed [b]		1	110	coffee beans	< 0.02 (4)	GHB-P 019
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.24 directed [b]		1	74 119	coffee beans	< 0.02 (4) < 0.02 (4)	GHB-P 023 Cravinhos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.48 directed [b]		1	74 119	coffee beans	< 0.02 (4) < 0.02 (4)	GHB-P 023 Cravinhos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.96 directed [b]		1	74 119	coffee beans	< 0.02 (4) < 0.02 (4)	GHB-P 023 Cravinhos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.24 directed [b]		1	103 165 201	coffee beans	< 0.02 (3) < 0.02 (3) < 0.02 (3)	GHB-P 023 São Carlos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.48 directed [b]		1	103 165 201	coffee beans	< 0.02 (3) < 0.02 (3) < 0.02 (3)	GHB-P 023 São Carlos

COFFEE	Application				PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days	[a]	mg/kg	
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.96 directed [b]		1	103 165 201	coffee beans	< 0.02 (3) < 0.02 (3) < 0.02 (3)	GHB-P 023 São Carlos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.24 directed [b]		2	137	coffee beans	< 0.02 (3)	GHB-P 023 São Carlos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.48 directed [b]		2	137	coffee beans	< 0.02 (3)	GHB-P 023 São Carlos
Brazil (SP), 1984 (Mundo Novo)	EC + oil haloxyfop-methyl	0.96 directed [b]		2	137	coffee beans	< 0.02 (3)	GHB-P 023 São Carlos
Colombia, 1983 (Caturra)	EC haloxyfop-methyl	0.18 directed [b c]		1	43 75 105	coffee beans	< 0.02 (3) < 0.02 (3) < 0.02 (3)	GHB-P 020
Colombia, 1983 (Caturra)	EC haloxyfop-methyl	0.36 directed [b c]		1	43 75 105	coffee beans	< 0.02 (3) < 0.02 (3) < 0.02 (3)	GHB-P 020

[a] GHB-P 023 GHB-P 019: Coffee berries were sun dried and depulped following normal commercial procedures to yield coffee beans.
GHB-P 020: Harvested coffee berries were air dried under shade for 2 days, followed by a 3 hours drying at 30 °C.

[b] Directed application for grass control.

[c] The product sometimes contacted the lower leaves, but phytotoxicity symptoms were not observed.

Table 34 Haloxyfop residues in alfalfa resulting from supervised trials in Australia, France, Germany and Poland

ALFALFA	Application				PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days		mg/kg	
Australia (NSW), 1988 (Hunter River)	EC haloxyfop-ethoxyethyl	0.10	100	1	4 7 14 21	alfalfa foliage (fresh weight)	4.6 4.7 2.5 2.1	GHF-P 857
Australia (NSW), 1988 (Hunter River)	EC haloxyfop-ethoxyethyl	0.21	100	1	4 7 14 21	alfalfa foliage (fresh weight)	8.4 8.2 3.1 3.1	GHF-P 857
Australia (NSW), 1988 (Hunter River)	EC haloxyfop-ethoxyethyl	0.31	100	1	4 7 14 21	alfalfa foliage (fresh weight)	11.8 10.1 5.9 2.9	GHF-P 857
Australia (NSW), 1989 (CUF 101)	EC haloxyfop-P-methyl	0.052	99	1	8 14 22 32	alfalfa foliage (fresh weight)	1.3 1.3 0.76 0.65	GHF-P 1355 GHF-P 997
Australia (NSW), 1989 (CUF 101)	EC + oil haloxyfop-P-methyl	0.052	99	1	8 14 22 32	alfalfa foliage (fresh weight)	1.6 1.9 1.6 1.0	GHF-P 1355
Australia (NSW), 1989 (CUF 101)	EC haloxyfop-P-methyl	0.10	99	1	8 14 22 32	alfalfa foliage (fresh weight)	3.2 Note [d] 2.4 1.8 1.9	GHF-P 1355 GHF-P 997
Australia (NSW), 1989 (CUF 101)	EC + oil haloxyfop-P-methyl	0.10	99	1	8 14 22 32	alfalfa foliage (fresh weight)	3.5 3.4 2.2 1.9	GHF-P 1355

ALFALFA	Application				PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days		mg/kg	
Australia (NSW), 1989 (CUF 101)	EC haloxyfop-ethoxyethyl	0.10	99	1	8 14 22 32	alfalfa foliage (fresh weight)	3.1 2.8 1.5 1.1	GHF-P 1355 GHF-P 925 GHF-P 997
Australia (NSW), 1989 (CUF 101)	EC + oil haloxyfop-ethoxyethyl	0.10	99	1	8 14 22 32	alfalfa foliage (fresh weight)	3.6 4.4 3.4 1.9	GHF-P 1355
Australia (NSW), 1989 (CUF 101)	EC haloxyfop-ethoxyethyl	0.21	99	1	8 14 22 32	alfalfa foliage (fresh weight)	8.4 6.5 2.5 1.3	GHF-P 1355 GHF-P 925 GHF-P 997
Australia (NSW), 1998 (Auroa)	EC + oil haloxyfop-P-methyl	0.052	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	4.0 3.3 3.3 2.4 1.4 1.0 0.57	GHF-P 1943 98453-01 Note [b]
Australia (NSW), 1998 (Auroa)	EC + oil haloxyfop-P-methyl	0.10	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	7.3 6.1 6.5 5.1 3.1 2.0 1.0	GHF-P 1943 98453-01 Note [b]
Australia (NSW), 1998 (Pioneer brand, unknown variety)	EC + oil haloxyfop-P-methyl	0.052	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	4.3 3.1 3.5 2.5 2.0 1.7 0.74	GHF-P 1943 98453-02 Note [b]
Australia (NSW), 1998 (Pioneer brand, unknown variety)	EC + oil haloxyfop-P-methyl	0.10	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	8.1 6.9 6.5 4.7 3.5 2.9 0.76	GHF-P 1943 98453-02 Note [b]
Australia (NSW), 1998–1999 (not recorded)	EC + oil haloxyfop-P-methyl	0.078	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	7.6 6.4 6.2 5.7 4.3 2.2 3.1	GHF-P 1943 98453-04 Note [b]
Australia (NSW), 1998–1999 (not recorded)	EC + oil haloxyfop-P-methyl	0.16	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	18.5 13 11 13 7.5 4.8 5.5	GHF-P 1943 98453-04 Note [b]
Australia (Qld), 1998 (Scepture)	EC + oil haloxyfop-P-methyl	0.078	100	1	0 1 3 7 14 21 28	alfalfa foliage (fresh weight)	6.5 3.9 3.3 2.2 0.90 0.34 0.10	GHF-P 1943 98453-03 Note [b]

ALFALFA	Application				PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days		mg/kg	
Australia (Qld), 1998 (Scepture)	EC + oil haloxyfop-P-methyl	0.16	100	1	0	alfalfa foliage (fresh weight)	8.9	GHF-P 1943 98453-03 Note [b]
					1		7.8	
					3		6.1	
					7		4.2	
					14		0.93	
					21		1.3	
					28		0.30	
France, 2004 (Bellefeuilles)	EC haloxyfop-P-methyl	0.10	280	1	0	alfalfa foliage	8.1	GHE-P-11071 CEMS-2337B Note [c]
					4	alfalfa foliage	4.3	
					10	alfalfa foliage	4.5 [e] 0.01	
					15	alfalfa foliage	3.5	
					28	alfalfa foliage	3.2	
					28	32 alfalfa hay [a]	8.1	
France, 2004 (Diane)	EC haloxyfop-P-methyl	0.10	290	1	0	alfalfa foliage	10.7	GHE-P-11071 CEMS-2337A Note [c]
					4	alfalfa foliage	7.0	
					10	alfalfa foliage	3.6	
					15	alfalfa foliage	3.1 [e] 0.01	
					28	alfalfa foliage	2.5	
					28	32 alfalfa hay [a]	5.6	
Germany, 2004 (Mercedes)	EC haloxyfop-P-methyl	0.10	300	1	0	alfalfa foliage	13	GHE-P-11071 CEMS-2337C Note [c]
					5	alfalfa foliage	6.1	
					10	alfalfa foliage	3.0	
					15	alfalfa foliage	2.6	
					28	alfalfa foliage	0.46	
					28	35 alfalfa hay [a]	1.2	
Poland, 2004 (variety not recorded)	EC haloxyfop-P-methyl	0.10	310	1	0	alfalfa foliage	10.5	GHE-P-11071 CEMS-2337D Note [c]
					4	alfalfa foliage	3.8 [e] 0.12	
					11	alfalfa foliage	2.4 [e] 0.04	
					14	alfalfa foliage	1.4	
					28	alfalfa foliage	0.64	
					28	35 alfalfa hay [a]	2.0	

[a] Cut on day 28, left to dry in field and sampled on indicated day.

[b] GHF-P 1943. Interval of freezer storage before analysis: 19-23 months.

[c] GHE-P-11071. Interval of freezer storage before analysis: 2 years.

[d] GHF-P 977 reports 3.20 mg/kg for this sample while GHF-P 1355 reports 3.36 mg/kg.

[e]: sample from control plot.

Table 35 Haloxyfop residues in legume animal feeds resulting from supervised trials in Australia, France, Germany, Hungary, Italy and Spain. Crops: chickpeas, pea nuts, peas and soya beans

LEGUME CROP	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	[a]	mg/kg	
CHICKPEAS Australia (SA), 1997 (Desavic)	EC + oil haloxyfop-P-methyl	0.052	1–3 branch	100	1	0	whole plant dw	33	GHF-P 1718 97355.02
						7	whole plant dw	19	
						14	whole plant dw	9.4	
						28	whole plant dw	2.9	
						42	whole plant dw	0.95	
			some bloom		2	109	straw dw	0.13	
CHICKPEAS Australia (SA), 1997 (Desavic)	EC + oil haloxyfop-P-methyl	0.10	1–3 branch	100	1	0	whole plant dw	65	GHF-P 1718 97355.02
						7	whole plant dw	31	
						14	whole plant dw	18	
						28	whole plant dw	6.7	
						42	whole plant dw	0.93	
			some bloom		2	109	straw dw	0.28	

LEGUME CROP	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	[a]	mg/kg	
CHICKPEAS Australia (WA), 1997 (Koniva)	EC + oil haloxyfop-P-methyl	0.052	8–9 leaf	100	1	0	whole plant dw	46	GHF-P 1718 97355.01
						7	whole plant dw	23	
						14	whole plant dw	11.4	
						28	whole plant dw	4.3	
						42	whole plant dw	1.9	
			3–7 branch		2	118	straw	< 0.05	
CHICKPEAS Australia (WA), 1997 (Koniva)	EC + oil haloxyfop-P-methyl	0.10	8–9 leaf	100	1	0	whole plant dw	87	GHF-P 1718 97355.01
						7	whole plant dw	52	
						14	whole plant dw	29	
						28	whole plant dw	10.2	
						42	whole plant dw	4.6	
			3–7 branch		2	118	straw	< 0.05	
PEANUTS Australia (Qld), 1997–1998 (A46)	EC + oil haloxyfop-P-methyl	0.078	4–6 leaf	100	1	0	forage dw	29	GHF-P 1773 974040GW
						7	forage dw	7.7	
						14	forage dw	2.6	
						28	forage dw	0.28	
						42	forage	< 0.02	
			pegging	100	2	56	straw dw	0.42	
						56	shell	0.02	
PEANUTS Australia (Qld), 1997–1998 (A46)	EC + oil haloxyfop-P-methyl	0.16	4–6 leaf	100	1	0	forage dw	60	GHF-P 1773 974040GW
						7	forage dw	17	
						14	forage	< 0.02	
						28	forage dw	1.5	
						42	forage	< 0.02	
			pegging	100	2	56	straw dw	1.1	
						56	shell	0.03	
PEANUTS Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.078	5–6 leaf	100	1	0	forage dw	33	GHF-P 1773 974039GW
						7	forage dw	9.8	
						14	forage dw	4.4	
						28	forage dw	1.1	
						42	forage dw	0.25	
			pegging	100	2	56	straw dw	1.2	
						56	shell	0.03	
PEANUTS Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.16	5–6 leaf	100	1	0	forage dw	65	GHF-P 1773 974039GW
						7	forage dw	18	
						14	forage dw	7.8	
						28	forage dw	2.3	
						42	forage dw	0.25	
			pegging	100	2	56	straw dw	1.9	
						56	shell	0.10	
PEANUTS Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.078	6–8 leaf	100	1	0	forage dw	35	GHF-P 1773 974042GW
						7	forage dw	11	
						15	forage dw	2.2	
						28	forage	< 0.02	
						42	forage	< 0.02	
			late pegging	100	2	56	straw dw	3.0	
						56	shell	0.04	
PEANUTS Australia (Qld), 1997–1998 (Streeton)	EC + oil haloxyfop-P-methyl	0.16	6–8 leaf	100	1	0	forage dw	84	GHF-P 1773 974042GW
						7	forage dw	26	
						15	forage dw	4.7	
						28	forage dw	0.14	
						42	forage dw	0.08	
			late pegging	100	2	56	straw dw	5.4	
						56	shell	0.09	
PEANUTS Australia (Qld), 1997–1998 (White Spanish)	EC + oil haloxyfop-P-methyl	0.078	6–7 leaf	100	1	0	forage dw	45	GHF-P 1773 974043GW
						7	forage dw	15	
						15	forage dw	2.7	
						28	forage dw	0.13	
						42	forage	< 0.02	
			late pegging	100	2	56	straw dw	2.9	
						56	shell	0.03	

LEGUME CROP	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	[a]	mg/kg	
PEANUTS Australia (Qld), 1997–1998 (White Spanish)	EC + oil haloxyfop-P-methyl	0.16	6–7 leaf	100	1	0	forage dw	106	GHF-P 1773 974043GW
						7	forage dw	29	
						15	forage dw	4.0	
						28	forage dw	0.26	
						42	forage dw	0.06	
			late pegging	100	2	56	straw dw	3.8	
						56	shell	0.05	
PEAS France, 2000 (Vegetable peas, variety not recorded)	EC haloxyfop-P-methyl	0.10	BBCH 51-60	410	1	29	plant	0.08	GHE-P-8852 CEMS-1249A
PEAS Italy, 2000 (Vegetable peas, Lambado)	EC haloxyfop-P-methyl	0.10	BBCH 50	420	1	28	plant	< 0.05	GHE-P-8852 CEMS-1249B
SOYA BEANS France, 2000 (Queen)	EC haloxyfop-P-methyl	0.10	BBCH 12-13	290	1	90	plant	< 0.05	GHE-P-8859 CEMS-1257B
SOYA BEANS France, 2000 (Queen)	EC haloxyfop-P-methyl	0.10	BBCH 67-69	300	1	60	plant	0.12	GHE-P-8859 CEMS-1257B
SOYA BEANS France, 2000 (Sapporo)	EC haloxyfop-P-methyl	0.10	BBCH 13-14	300	1	90	plant	< 0.05	GHE-P-8859 CEMS-1257C
SOYA BEANS France, 2000 (Sapporo)	EC haloxyfop-P-methyl	0.10	BBCH 65-71	320	1	57	plant	0.13	GHE-P-8859 CEMS-1257C
SOYA BEANS Germany, 2001 (Dorena)	EC haloxyfop-P-methyl	0.11	BBCH 12	310	1	94	plant	< 0.05	GHE-P-9782 CEMS-1569B
SOYA BEANS Germany, 2001 (Dorena)	EC haloxyfop-P-methyl	0.11	BBCH 23	310	1	61	plant	0.10	GHE-P-9782 CEMS-1569B
SOYA BEANS Hungary, 2001 (Borostyan)	EC haloxyfop-P-methyl	0.10	BBCH 12	250	1	91	plant	< 0.05	GHE-P-9782 CEMS-1569A
SOYA BEANS Hungary, 2001 (Borostyan)	EC haloxyfop-P-methyl	0.10	BBCH 33	250	1	60	plant	0.18	GHE-P-9782 CEMS-1569A
SOYA BEANS Italy, 2000 (Silia)	EC haloxyfop-P-methyl	0.10	BBCH 11-19	300	1	86	plant	< 0.05	GHE-P-8859 CEMS-1257D
SOYA BEANS Italy, 2000 (Silia)	EC haloxyfop-P-methyl	0.10	BBCH 67-71	290	1	56	plant	0.08	GHE-P-8859 CEMS-1257D
SOYA BEANS Spain, 2000 (Osumi)	EC haloxyfop-P-methyl	0.10	BBCH 19	300	1	90	plant	0.07	GHE-P-8859 CEMS-1257A
SOYA BEANS Spain, 2000 (Osumi)	EC haloxyfop-P-methyl	0.10	BBCH 55	300	1	60	plant	0.06	GHE-P-8859 CEMS-1257A

[a] dw: dry weight

Peas, BBCH growth stages (Meier, 2001)

51 First flower buds visible outside leaves.

60 First flowers open (sporadically within the population)

Soya beans, BBCH growth stages (Meier, 2001)

11 First pair of true leaves unfolded.

12 Trifoliolate leaf on the 2nd node unfolded.

13 Trifoliolate leaf on the 3rd node unfolded.

19 Trifoliolate leaf on the 9th node unfolded. No side shoots visible.

23 3rd side shoot of first order visible.

55 First flower buds enlarged.

65 Full flowering: about 50% of flowers open.

67 Flowering declining.

69 End of flowering: first pods visible.

71 About 10% of pods have reached final length

Table 36 Haloxyfop residues in sugar beet plants and tops resulting from supervised trials in Belgium, Germany, Italy and Spain

SUGAR BEET	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Belgium, 2000 (Semper)	EC haloxyfop-P-methyl	0.10	BBCH 39	310	1	116	beet tops	0.07	GHE-P-8867 CEMAS-1266C
Germany, 1988 (Kaweduka)	EC haloxyfop-P-methyl	0.10	6 leaf (EC23)	400	1	1 15 98 125	whole plant leaves leaves leaves	2.2 c 0.10 0.17 < 0.02 < 0.02	GHE-P-2036 RT 108/88
Germany, 1988 (Kaweduka)	EC haloxyfop-ethoxyethyl	0.21	6 leaf (EC23)	400	1	1 15 98 125	whole plant leaves leaves leaves	3.9 c 0.10 0.21 < 0.02 < 0.02	GHE-P-2036 RT 108/88
Germany, 1988 (Kawemono)	EC haloxyfop-P-methyl	0.10	crop cover complete (EC45)	400	1	0 24 76 108 128	whole plant leaves leaves leaves leaves	2.0 0.42 Note [b] 0.10 c0.02 0.09 0.08	GHE-P-2036 RT 85&86/88
Germany, 1988 (Kawemono)	EC haloxyfop-ethoxyethyl	0.21	crop cover complete (EC45)	400	1	0 24 76 108 128	whole plant leaves leaves leaves leaves	4.4 0.18 Note [b] 0.28 c 0.02 0.18 0.16	GHE-P-2036 RT 85&86/88
Germany, 1988 (Nowadima)	EC haloxyfop-P-methyl	0.10	6-8 leaf (EC 23-25)	400	1	1 13 76 104 118	whole plant leaves leaves leaves leaves	2.5 0.38 0.14 c 0.02 0.10 0.09	GHE-P-2036 RT 76/88
Germany, 1988 (Nowadima)	EC haloxyfop-ethoxyethyl	0.21	6-8 leaf (EC 23-25)	400	1	1 13 76 104 118	whole plant leaves leaves leaves leaves	4.5 0.76 0.30 c 0.02 0.20 0.16	GHE-P-2036 RT 76/88
Germany, 2000 (Helix)	EC haloxyfop-P-methyl	0.10	BBCH 39	220	1	92	beet tops	0.12	GHE-P-8867 CEMAS-1266A
Germany, 2000 (Kontrast)	EC haloxyfop-P-methyl	0.10	BBCH 39	310	1	116	beet tops	0.08	GHE-P-8867 CEMAS-1266B

SUGAR BEET	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
Italy, 1992 (Break)	EC haloxyfop-P-methyl	0.10	8 leaf	400	1	65	beet tops	0.09	GHE-P-3078 R92-45B
Italy, 1992 (Mirto)	EC haloxyfop-P-methyl	0.10	8–9 leaf	400	1	67	beet tops	0.09	GHE-P-3078 R92-45A
Spain, 2000 (Lola)	EC haloxyfop-P-methyl	0.10	BBCH 39	370	1	99	beet tops	0.07	GHE-P-8868 CEMS-1267A

[a] GHE-P-2036, GHE-P-8867, GHE-P-8868: broadcast application.

[b] RT 85&86/88. The study author suggests these two leaf samples (0.42 and 0.18 mg/kg) have been incorrectly labelled.

Beet, BBCH growth stages (Meier, 2001)

39 Crop cover complete: leaves cover 90% of ground.

Table 37 Haloxyfop residues in oilseed rape plant resulting from supervised trials in Australia, France, Germany, Greece, Italy, Poland and Spain

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
Australia (NSW), 1997 (Canola, Rainbow)	EC + oil haloxyfop-P-methyl	0.052	cotyledon to 8 leaf	100	1	0 14 28 42	forage dw forage dw forage dw forage	30 1.0 0.32 < 0.02	GHF-P 1700 97358.04
Australia (NSW), 1997 (Canola, Rainbow)	EC + oil haloxyfop-P-methyl	0.052	1% flowering	100	1	96	fodder dw	0.13	GHF-P 1700 97358.04
Australia (NSW), 1997 (Canola, Rainbow)	EC + oil haloxyfop-P-methyl	0.10	1% flowering	100	1	96	fodder dw	0.25	GHF-P 1700 97358.04
Australia (NSW), 1997 (Canola, Rainbow)	EC + oil haloxyfop-P-methyl	0.10	cotyledon to 8 leaf	100	1	0 14 28 42	forage dw forage dw forage dw forage	65 2.2 0.81 0.14	GHF-P 1700 97358.04
Australia (NSW), 1997–1998 (Canola, Oscar)	EC + oil haloxyfop-P-methyl	0.052	6 leaf	100	1	0 7 42	forage dw forage dw forage dw	43 7.2 0.38	GHF-P 1700 97358.03
Australia (NSW), 1997–1998 (Canola, Oscar)	EC + oil haloxyfop-P-methyl	0.052	early flower	100	1	64	fodder dw	0.29	GHF-P 1700 97358.03
Australia (NSW), 1997–1998 (Canola, Oscar)	EC + oil haloxyfop-P-methyl	0.10	early flower	100	1	64	fodder dw	0.41	GHF-P 1700 97358.03
Australia (NSW), 1997–1998 (Canola, Oscar)	EC + oil haloxyfop-P-methyl	0.10	6 leaf	100	1	0 7 14 28 42	forage dw forage dw forage dw forage dw forage dw	52 13 6.4 1.4 0.75	GHF-P 1700 97358.03
Australia (SA), 1997–1998 (Canola, Siren)	EC + oil haloxyfop-P-methyl	0.052	4–8 leaf	100	1	0 7 14 28 42	forage dw forage dw forage dw forage dw forage dw	27 10.2 3.7 1.3 0.32	GHF-P 1700 97358.02

OILSEED RAPE country, year (variety)	Application [a] Form [c]	 kg ai/ha	 growth stage	 water (L/ha)	 no.	PHI days	Commodity	haloxyfop, mg/kg	Ref
Australia (SA), 1997–1998 (Canola, Siren)	EC + oil haloxyfop-P-methyl	0.052	budding, pre-flower	100	1	94	fodder dw	0.06	GHF-P 1700 97358.02
Australia (SA), 1997–1998 (Canola, Siren)	EC + oil haloxyfop-P-methyl	0.10	budding, pre-flower	100	1	94	fodder dw	0.17	GHF-P 1700 97358.02
Australia (SA), 1997–1998 (Canola, Siren)	EC + oil haloxyfop-P-methyl	0.10	4–8 leaf	100	1	0 7 14 28 42	forage dw forage dw forage dw forage dw forage dw	56 21 6.9 4.1 1.5	GHF-P 1700 97358.02
Australia (WA), 1997–1998 (Canola, Karoo)	EC + oil haloxyfop-P-methyl	0.052	6–8 leaf	100	1	1 7 14 28 42	forage dw forage dw forage dw forage dw forage dw	19 13 7.7 5.0 3.3	GHF-P 1700 97358.01
Australia (WA), 1997–1998 (Canola, Karoo)	EC + oil haloxyfop-P-methyl	0.052	pre-flower, bud emergence	100	1	98	fodder dw	0.22	GHF-P 1700 97358.01
Australia (WA), 1997–1998 (Canola, Karoo)	EC + oil haloxyfop-P-methyl	0.10	pre-flower, bud emergence	100	1	98	fodder dw	0.39	GHF-P 1700 97358.01
Australia (WA), 1997–1998 (Canola, Karoo)	EC + oil haloxyfop-P-methyl	0.10	6–8 leaf	100	1	1 7 14 28 42	forage dw forage dw forage dw forage dw forage dw	47 19 17 8.9 5.5	GHF-P 1700 97358.01
France, 2000 (Capitol)	EC haloxyfop-P-methyl	0.055	BBCH 57	200	1	104	plant	0.28	GHE-P-8857 CEMS-1254D
France, 2000 (Carolus)	EC haloxyfop-P-methyl	0.055	BBCH 60	215	1	102	plant	0.08	GHE-P-8857 CEMS-1254C
France, 2000 (Pronto)	EC haloxyfop-P-methyl	0.052	BBCH 50	255	1	102	plant	0.15	GHE-P-8858 CEMS-1255
France, 2001–2002 (Synergy)	EC haloxyfop-P-methyl	0.097	BBCH 16	190	1	233	plant	< 0.01	GHE-P-10092 CEMS-1572A
France, 2001–2002 (Zenith)	EC haloxyfop-P-methyl	0.10	BBCH 16	200	1	204	plant	< 0.01	GHE-P-10092 CEMS-1572B
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.079	BBCH 32	300	1	63	plant	0.14	GHE-P-11656 F06W028R T2
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.083	BBCH 39	320	1	63	plant	0.37	GHE-P-11656 F06W028R T3
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.085	BBCH 50	330	1	61	plant	0.36	GHE-P-11656 F06W028R T4
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.056	BBCH 32	320	1	63	plant	0.17	GHE-P-11656 F06W028R T5
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.052	BBCH 39	300	1	63	plant	0.20	GHE-P-11656 F06W028R T6
France, 2006 (Prima)	EC haloxyfop-P-methyl	0.052	BBCH 50	310	1	63	plant	0.27	GHE-P-11656 F06W028R T7

OILSEED RAPE	Application ^{a}					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form ^{c}	kg ai/ha	growth stage	water (L/ha)	no.	days			
France, 2006 (Prima)	EC+adjuvant haloxyfop-P-methyl	0.053	BBCH 32	310	1	61	plant	0.10	GHE-P-11656 F06W028R T8
France, 2006 (variety not recorded)	EC haloxyfop-P-methyl	0.080	BBCH 30-34	200	1	0 7 14 21 28	plant plant plant plant plant	3.1 0.77 0.47 0.36 0.37	GHE-P-11657 F06W080R T2
France, 2006 (variety not recorded)	EC haloxyfop-P-methyl	0.079	BBCH 34-50	200	1	0 7 14 21 28	plant plant plant plant plant	5.4 1.4 0.72 0.34 0.50	GHE-P-11657 F06W080R T3
France, 2006 (variety not recorded)	EC haloxyfop-P-methyl	0.053	BBCH 30-34	200	1	0 7 14 21 28	plant plant plant plant plant	2.7 0.66 0.49 0.33 0.26	GHE-P-11657 F06W080R T5
France, 2006 (variety not recorded)	EC haloxyfop-P-methyl	0.083	BBCH 50	200	1	0 7 14 21 28	plant plant plant plant plant	1.5 1.0 0.64 0.24 0.26	GHE-P-11657 F06W080R T7
France, 2006 (variety not recorded)	EC + adjuvant haloxyfop-P-methyl	0.052	BBCH 30-34	200	1	0 7 14 21 28	plant plant plant plant plant	3.1 0.64 0.30 0.25 0.13	GHE-P-11657 F06W080R T8
Germany, 1988–1989 (Arabella)	EC haloxyfop-P-methyl	0.10	6 leaf (EC 23)	400	1	1 34 184 229 259	whole plant whole plant whole plant whole plant straw	1.4 1.6 0.80 0.77 < 0.02 (2) < 0.02 (2) < 0.05 (2)	GHE-P-2144 R88-21B
Germany, 1988–1989 (Arabella)	EC haloxyfop-ethoxyethyl	0.21	6 leaf (EC 23)	400	1	1 34 184 229 259	whole plant whole plant whole plant whole plant straw	< 0.02 0.02 0.11 0.19 < 0.02 (2) < 0.02 (2) < 0.05 (2)	GHE-P-2144 R88-21B
Germany, 1988–1989 (Arabella)		0 control plot	6 leaf (EC 23)	400	0	1 34 184 229 259	whole plant whole plant whole plant whole plant straw	< 0.02 0.02 < 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.05 (2)	GHE-P-2144 R88-21B
Germany, 1988–1989 (Ceres)	EC haloxyfop-P-methyl	0.10			1	1 42 189 241 272	whole plant whole plant whole plant whole plant straw	4.9 5.7 1.2 1.3 0.04 0.04 0.03 0.02 < 0.05 (2)	GHE-P-2144 R88-21A
Germany, 1988–1989 (Ceres)	EC haloxyfop-ethoxyethyl	0.21	6 leaf (EC 23)	400	1	1 42 189 241 272	whole plant whole plant whole plant whole plant straw	8.7 7.9 1.9 1.7 0.03 < 0.02 0.04 0.04 < 0.05 (2)	GHE-P-2144 R88-21A
Germany, 1988–1989 (Ceres)		0 control plot			0	1 42 189 241 272	whole plant whole plant whole plant whole plant straw	0.06 0.05 < 0.02 (2) < 0.02 (2) 0.02 0.03 < 0.05 (2)	GHE-P-2144 R88-21A
Germany, 2000 (Mohican)	EC haloxyfop-P-methyl	0.051	BBCH 50	255	1	113	plant	0.25	GHE-P-8857 CEMS-1254A

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
Germany, 2000 (Zenith)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	113	plant	0.16	GHE-P-8857 CEMS-1254B
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.081	BBCH 32	310	1	0 7 14 21 28 97	plant plant plant plant plant plant	6.8 0.84 0.36 0.19 0.10 0.034	GHE-P-11657 G06W018R T2
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.084	BBCH 39	320	1	0 7 14 21 28 92	plant plant plant plant plant plant	5.6 0.83 0.53 0.28 0.10 0.077	GHE-P-11657 G06W018R T3
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.075	BBCH 50	290	1	91	plant	0.092	GHE-P-11657 G06W018R T4
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.055	BBCH 32	320	1	0 7 14 21 28 97	plant plant plant plant plant plant	4.0 0.53 0.24 0.16 0.065 0.037	GHE-P-11657 G06W018R T5
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.054	BBCH 39	310	1	92	plant	0.01	GHE-P-11657 G06W018R T6
Germany, 2006 (Heros)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	0 7 14 21 28 91	plant plant plant plant plant plant	1.9 0.52 0.22 0.21 0.19 0.055	GHE-P-11657 G06W018R T7
Germany, 2006 (Heros)	EC + adjuvant haloxyfop-P-methyl	0.055	BBCH 32	320	1	0 7 14 21 28 97	plant plant plant plant plant plant	4.8 0.48 0.23 0.10 0.090 0.03	GHE-P-11657 G06W018R T8
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.082	BBCH 32	310	1	0 7 14 21 28 88	plant plant plant plant plant plant	4.3 1.1 0.66 0.60 0.42 0.35	GHE-P-11657 G06W017R T2
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.078	BBCH 39	300	1	0 7 14 21 28 85	plant plant plant plant plant plant	3.9 1.2 0.77 0.62 0.44 0.35	GHE-P-11657 G06W017R T3
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.085	BBCH 50	330	1	82	plant	0.41	GHE-P-11657 G06W017R T4
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.053	BBCH 32	310	1	0 7 14 21 28 88	plant plant plant plant plant plant	3.6 0.75 0.46 0.32 0.29 0.13	GHE-P-11657 G06W017R T5

OILSEED RAPE country, year (variety)	Application [a] Form [c]	 kg ai/ha	 growth stage	 water (L/ha)	 no.	PHI days	Commodity	haloxyfop, mg/kg	Ref
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.056	BBCH 39	320	1	85	plant	0.26	GHE-P-11657 G06W017R T6
Germany, 2006 (Oase)	EC haloxyfop-P-methyl	0.055	BBCH 50	310	1	0 7 14 21 28 82	plant plant plant plant plant plant	2.3 0.84 0.53 0.47 0.26 0.27	GHE-P-11657 G06W017R T7
Germany, 2006 (Oase)	EC + adjuvant haloxyfop-P-methyl	0.054	BBCH 32	310	1	0 7 14 21 28 88	plant plant plant plant plant plant	1.5 0.74 0.44 0.32 0.27 0.11	GHE-P-11657 G06W017R T8
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.081	BBCH 32	210	1	80	plant	0.16	GHE-P-11656 GR06W013R T2
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.076	BBCH 39	290	1	69	plant	0.092	GHE-P-11656 GR06W013R T3
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.079	BBCH 50-63	300	1	65	plant	0.073	GHE-P-11656 GR06W013R T4
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.052	BBCH 32	200	1	80	plant	0.05	GHE-P-11656 GR06W013R T5
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.052	BBCH 39	300	1	69	plant	0.062	GHE-P-11656 GR06W013R T6
Greece, 2006 (Licosmos)	EC haloxyfop-P-methyl	0.051	BBCH 50-63	290	1	65	plant	0.065	GHE-P-11656 GR06W013R T7
Greece, 2006 (Licosmos)	EC + adjuvant haloxyfop-P-methyl	0.054	BBCH 32	210	1	80	plant	0.055	GHE-P-11656 GR06W013R T8
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.082	BBCH 32	319	1	60	plant	0.17	GHE-P-11656 I06W023R T2
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.078	BBCH 39	300	1	57	plant	0.17	GHE-P-11656 I06W023R T3
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.078	BBCH 50	300	1	53	plant	0.23	GHE-P-11656 I06W023R T4
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.051	BBCH 32	290	1	60	plant	0.075	GHE-P-11656 I06W023R T5
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.052	BBCH 39	300	1	57	plant	0.091	GHE-P-11656 I06W023R T6
Italy, 2006 (Heros)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	53	plant	0.14	GHE-P-11656 I06W023R T7
Italy, 2006 (Heros)	EC + adjuvant haloxyfop-P-methyl	0.054	BBCH 32	310	1	60	plant	0.13	GHE-P-11656 I06W023R T8

OILSEED RAPE	Application [a]					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form [c]	kg ai/ha	growth stage	water (L/ha)	no.	days			
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.082	BBCH 32	320	1	0 7 14 21 28 92	plant plant plant plant plant plant	3.4 0.63 0.23 0.20 0.16 0.16	GHE-P-11657 PL06W009R T2
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.080	BBCH 39	310	1	0 7 14 21 28 89	plant plant plant plant plant plant	2.2 0.51 0.49 0.35 0.13 0.21	GHE-P-11657 PL06W009R T3
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.084	BBCH 50	320	1	87	plant	0.27	GHE-P-11657 PL06W009R T4
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.051	BBCH 32	300	1	0 7 14 21 28 92	plant plant plant plant plant plant	1.6 0.44 0.19 0.21 0.097 0.14	GHE-P-11657 PL06W009R T5
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.055	BBCH 39	320	1	89	plant	0.097	GHE-P-11657 PL06W009R T6
Poland, 2006 (Californium)	EC haloxyfop-P-methyl	0.052	BBCH 50	300	1	0 7 14 21 28 87	plant plant plant plant plant plant	3.0 0.49 0.29 0.20 0.16 0.15	GHE-P-11657 PL06W009R T7
Poland, 2006 (Californium)	EC + adjuvant haloxyfop-P-methyl	0.052	BBCH 32	300	1	0 7 14 21 28 87	plant plant plant plant plant plant	1.5 0.41 0.24 0.16 0.12 0.069	GHE-P-11657 PL06W009R T8
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.079	BBCH 32	300	1	147	plant [b]	< 0.01	GHE-P-11656 S06W027R T2
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.077	BBCH 39	300	1	141	plant [b]	< 0.01	GHE-P-11656 S06W027R T3
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.077	BBCH 50	290	1	134	plant [b]	0.02	GHE-P-11656 S06W027R T4
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.056	BBCH 32	320	1	147	plant [b]	< 0.01	GHE-P-11656 S06W027R T5
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.053	BBCH 39	300	1	141	plant [b]	< 0.01	GHE-P-11656 S06W027R T6
Spain, 2006 (Tracia)	EC haloxyfop-P-methyl	0.053	BBCH 50	300	1	134	plant [b]	< 0.01	GHE-P-11656 S06W027R T7
Spain, 2006 (Tracia)	EC + adjuvant haloxyfop-P-methyl	0.052	BBCH 32	300	1	147	plant [b]	< 0.01	GHE-P-11656 S06W027R T8

[a] Directed: spray directed at ground.

[b] Plants were dried at 30 ºC in a drying oven for 40 hours and separated afterwards by threshing.

[c] Adjuvant used in European trials: rapeseed oil.

Oilseed rape, BBCH growth stages (Meier, 2001)

16 6 leaves unfolded.

30 Beginning of stem elongation: no internodes (“rosette”).

32 2 visibly extended internodes.

34 4 visibly extended internodes.

39 9 or more visibly extended internodes.

50 Flower buds present, still enclosed by leaves.

57 Individual flower buds (secondary inflorescences) visible but still closed.

60 First flowers open.

63 30% of flowers on main raceme open.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on the fate of haloxyfop residues during the processing of oilseed rape for oil and meal, soya beans for oil and meal and sugar beet for sugar. A processing study was also provided for apples, but haloxyfop uses as a directed spray on weeds around apple trees did not produce detectable residues in the apples or processed commodities.

Processing studies were available on the following commodities.

Apples	→	juice, pomace	Table 38
Oilseed rape	→	crude oil, refined and deodorised oil, meal	Table 39
Soya beans	→	crude oil, refined oil, meal	Table 40
Sugar beet	→	sugar, green syrup	Table 41

Processing factors have been calculated for haloxyfop residues in oilseed rape, soya beans and sugar beet.

Haloxyfop-methyl was applied to the grassy area under apple trees (Gardner and Schotts, 1988, GHC-2037) from which apples were subsequently harvested and processed into chopped apples, juice and pomace. Approximately 9–10 kg of apples were harvested and processed in each trial. The haloxyfop residue data are summarised in Table . No residues of haloxyfop occurred in the apples, juice or pomace.

Table 38 Haloxyfop residues in apples and processed commodities resulting from supervised trials in the USA

APPLES	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
USA (CA), 1987 (Golden Delicious)	WG + oil haloxyfop-P-methyl	1.7 [a]		190	1	60	chopped apples juice wet pomace dry pomace	< 0.05 < 0.05 < 0.05 < 0.05	GHC-2037

APPLES	Application					PHI	Commodity	haloxyfop	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		mg/kg	
USA (CA), 1987 (Rome)	WG + oil haloxyfop-P-methyl	0.56 [a]		190	1	59	chopped apples juice wet pomace dry pomace	< 0.05 < 0.05 < 0.05 < 0.05	GHC-2037
USA (MI), 1987 (Macintosh)	WG + oil haloxyfop-P-methyl	1.7 [a]		220	1	60	chopped apples juice wet pomace dry pomace	< 0.05 < 0.05 < 0.05 < 0.05	GHC-2037
USA (MI), 1987 (Golden Delicious)	WG + oil haloxyfop-P-methyl	1.7 [a]		220	1	60	chopped apples juice wet pomace dry pomace	< 0.05 < 0.05 < 0.05 < 0.05	GHC-2037

[a] Directed application for grass control.

Yon (1987, GHE-P-1313R) examined the fate of incurred haloxyfop residues in oilseed rape during processing. Rapeseed (1 kg) was processed in the laboratory simulating commercial practice. Seeds were coarsely ground and extracted with hot hexane. Solvent was evaporated from the oil and the meal. Crude oil was degummed by agitation with 10% phosphoric acid at 90 ºC for 20 minutes. The mixture was then cooled to 40 ºC, neutralized with NaOH and then a 30% alkali was added and the soap was allowed to settle. The oil was decanted, filtered and washed free of soap and dried. The oil was bleached by agitation at 90 ºC for 30 minutes with added Fuller's earth followed by filtration. Deodorisation was effected by steam distillation at 240 ºC under reduced pressure for 1 hour. The oil was then cooled to 100 ºC and citric acid was added as a trace metal sequestrant. Residues in the rape seed and processed fractions are summarised in Table 39.

Table 39 Haloxyfop residues in oilseed rape and processed commodities resulting from supervised trials in France

OILSEED RAPE	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]		
France, 1982–1983 (Winter oilseed rape, Jet Neuf)	EC haloxyfop-ethoxyethyl	0.10	5–6 leaves	400	1	228	rape seed oil, crude oil, ref & d meal	< 0.05 (3) 0.05 0.06 0.06 0.09 0.07 0.08 0.04 0.03 0.04	GHE-P-1313R RT97-100/83
France, 1982–1983 (Winter oilseed rape, Jet Neuf)	EC haloxyfop-ethoxyethyl	0.21	5–6 leaves	400	1	228	rape seed oil, crude oil, ref & d meal	< 0.05 (3) 0.06 0.06 0.06 0.05 0.04 0.05 0.05 0.04 0.05	GHE-P-1313R RT97-100/83
France, 1982–1983 (Winter oilseed rape, Jet Neuf)	EC haloxyfop-ethoxyethyl	0.63	5–6 leaves	400	1	228	rape seed oil, crude oil, ref & d meal	0.06 0.06 0.06 0.09 0.11 0.10 0.08 0.07 0.08 0.11 0.09 0.10	GHE-P-1313R RT97-100/83
France, 1982–1983 (Winter oilseed rape, Jet Neuf)		0 control plot			0	228	rape seed oil, crude oil, ref & d meal	< 0.01 (3) 0.02 0.01 0.02 0.03 0.03 0.02 0.03 < 0.02 (3)	GHE-P-1313R RT97-100/83
France, 1982–1983 (Winter oilseed rape, Jet Neuf)	EC haloxyfop-ethoxyethyl	0.10	Blooming begins	400	1	122	rape seed oil, crude oil, ref & d meal	0.27 0.29 0.28 0.36 0.36 0.49 0.40 0.26 0.41 0.22 0.30 0.24 0.25 0.25	GHE-P-1313R RT93-96/83
France, 1982–1983 (Winter oilseed rape, Jet Neuf)	EC haloxyfop-ethoxyethyl	0.21	Blooming begins	400	1	122	rape seed oil, crude oil, ref & d meal	0.40 0.33 0.37 0.73 0.72 0.73 0.83 0.79 0.81 0.27 0.40 0.34	GHE-P-1313R RT93-96/83

OILSEED RAPE	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	Note [a]		
France, 1982–1983 (Winter oilseed rape, Jet Neuf)	EC haloxyfop-ethoxyethyl	0.63	Blooming begins	400	1	122	rape seed oil, crude oil, ref & d meal	1.25 1.20 1.23 2.27 2.12 2.20 2.31 2.36 2.33 1.09 1.10 1.10	GHE-P-1313R RT93-96/83
France, 1982–1983 (Winter oilseed rape, Jet Neuf)		0 control plot			0	122	rape seed oil, crude oil, ref & d meal	0.017 0.025 0.02 0.067 0.047 0.06 0.059 0.066 0.07 0.031 0.036 0.03	GHE-P-1313R RT93-96/83

[a] Oil, ref & d: refined and deodorised oil.

Gardner (1983, GH-C 1625) reported on the haloxyfop residue levels in soya bean commodities that resulted from laboratory scale processing of soya beans treated in the field with haloxyfop-methyl (Table 40)

Phillips (1991, 89026) described the small-scale processing of soya bean used in that residue study. After bringing the moisture content to a suitable level, ideally 7–10%, the hull and kernel can be separated and the seed can be cleaned of light impurities such as dust and soil by aspiration. Passage over a screen of suitable size is used to separate whole seed from small and large pieces of plant material. Kernels are cracked in a mill and the kernel material is heated to 66–74 ºC in a small cooker. Crude oil is then extracted from the flaked kernels with hexane in a steam-jacketed extractor. The extracted flakes are milled to a smaller particle size—the meal. Crude oil is obtained by evaporation of the hexane. Crude oil is refined by an AOCS method that removes free fatty acids.

Soya beans, hulls and meal were analysed for haloxyfop residues by method ACR 83.1R.s1, while crude oil, refined oil and soapstock relied on method ACR 86.6.S2 (Phillips, 1991, 89026).

Table 40 Haloxyfop residues in soya beans and processed commodities resulting from supervised trials in the USA

SOYA BEANS	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days			
USA (IL), 1982 (Northrup King 1492)	EC + oil haloxyfop-methyl	0.28	in bloom		1	69	soya beans hulls meal crude oil refined oil soapstock	0.68 0.56 0.81 0.85 0.83 0.72	GH-C 1625
USA (MI), 1989 (Harden)	EC + oil haloxyfop-P-methyl	0.70	5th trifoliate	190	1	97	soya beans hulls meal crude oil refined oil soapstock	0.55 0.39 0.71 0.22 0.18 0.24	89026
USA (IL), 1989 (Agri Pro 2021)	EC + oil haloxyfop-P-methyl	0.70	5th trifoliate	190	1	96	Composite soya bean sample from IL and MS trials processed		89026
USA (MS), 1989 (Braggs)	EC + oil haloxyfop-P-methyl	0.70	5th trifoliate	190	1	88	soya beans hulls meal crude oil refined oil soapstock	0.24 0.15 0.30 0.19 0.18 0.27	89026

Yon (1984, GHE-P-1125) treated a sugar beet crop with haloxyfop-ethoxyethyl and processed the harvested beets to sugar. The process was pilot scale (25 kg) and consisted of washing, slicing, water extraction, pressing, filtration, calcium carbonate precipitation-filtration, boiling and centrifuging. The residue data are summarised in Table 41.

Table 41 Haloxyfop residues in sugar beet and processed commodities resulting from supervised trials in the UK

SUGAR BEET	Application					PHI	Commodity	haloxyfop, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	growth stage	water (L/ha)	no.	days	[a]	[b]	
UK, 1983 (Amono)	EC haloxyfop-ethoxyethyl	0.25	6–8 leaf	200	1	66	cossettes raw juice pressed pulp refined sugar green syrup	0.04 0.09 0.02 0.04 0.02 0.04 < 0.01 (2) 0.15 0.28	GHE-P-1125
UK, 1983 (Amono)	EC haloxyfop-ethoxyethyl	0.50	6–8 leaf	200	1	66	cossettes raw juice pressed pulp refined sugar green syrup	0.15 0.07 0.09 0.08 0.05 0.03 < 0.01 (2) 0.33 0.32	GHE-P-1125

[a] Cossettes: sliced raw beets. Green syrup: the liquor from the second last crystallizer, comparable with molasses, the liquor from the final crystallizer.

[b] GHE-P-1125: 2 replicate batches processed.

Table 42 Summary of processing factors for haloxyfop residues. The factors are calculated from the data recorded in tables in this section

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors.	Median or best estimate
Oilseed rape	crude oil	1.1, 1.2, 1.4, 1.7, 1.8, 2.0	1.55
Oilseed rape	refined oil	0.93, 1.1, 1.3, 1.7, 1.9, 2.2	1.47
Oilseed rape	meal	0.73, 0.88, 0.89, 0.92, 0.93, 1.7	0.91
Soya beans	crude oil	0.40, 0.79, 1.3	0.79
Soya beans	refined oil	0.33, 0.75, 1.2	0.75
Soya beans	meal	1.19, 1.25, 1.29	1.25
Sugar beet	refined sugar	< 0.09, < 0.15	< 0.09
Sugar beet	green syrup	2.95, 3.31	3.1

RESIDUES IN ANIMAL COMMODITIES

Livestock feeding studies

The meeting received beef cattle feeding studies with haloxyfop and haloxyfop-P, dairy cattle studies with haloxyfop and haloxyfop-P and a laying hen study with haloxyfop. These livestock feeding studies provided information on likely haloxyfop residues resulting in bovine tissues and milk and poultry tissues and eggs from haloxyfop residues in the animal diet.

Beef cattle

Groups of three beef calves, Hereford, Angus, Angus/Hereford, Limousin, Hereford/Limousin, (animals weighing 219–280 kg at pre-dose) were dosed once daily via gelatin capsule with haloxyfop at nominal 0.25, 0.5, 1, 5 and 10 ppm in the dry-weight diet, for 28 consecutive days (Kutschinski and Bjerke, 1984, GH-C 1680). The animal ration consisted of corn silage (23% dry matter) *ad libitum* and approximately 0.5 kg of protein supplement (91% dry matter). Average feed consumption per day was typically in the 15–20 kg range. Animals were slaughtered 18 to 21 hours after the final dose for tissue collection. Additional groups of animals at the highest dose were kept for 7 and 14 days after the final dose to observe declines in residue levels. Tissue samples were analysed for haloxyfop and its conjugates by method ACR 84.1. Samples were analysed within 10 weeks of sampling day. The residue data are summarised in Table 43.

Although large variations in residue levels occurred between animals receiving the same dose, residue levels were approximately proportional to doses for kidney, liver and fat. Residue levels for muscle were below LOQ, except at the two highest doses. Residues in kidney and liver depleted substantially within 7 days of the final dose. In fat, residues depleted in 7 days but were apparently persistent in the longer interval.

For animals dosed at 5 and 10 ppm, residues in muscle equalled or exceeded LOQ and ratios between residue levels in fat and muscle were calculated: mean = 7.8, range 3.6–18.5, n = 5. These values suggest fat solubility, at least for the conjugate.

Table 43 Haloxyfop residues in tissues of beef calves dosed once daily via gelatin capsule with haloxyfop at nominal 0.25, 0.5, 1, 5 and 10 ppm in the dry-weight diet, for 28 consecutive days (Kutschinski and Bjerke, 1984, GH-C 1680). Animals dosed at 10 ppm were kept for 7 and 14 days after the final dose to observe declines in residue levels.

	Haloxyfop, mg/kg. Individual animal data.			
Dosing group, ppm in feed	Muscle	Liver	Kidney	Fat
0.25 ppm	< 0.01 (3)	0.03 < 0.01 (2)	0.10 0.02 0.03	0.03 < 0.01 (2)
0.5 ppm	< 0.01 (3)	0.01 0.02 0.03	0.03 0.06 0.11	0.01 0.01 < 0.01
1 ppm	< 0.01 (3)	0.03 0.06 0.05	0.11 0.15 0.15	< 0.01 0.01 < 0.01
5 ppm	0.01 0.01	0.12 0.14	0.46 0.32	0.068 0.045
10 ppm	0.02 0.05 0.03	0.36 0.65 0.61	0.75 1.7 1.3	0.41 0.19 0.21
10 ppm + 7 days	< 0.01 (3)	0.02 0.02 0.21	0.05 0.03 0.54	0.16 0.06 0.01
10 ppm + 14 days	< 0.01 (3)	0.02 < 0.01 (2)	0.05 0.03 0.02	0.17 0.05 0.05

Groups of three Angus cross beef cattle (animals weighing 450–650 kg) were dosed twice a day via gelatin capsules with haloxyfop-P at nominal concentrations 10, 20 and 30 ppm in the dry-weight diet, for 28 consecutive days (Cowles, 2003, GHF-P 1939). The animal ration consisted of hay. Average dry matter consumption was 8.4 kg per animal per day during the acclimatisation period. Animals were slaughtered on day 28 for tissue collection. Additional animals at the highest dose were kept for 7, 14, 21 and 28 days after the final dose to observe declines in residue levels. Tissue samples were analysed for haloxyfop by methods NCRL-PHT-01 (liver, kidney, muscle) and NCRL-PHF-01 (fat). Samples were stored for 9–12 months before analysis. The residue data are summarised in Table 44.

Table 44 Haloxyfop residues in tissues from Angus cross beef cattle dosed via gelatin capsules with haloxyfop-P at nominal concentrations 10, 20 and 30 ppm in the dry-weight diet, for 28 consecutive days (Cowles, 2003, GHF-P 1939)

	Haloxyfop residues, mg/kg. Individual animal data		
TISSUE	Dosing group 10 ppm	Dosing group 20 ppm	Dosing group 30 ppm
Abdominal fat [a]	0.02 0.01 0.01	0.05 0.03 0.03	0.02 0.02 0.04
Kidney	0.51 0.53 0.57	1.1 1.5 0.53	1.2 0.48 1.8
Liver	0.23 0.33 0.20	0.38 0.45 0.30	0.46 0.20 0.18
Muscle	0.02 0.05 0.02	0.06 0.06 0.03	0.05 0.01 0.05
Renal fat [a]	0.02 0.01 0.01	0.04 0.02 0.02	0.02 0.02 0.04
SC fat [a]	0.02 0.01 0.01	0.03 < 0.01 0.03	0.02 0.01 0.03

[a] Analytical method NCRL-PHF-01 has no hydrolysis step, so the residue data for fat are unlikely to recover haloxyfop residues quantitatively from their triacylglyceride conjugates.

Table 45 Haloxyfop residues in tissues from Angus cross beef cattle dosed via gelatin capsules with haloxyfop-P at a nominal concentration of 30 ppm in the dry-weight diet, for 28 consecutive days and then observed for a further 28 days on a residue free diet (Cowles, 2003, GHF-P 1939)

	Haloxyfop residues, mean, mg/kg.		
Days after final dose	Muscle	Liver	Kidney
0	0.04	0.28	1.15
7	< 0.01	0.09	0.08
14	< 0.01	0.02	0.02
21	< 0.01	0.01	0.01
28	< 0.01	< 0.01	0.01

Dairy cattle

Groups of three lactating Holstein dairy cows (animals weighing 540–720 kg at pre-dose) were dosed through the feed with haloxyfop at nominal concentrations 0.25, 0.75 and 2.5 ppm in the dry-weight diet (measured 0.24, 0.72 and 2.7 ppm), for 28 consecutive days (Gardner, 1984, GH-C 1709). The animal ration consisted of corn silage, alfalfa hay, high moisture corn, whole cotton seed, dried brewer's grain and ground dry corn. Average daily feed consumption was 24–25 kg fresh weight or 14–15 kg dry weight. Total dry weight = 57.8% of fresh weight.

Milk was collected twice daily and milk samples for each cow were obtained by combining equal volumes from the two milkings. Milk production was approximately 9–18 kg/day. Milk from the morning milking was put through a separator to produce the cream. Milk and cream samples were analysed for haloxyfop and its conjugates by method ACR 84.6. It is not entirely clear, but samples may have been stored for approximately 12 months before analysis. The residue data are summarised in Table 46.

The butter fat content of the milk on day 10 was measured, with values of 3.9%, 4.1% and 4.1% for the milk from the three cows at the 2.5 ppm dosing level. The butter fat content of cream (not only day 10) was found to be in the range 44% to 62%.

Residue data were available for cream and milk on an individual animal basis for days 3 and 10. Average (and range) of haloxyfop residue levels were: day 3 cream 0.303 mg/kg (0.24–0.34 mg/kg) and milk 0.013 mg/kg (0.01–0.015 mg/kg); and day 10 cream 0.33 mg/kg (0.28–0.42 mg/kg) and milk 0.025 mg/kg (0.005–0.039 mg/kg). The average for 'milk residues ÷ cream residues' from day 3 and day 10 were 0.041 and 0.076 respectively.

Residue levels in milk and cream show reasonable proportionality to the dose. The residue is fat soluble. When the dosing is withdrawn, haloxyfop residues in milk and cream deplete within a few days.

Table 46 Haloxyfop residues in milk and cream from Holstein dairy cows dosed through the feed with haloxyfop at nominal concentrations 0.25, 0.75 and 2.5 ppm in the dry-weight diet for 28 consecutive days (Gardner, 1984, GH-C 1709)

	Haloxyfop residues, mg/kg. Individual cow data		
MILK	Dosing group 0.25 ppm	Dosing group 0.75 ppm	Dosing group 2.5 ppm
Day 3	< 0.01 (3)	< 0.01 < 0.01 0.01	0.015 0.01 0.01
5	< 0.01 (3)	0.01 < 0.01 0.02	0.041 0.01 0.031
7	< 0.01 (3)	0.01 0.01 0.026	0.042 0.055 0.038
10	< 0.01 (3)	0.01 < 0.01 0.023	0.039 < 0.01 0.031
15	< 0.01 (3)	< 0.01 < 0.01 0.02	0.037 0.021 0.036
20	< 0.01 (3)	0.01 < 0.01 0.02	0.044 0.033 0.032
25	0.01 < 0.01 < 0.01	0.015 < 0.01 0.01	0.040 0.036 0.035
28	< 0.01 (3)	0.01 < 0.01 0.015	0.032 0.024 0.035
Withdraw 1 day	< 0.01 (3)	< 0.01 < 0.01 0.01	0.032 0.030 0.027

	Haloxyfop residues, mg/kg. Individual cow data		
Withdraw 3 days	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
Withdraw 5 days	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
Withdraw 7 days	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
Withdraw 14 days	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
CREAM	Dosing group 0.25 ppm	Dosing group 0.75 ppm	Dosing group 2.5 ppm
Day 3	0.037 0.084 0.029	0.11 0.10 0.27	0.34 0.24 0.33
10	0.046 0.046 0.048	0.13 0.12 0.22	0.42 0.29 0.28
17	0.043 0.045 0.051	0.13 0.11 0.15	0.41 0.30 0.34
28	< 0.02 0.02 < 0.02	0.02 0.035 0.093	0.20 0.18 0.090
Withdraw 1 day	0.041 0.056 0.029	0.17 0.12 0.088	0.42 0.39 0.39
Withdraw 7 days	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)

Groups of three lactating Friesian dairy cows (animals weighing 452–586 kg) were dosed twice a day via gelatin capsules with haloxyfop-P at nominal concentrations 10, 20 and 30 ppm in the dry-weight diet, for 28 consecutive days (Cowles, 2003, GHF-P 1939). The sole feed was pasture, eaten on a voluntary basis, later supplemented with grass silage. Average dry matter consumption was 14 kg per cow per day during the acclimatisation period.

Milk was collected twice daily and milk samples for each cow were obtained by combining volumes from the two milkings in proportion to production volumes. Information on milk production was not available. Milk was analysed for haloxyfop using method NCRL-PHM-01, which does not include a hydrolysis step and therefore is unlikely to recover haloxyfop residues quantitatively from their triacylglyceride conjugates. Samples were stored for approximately 12–15 months before analysis. The residue data are summarised in Table 47.

Haloxyfop residue levels in milk reached a plateau by day 10 of the dosing. Residue levels were approximately proportional to doses, but the relationship was obscured by the large variations that occurred between animals of the same dosing group. Residue levels had noticeably declined by day 30, i.e. 2 days after dosing had ceased and, by day 38, residue levels from the 10 and 20 ppm groups had fallen to the LOQ (0.01 mg/kg) or lower.

Table 47 Haloxyfop residues in milk from Friesian dairy cows dosed via gelatin capsules with haloxyfop-P at nominal concentrations 10, 20 and 30 ppm in the dry-weight diet, for 28 consecutive days (Cowles, 2003, GHF-P 1939). Samples were analysed by method NCRL-PHM-01, which apparently would not recover haloxyfop residues quantitatively from their triacylglyceride conjugates.

	Haloxyfop residues, mg/kg. Individual cow data.		
MILK	Dosing group 10 ppm	Dosing group 20 ppm	Dosing group 30 ppm
Day 1	0.01 0.01 0.01	0.01 0.02 0.02	0.02 0.02 0.03
2	0.12 0.20 0.14	0.19 0.39 0.33	0.42 0.24
6	0.18 0.23 0.20	0.24 0.43 0.40	0.54 0.57 0.27
10	0.33 0.38 0.31	0.48 0.87 0.59	1.29 1.83 0.42
14	0.28 0.27 0.20	0.36 0.54 0.56	0.60 0.73 0.28
18	0.22 0.29 0.22	0.28 0.70 0.54	0.70 0.74 0.33
22	0.23 0.25 0.17	0.20 0.59 0.56	0.63 0.72 0.20
26	0.58 0.65 0.37	0.22 0.97 0.91	1.01 2.21 0.37
30	0.40 0.32 0.09	0.09 0.48 0.59	0.61 0.95 0.19
34	0.13 0.07 0.01	0.03 0.04 0.06	0.35 0.52 0.02
38	0.01 0.01 < 0.01	< 0.01 0.01 1.00	0.07 0.07 < 0.01
42	< 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01	< 0.01 0.11 < 0.01

Laying hens

Groups of 12 White Leghorn laying hens, (birds weighing approximately 1.5 kg) received haloxyfop in the feed at nominal concentrations of 0.25, 0.75, and 2.5 ppm in the diet, for 28 consecutive days (Kutschinski and Bjerke, 1984, GH-C 1701). Birds were fed with a proprietary layer feed, provided 400 grams at a time (88% dry weight). Total feed consumption for each hen was determined each day by direct weighing. Average feed consumption per day was typically in the 90–120 g range. Eggs were collected twice daily. Eggs collected each day from a group were opened and the contents were pooled. Birds were slaughtered approximately 24 hours after the final dose for tissue collection. Additional groups of birds at the highest dose were kept for 7 and 14 days after the final dose to observe declines in residue levels. Tissue samples and eggs were analysed for haloxyfop and its conjugates by method ACR 84.1. Tissue samples were analysed within 3 months of sampling day and eggs within 6 months. The residue data are summarised in Table 48.

Haloxyfop residue levels were approximately proportional to dose for liver and fat. Residue levels for muscle and skin were below LOQ for the lowest dose group, but were above LOQ for all birds at the highest dose, 2.5 ppm. Residues in liver and muscle depleted substantially within 7 days of the final dose. In fat, residues were much more persistent. Residue levels in eggs were generally low and disappeared quickly (below LOQ after approximately 7 days) when dosing (2.5 ppm) was withdrawn.

Residue levels in fat were approximately 4–5 times as high as in the muscle for the 2.5 ppm dosing group on day 28 and an average 14 times on day 35 for cases where residues in muscle exceeded the LOQ (0.01 mg/kg). This suggests a fat soluble residue, at least for the conjugated residue.

Table 48 Haloxyfop residues in tissues of laying hens dosed via the feed with haloxyfop at nominal concentrations of 0.25, 0.75, and 2.5 ppm in the diet, for 28 consecutive days (Kutschinski and Bjerke, 1984, GH-C 1701). Birds dosed at 2.5 ppm were kept for 7 and 14 days after the final dose to observe declines in residue levels.

	Haloxyfop, mg/kg. Mean and range for the group.			
Dosing group, ppm in feed	Muscle + skin	Liver	Fat	Eggs, day 4 to day 28 (n = 11)
0.25 ppm	< 0.01	0.033 (0.011–0.083)	0.013 (< 0.01–0.028)	< 0.01
0.75 ppm	0.014 (< 0.01–0.02)	0.12 (0.060–0.19)	0.045 (0.014–0.11)	0.014 (< 0.01–0.020)
2.5 ppm	0.063 (0.023–0.11)	0.36 (0.18–0.64)	0.26 (0.11–0.54)	0.036 (0.015-0.052)
2.5 ppm + 7 days	0.01 (< 0.01–0.023)	< 0.01 (< 0.01–0.018)	0.17 (0.040–0.68)	–
2.5 ppm + 14 days	0.013 (< 0.01–0.024)	< 0.01	0.16 (0.053–0.31)	< 0.01 (day 36–42)

Livestock feeding summary

Table 49 Summary of haloxyfop livestock feeding studies showing the dose, expressed as concentration in the dry matter diet, and the resultant residue levels in animal commodities

Dose equivalent, ppm in feed	Residues, mg/kg Mean, highest (in parentheses)				
BEEF CATTLE	Muscle	Liver	Kidney	Fat	Study
0.25 ppm haloxyfop	< 0.01	0.02 (0.03)	0.05 (0.1)	0.02 (0.03)	GH-C 1680
0.5 ppm haloxyfop	< 0.01	0.02 (0.03)	0.07 (0.11)	0.01 (0.01)	GH-C 1680
1 ppm haloxyfop	< 0.01	0.05 (0.05)	0.14 (0.15)	0.01 (0.01)	GH-C 1680
5 ppm haloxyfop	0.01 (0.01)	0.13 (0.14)	0.39 (0.46)	0.057 (0.068)	GH-C 1680
10 ppm haloxyfop	0.03 (0.05)	0.54 (0.65)	1.3 (1.7)	0.27 (0.41)	GH-C 1680
10 ppm haloxyfop-P	0.03 (0.05)	0.25 (0.33)	0.58 (0.70)	0.013 (0.02) [a]	GHF-P 1939

Dose equivalent, ppm in feed	Residues, mg/kg Mean, highest (in parentheses)				
20 ppm haloxyfop-P	0.05 (0.06)	0.38 (0.45)	1.0 (1.5)	0.029 (0.05) [a]	GHF-P 1939
30 ppm haloxyfop-P	0.04 (0.05)	0.28 (0.46)	1.2 (1.8)	0.024 (0.04) [a]	GHF-P 1939
DAIRY CATTLE	Milk	Cream			
0.25 ppm haloxyfop	< 0.01	0.041			GH-C 1709
0.75 ppm haloxyfop	0.01	0.12			GH-C 1709
2.5 ppm haloxyfop	0.034	0.29			GH-C 1709
10 ppm haloxyfop-P	0.317 [a]		Mean from days 10–26, i.e. the plateau		GHF-P 1939
20 ppm haloxyfop-P	0.558 [a]		Mean from days 10–26, i.e. the plateau		GHF-P 1939
30 ppm haloxyfop-P	0.804 [a]		Mean from days 10–26, i.e. the plateau		GHF-P 1939
LAYING HENS	Muscle	Liver	Fat	Eggs	
0.25 ppm haloxyfop	< 0.01	0.033 (0.083)	0.013 (0.028)	< 0.01	GH-C 1701
0.75 ppm haloxyfop	0.014 (0.02)	0.12 (0.19)	0.045 (0.11)	0.014 (0.020)	GH-C 1701
2.5 ppm haloxyfop	0.063 (0.11)	0.36 (0.64)	0.26 (0.54)	0.036 (0.052)	GH-C 1701

[a] The analytical methods for fat (NCRL-PHF-01) and milk (NCRL-PHM-01) did not include a hydrolysis step, so may not have quantitatively recovered haloxyfop from triacylglyceride conjugates.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was available.

NATIONAL RESIDUE DEFINITIONS

Australia

Haloxyfop: Sum of haloxyfop, its esters and conjugates, expressed as haloxyfop.

European Union

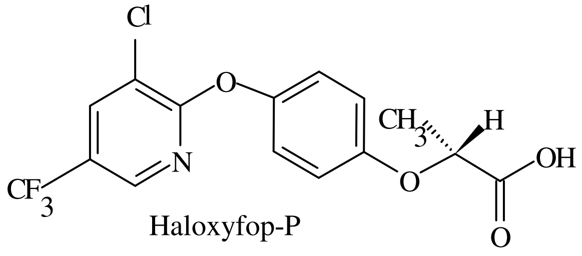
Haloxyfop including haloxyfop-R (haloxyfop-R methyl ester, haloxyfop-R and conjugates of haloxyfop-R expressed as haloxyfop-R).
The residue is considered as fat soluble.

New Zealand

Sum of haloxyfop esters, haloxyfop and its conjugates, expressed as haloxyfop.

APPRAISAL

Residue and analytical aspects of haloxyfop were evaluated by the JMPR in 1995, 1996 and 2001. The compound was listed in the Periodic Re-Evaluation Program at the Thirty-ninth Session of the CCPR (2007) for periodic review by the 2009 JMPR. The most recent toxicological review by JMPR was in 2006 when a group ADI of 0–0.0007 mg/kg bw and a group ARfD of 0.08 mg/kg bw were established for racemic haloxyfop, haloxyfop-R and their methyl esters. For the residue evaluation, the primary manufacturer provided a full residue data package. GAP information was also provided by Australia and The Netherlands.



Haloxyfop was originally produced as a racemic mixture for use as a herbicide for controlling grassy weeds. The compound is now available as the R-isomer, which is the herbicidally active one and is produced commercially as the methyl ester. The ISO name for the R isomer is haloxyfop-P. The ISO name for the unresolved isomeric mixture is haloxyfop.

Animal metabolism

The 2006 JMPR evaluated laboratory animal (mice, rats, dogs and monkeys) metabolism studies of orally administered haloxyfop esters and salts and reported that the different isomers, esters and salts of haloxyfop end up as the de-esterified R enantiomer. This suggests that studies on haloxyfop or haloxyfop-P are mutually supportive.

When two lactating goats were dosed with phenyl ring labelled haloxyfop via gelatin capsule twice daily for 10 consecutive days at the equivalent of 16 ppm haloxyfop in the feed, most of the administered dose (92% and 84%) was excreted in urine, with 1.9% and 1.5% in faeces. Milk accounted for 1.9% and 3.2% of the dose, and tissues less than 0.5% of the dose. Residues in milk reached a plateau very quickly, within 24 hours of the first dose. Monitoring on one goat for 10 hours did not detect [^{14}C] volatiles or $^{14}CO_2$.

Radiolabel, expressed as haloxyfop, was higher in the kidney (1.45 and 1.07 mg/kg) and liver (0.45 and 0.31 mg/kg) than in fat or muscle. The residues in kidney and liver consisted mostly of parent haloxyfop, but some may have been present as labile conjugates.

Radiolabel levels in milk were 0.25 and 0.20 mg/kg. The ^{14}C residues in milk fat were nonpolar and were susceptible to alkaline hydrolysis or lipase hydrolysis releasing haloxyfop. The behaviour was consistent with haloxyfop conjugated as triacylglycerides. Residues in body fat were of the same nature as the residues in milk fat.

When four laying hens were dosed with phenyl ring labelled haloxyfop via gelatin capsule for 11 consecutive days at the equivalent of 12 ppm haloxyfop in the feed, most of the administered dose (82–90%) was excreted in the droppings or present as gut contents (5.8–8.6%). Eggs accounted for an average of 1.6% of the label, and tissues approximately 2%.

Radiolabel, as haloxyfop, was higher in the liver (1.2–2.5 mg/kg) than in the muscle (0.02–0.35 mg/kg) or fat (0.46–2.0 mg/kg). Alkaline hydrolysis of solvent extracts from liver, kidney and fat converted the residues almost quantitatively to a single product, haloxyfop. Most likely, parent haloxyfop was largely incorporated into lipids from which it could be readily released by hydrolysis.

Radio-labelled residue levels were much higher in the yolk (2.0–4.0 mg/kg) than in the whites (0.12–0.37 mg/kg) of eggs (day 10) and reached a plateau in yolks on approximately the 7th day of dosing. Almost the entire residue in the yolks was present as triacylglycerides. Mild alkaline hydrolysis or lipase hydrolysis produced haloxyfop as the single product.

In summary, the metabolism of haloxyfop in goats and hens is similar and also similar to metabolism in laboratory animals in the respect that the esters are de-esterified with little further breakdown of the parent compound. Haloxyfop is readily conjugated and incorporated into fats or secreted in the lipid of milk or eggs. The intact haloxyfop may be released from its conjugates by mild alkaline or enzymatic hydrolysis.

Plant metabolism

The Meeting received plant metabolism studies with haloxyfop-butyl in cotton; haloxyfop-methyl, haloxyfop-butyl and haloxyfop-ethoxyethyl in soya beans; and haloxyfop-P-methyl in sugar beet and lettuce.

The distribution of radiolabel in cotton seed, oil, lint and field trash was reported for cotton that had been foliar treated with phenyl ring labelled haloxyfop butyl ester at a rate equivalent to 0.56 kg ai/ha and sampled 78 and 105 days after treatment. Concentrations of radiolabel on day 78, expressed as haloxyfop, were: cotton seed 0.78 mg/kg, oil 1.1 mg/kg and lint 0.19 mg/kg. By day 105, radiolabel concentrations had become: cotton seed 0.20 mg/kg, oil 0.38 mg/kg, lint 0.04 mg/kg and field trash 1.1 mg/kg.

None of the residue in any component was identified as haloxyfop butyl ester. In the cotton seed, almost all of the ^{14}C was accounted for as haloxyfop free acid (32% at day 105) and haloxyfop conjugates (66% at day 105). In the oil, 100% of the ^{14}C was present as haloxyfop conjugates. In the field trash, the radiolabel was present as free acid (39%) and conjugates (55%).

The ^{14}C in the oil was associated with the triglycerides. Lipase hydrolysis and alkali hydrolysis released 91–99.8% of the ^{14}C as haloxyfop, suggesting that the non-polar residues were triglyceride esters of haloxyfop.

In a soya bean metabolism study, the mature second and developing third trifoliate leaves of 20 day old soya bean plants were treated with [^{14}C]labelled haloxyfop in the form of an ester (methyl, butyl and ethoxyethyl) at a dose equivalent to 0.2 mg per plant. Labelling was in the phenyl ring or the pyridyl ring. Treated leaves and the remainder of the plant were sampled 2, 4 and 8 days after treatment.

The distribution of radiolabel was very similar in plants treated with haloxyfop-methyl phenyl label and pyridyl label, suggesting that the haloxyfop molecule had remained intact.

The esters hydrolysed rapidly. Even after 2 days, little of the applied ester remained in the treated leaves. After 8 days, polar metabolites and haloxyfop accounted for 58–65% and 34–40% of the label respectively in the treated leaves. The nature of the applied ester seemed to have little influence on the nature and distribution of the residue.

Applied ester did not appear in untreated portions of the plant. After 8 days, polar metabolites and haloxyfop accounted for 35–39% and 61–65% of the label respectively in the untreated parts of the plant, i.e., unconjugated haloxyfop was the major component of the residue. Mild alkaline hydrolysis of the polar translocated residue released haloxyfop, demonstrating that at least part of the polar fraction consisted of haloxyfop conjugates.

In a second soya bean metabolism study, plants were treated once with [^{14}C]haloxyfop-butyl at two plant growth stages, 89 and 61 days before harvest and at two application rates, 0.28 and 0.56 kg ai/ha. Two labels were used: a phenyl ring label and a pyridyl label.

The parallel behaviour of the phenyl ring and the pyridyl labelled haloxyfop showed that the haloxyfop molecule remained intact and essentially the entire residue contained both the phenyl and pyridyl rings.

Radiolabelled residue levels, expressed as haloxyfop, in the beans for the two treatment rates were 3.1–5.8 mg/kg 61 days after treatment and 0.8–1.3 mg/kg 89 days after treatment. The composition of the residue was essentially the same after both treatments, i.e., unconjugated haloxyfop 57–59%, polar conjugates 17–20% and non-polar conjugates 17–18%. Alkaline and lipase hydrolysis of the non-polar residue from the beans suggested that haloxyfop was incorporated into the oil triglycerides. Most of the polar conjugates also produced haloxyfop on hydrolysis.

Polar conjugates (65–66%) were the main component of the residues in treated soya bean forage 15 days after treatment, with unconjugated haloxyfop (27–30%) accounting for most of the remainder. In new-growth forage sampled at the same time, unconjugated haloxyfop and polar conjugates accounted for 42% and 57% of the residue respectively. In soya bean straw, unconjugated haloxyfop accounted for the majority of the residue (60–66%) with polar conjugates (24–32%) making up most of the remainder.

In a sugar-beet metabolism study, young plants in field plots were foliar sprayed at 0.22 kg ai/ha with pyridyl-labelled haloxyfop-P-methyl formulated as an EC. At maturity, 92 days after application, ^{14}C residues expressed as haloxyfop-P-methyl were much lower in the roots (0.019 mg/kg) than in the shoots (0.079 mg/kg).

The composition of the residue in sugar beet roots at maturity was: 31% haloxyfop-P acid, 19% conjugate 1, 20% haloxyfop-P glycoside conjugate 1 and 20% unextracted. The composition of the residue in sugar beet shoots at maturity was: 33% haloxyfop-P acid, 24% conjugate 1, 14% haloxyfop-P glycoside conjugate 1 and 12% unextracted.

In summary, haloxyfop-P readily translocated to the roots of treated sugar beet. The majority of the residue was present as polar conjugates.

In a lettuce metabolism study, plants in field plots were foliar sprayed at 0.11 kg ai/ha with pyridyl-labelled haloxyfop-P-methyl formulated as an EC. By day 14, haloxyfop-P-methyl had disappeared and haloxyfop-P acid was the major component of the residue. At maturity, 29 days after treatment, ^{14}C residues expressed as haloxyfop-P-methyl were at higher levels in the outer leaves (0.16 mg/kg) than in the inner leaves (0.048 mg/kg).

The main residue component in lettuce inner leaves at maturity was haloxyfop-P acid at 93% of the ^{14}C, with 5.4% accounted for by various conjugates. The ^{14}C residue in lettuce outer leaves consisted of: 38% haloxyfop-P acid, 24% conjugate 1, 23% glycoside conjugate 2, 10% unextracted and 6.9% glycoside conjugate 1.

Summary of haloxyfop in plant metabolism—when applied to a plant, the esters of haloxyfop or haloxyfop-P are broken down quickly to release free acid which is readily translocated throughout the plant. The haloxyfop (or haloxyfop-P) becomes conjugated, typically as glycosides (polar metabolites) or as triglycerides (non-polar metabolites), the conjugates often accounting for the major part of the residue.

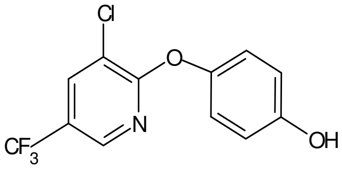
Environmental fate in soil

The Meeting received information on soil aerobic metabolism and soil photolysis properties of [^{14}C]haloxyfop-P-methyl. Studies were also received on the behaviour of [^{14}C]labelled haloxyfop-butyl in a rotational crop situation and haloxyfop-methyl in an unconfined rotational crop situation.

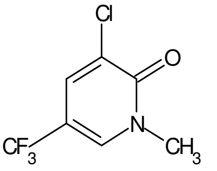
Haloxyfop residues are generally not persistent in soils. Haloxyfop residues in soils resulting from recommended uses should not contribute to the residues in root vegetables or to residues in succeeding crops.

In soil incubation studies under aerobic conditions at 20 °C, parent haloxyfop-P-methyl disappeared with a half-life of approximately 0.5 days. Haloxyfop-P-methyl was hydrolysed just as quickly in a sterile soil as in a fresh soil, demonstrating that the methyl ester is chemically labile. Haloxyfop-P acid was persistent in the sterile soil.

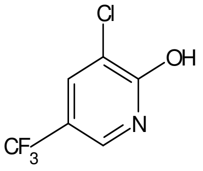
Under aerobic soil incubation, the first metabolite was haloxyfop-P acid, which mostly disappeared with half-lives in the range of 9–21 days (n = 8), but in subsoils with low organic carbon its disappearance half-lives were 28 and 129 days. After approximately 9 months, 6–33% of the dose (haloxyfop-P-methyl labelled in the pyridyl ring) had been mineralized and 28–46% was unextracted.



phenol metab



pyridinone metab



pyridinol metab

The metabolites 'phenol metab', 'pyridinone metab' and 'pyridinol metab' were consistently produced, with the 'pyridinone metab' apparently the most persistent.

In a soil photolysis study with labelled haloxyfop-P-methyl on the surface of a sandy clay loam, degradation rates in the dark controls and the photolysis samples were similar, suggesting that photolysis had negligible effect compared with hydrolysis and metabolism.

In a confined rotational crop study with wheat, soya beans, leaf lettuce, carrots and turnips, a plot of sandy loam soil was treated with [^{14}C]phenyl ring labelled haloxyfop-butyl at the equivalent of 0.56 kg ai/ha and the crops were sown 30 days later. Crops were harvested at various intervals after sowing: lettuce 49 days, soya bean forage 56 days, turnips 64 days, carrots 124 days, wheat 110 days and soya beans 145 days.

The ^{14}C contents of the plant tissues, expressed as haloxyfop on fresh weight, were: lettuce 0.01 mg/kg, turnip foliage < 0.01 mg/kg, turnip root < 0.01 mg/kg, wheat grain 0.01 mg/kg, wheat straw 0.02 mg/kg, soya bean forage 0.07 mg/kg, soya bean grain < 0.01 mg/kg, soya bean straw 0.01 mg/kg, carrot foliage < 0.01 mg/kg and carrot root < 0.01 mg/kg. The levels were all too low for identification of the residue.

In an unconfined rotational crop study haloxyfop-methyl was applied to soya beans (0.28 kg ai/ha) and to cotton (0.56 kg ai/ha) as the first crops. Approximately 30 and 120 days after treatment, rotational crops of lettuce, sugar beets and wheat were sown into the plots and grown to maturity. Haloxyfop residues generally did not occur in the rotational crops at levels exceeding LOQs (0.01 and 0.02 mg/kg). Residues were detected in wheat green forage, but detection in a sample from the control plot suggested possible contamination.

Summary of haloxyfop in soil metabolism–haloxyfop esters are quickly hydrolysed and the acid becomes the major residue in the short term, but also disappears readily with typical half-lives of 9–21 days. Three soil metabolites were identified. Soil photolysis has little effect on haloxyfop residues compared with soil metabolism. Haloxyfop residues in soil should contribute very little to residue levels in root crops or rotational crops.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of haloxyfop in animal and plant matrices.

Analytical methods must take account of the nature of the residue as observed in metabolism studies–much of the residue occurs as polar and non-polar conjugates.

Haloxyfop residue methods rely on an initial extraction and hydrolysis step, usually with methanolic NaOH to release haloxyfop from conjugates. After solvent partition cleanup, the haloxyfop is methylated or butylated ready for GC analysis or further cleanup before the GC analysis. Typically, haloxyfop residues can be measured in most matrices to an LOQ of 0.01–0.05 mg/kg.

For various substrates, the extraction and hydrolysis step ranges from a simple methanolic NaOH extraction to a period of shaking homogenised sample with extractant (2 hours or overnight) to a more vigorous hydrolysis at elevated temperature for 2 hours.

The completeness of extraction of haloxyfop and its conjugates and of their conversion to parent acid was tested on soya bean samples available from the previous metabolism study. Overnight shaking of substrate with 0.1 M NaOH in 98% methanol + 2% water extracted 93% of the ^{14}C from the soya beans. HPLC produced a single peak matching haloxyfop which accounted for 95% of the ^{14}C in the extract.

The completeness of extraction of haloxyfop, esters and conjugates from goat milk was tested on a sample from a goat dosed with [^{14}C]haloxyfop-butyl. The method relied on an initial diethyl ether extraction from milk, followed by hydrolysis of the extracted residue in benzene-KOH-ethanol at 50 °C to release conjugates. A high percentage of the ^{14}C (91%) was extracted and released as haloxyfop acid by this procedure.

Little information is available on the completeness of extraction by briefer contact of the substrate with the alkaline extractant. Most of the validations have not included a check on this step. However, some validations have used a haloxyfop ester such as haloxyfop-ethoxyethyl as the spiked analyte, which does check that the extraction conditions quantitatively hydrolyse the spiked ester. Haloxyfop esters are readily hydrolysed, so the release of conjugates by the alkaline extractant with the conditions of the analytical methods would be generally expected.

None of the methods separates the haloxyfop enantiomers. The methods effectively measure 'total' haloxyfop present as acid, salts, esters and conjugates (esters with natural compounds).

Haloxyfop residues are not suitable for analysis by multi-residue methods because the extraction step is typically also a base-hydrolysis step designed to release haloxyfop from non-polar and polar conjugates found in animal and plant tissues. Such an extraction-hydrolysis step is not suitable for many other pesticides.

Stability of residues in stored analytical samples

The Meeting received information on the stability, during frozen storage, of residues in samples of green peas, cabbage, rice, soya beans and cotton seed. The analytical methods for haloxyfop measure haloxyfop present as acid, salts, esters and conjugates, so changes among these different forms during storage would not be detected.

Haloxyfop residues fortified in homogenized green peas and chopped cabbage were stable for 16 months (the test interval) storage at -16 °C.

Haloxyfop residues fortified in rice were stable in freezer storage for 7 months, the test interval.

Haloxyfop residues in soya beans matrix were stable for 17 months (the test interval) storage at –20 °C.

In another study, haloxyfop residues in soya beans were reported to be stable under frozen conditions for 43 months, the period of the test.

Haloxyfop residues fortified in cotton seed matrix were stable in freezer storage at –20 °C for 17 months, the test interval.

No data are available on the freezer storage stability of haloxyfop residues in animal commodities, but from haloxyfop stability in animal metabolism and during storage as residues in various plant matrices, no storage stability problems would be expected.

Definition of the residue

The current residue definition for haloxyfop is: Haloxyfop esters, haloxyfop and its conjugates expressed as haloxyfop.

The question of fat solubility requires careful consideration because some components of the residue are clearly fat-soluble, but unconjugated haloxyfop and its salts are not:

- Goat metabolism study: the ^{14}C residue concentrations (mg/kg) in fat were higher than in muscle: fat/muscle = 0.06/0.02 and 0.11/< 0.01.
- Hen metabolism study: the ^{14}C residue concentration (mg/kg) in fat was higher than in muscle: fat/muscle = 0.99/0.12. Also residue levels in egg yolks were much higher than in egg whites.
- Beef cattle feeding study: total haloxyfop residue concentrations (mg/kg) in fat were higher than in muscle: fat/muscle = 0.057/0.01 and 0.27/0.03.

- Dairy cattle feeding study: the total haloxyfop residue concentrations (mg/kg) in cream were higher than in milk: cream/milk = 0.12/0.01 and 0.29/0.034.
- Laying hen feeding study: the total haloxyfop residue concentrations (mg/kg) in fat were higher than in muscle: fat/muscle = 0.045/0.014 and 0.26/0.063.

The evidence is that the residue in animal commodities is fat-soluble.

The definition should also recognize the inclusion of haloxyfop-P.

The Meeting recommended a revised residue definition for haloxyfop.

Definition of the residue for plants and animals (for compliance with the MRL and for estimation of dietary intake): *sum of haloxyfop (including haloxyfop-P), its esters and its conjugates expressed as haloxyfop.*

The residue is fat-soluble.

Results of supervised trials on crops

The Meeting received information on the use patterns and labels for haloxyfop-P-methyl from many countries. On many of the labels, the application rates are given for the weeds to be controlled. It is not always absolutely clear which rates apply to which crops without knowing which are the likely weeds for each crop.

Application rates for a herbicide should be understood in a different way from application rates for an insecticide or fungicide because the target is different. For an insecticide or fungicide the aim is for a high percentage of the applied pesticide to reach the crop. Whereas for a herbicide, the target is the weed(s) to be controlled.

Particularly in the early growth stages of a crop, only a small percentage of applied herbicide is likely to reach the crop. For the same application rate, expressed in kg ai/ha, the amount of herbicides actually applied to the crop will depend on the crop growth stage and the degree of area coverage by the crop.

The Meeting received supervised trials data for the uses of haloxyfop-P-methyl, haloxyfop-methyl and haloxyfop-ethoxyethyl.

Current GAP relies on haloxyfop-P-methyl. Because the esters hydrolyse reasonably quickly when exposed to the environment, the behaviour of the residue should be little influenced by the nature of the ester and the Meeting decided to make use of residue data from other esters where application rates and timing were comparable to the GAP conditions.

Supervised trials were available on the following crops: oranges, grapefruit, lemons, apples, peaches, grapes, bananas, onions, field beans, peas, pigeon peas, beans, chickpeas, peas (pulses), sugar beet, rice, cotton, oilseed rape, peanuts, soya beans, sunflowers, coffee and alfalfa.

No residue data were available for potatoes. The meeting withdrew the previous haloxyfop maximum residue level recommendation of 0.1 mg/kg for potatoes.

For present purposes, haloxyfop or haloxyfop-P are considered as the active ingredient. Application rates and residue concentrations are expressed in terms of haloxyfop acid equivalent.

The NAFTA calculator was used as a tool in the estimation of the maximum residue level from the selected residue data set obtained from trials conducted according to GAP. As a first step, the Meeting reviewed all relevant factors related to each data set in arriving at a best estimate of the maximum residue level using expert judgement. Then, the NAFTA calculator was employed. If the statistical calculation spreadsheet suggested a different value from that recommended by the JMPR, a brief explanation of the deviation was provided. Some common factors that may lead to rejection of the statistical estimate include those situations where the number of data points is less than 15 or where there are too many values below LOQ.

Fruit and vine crops

Haloxyfop is used for weed control in orchards, vineyards and plantations. It is applied as a directed spray on the weeds, not on the trees or vines. In this situation, residues are not expected to occur in the fruit and this is confirmed by the residue trials. LOQs for haloxyfop in the trials from the 1980s until more recent times ranged from 0.01 mg/kg to 0.1 mg/kg, with many trials at 0.02 and 0.05 mg/kg.

Although different LOQs were used in the fruit and vine crop trials, the Meeting decided to use a consistent value for recommending MRLs for fruits where no residue is expected, i.e., 0.02 mg/kg.

Citrus fruits

Supervised trials on citrus were available from Australia, Brazil, Italy and New Zealand.

Haloxyfop-P-methyl is registered in Australia for weed control in orchards, vines and plantations at 0.42 kg ai/ha. In two Australian trials on lemons with directed applications of haloxyfop-ethoxyethyl (0.42 and 0.83 kg ai/ha, PHI 28 days), haloxyfop residues were below LOQ (0.05 mg/kg).

In Uruguay, haloxyfop-P-methyl is registered for control of weeds around fruit trees at an application rate of 0.15 kg ai/ha. In six trials in Brazil (compare with Uruguay GAP), haloxyfop-methyl was used as a directed spray around orange trees at 0.24, 0.48, 0.72, 0.96, 1.4 and 1.9 kg ai/ha and fruit were harvested 67 days after treatment. In another six trials with the same application rates, fruit were harvested 206 days after treatment. Haloxyfop residues were all below LOQ (0.1 mg/kg).

Haloxyfop-P-methyl is registered in New Zealand for weed control around citrus trees at 0.15 kg ai/ha. In two NZ trials on grapefruit with directed applications of haloxyfop-ethoxyethyl (0.21 and 0.42 kg ai/ha, PHI 29 days) and six trials on lemons also with haloxyfop-ethoxyethyl (0.21–0.83 kg ai/ha, PHI 28 days), haloxyfop residues were all below LOQ (0.05 mg/kg).

The Syrian label allows the use of haloxyfop-P-methyl for weed control in fruit trees and vines at 0.13 kg ai/ha. In two Italian trials (compare with Syrian GAP) on oranges with a directed application (0.16 kg ai/ha, PHI 56 days), haloxyfop residues were below LOQ (0.02 mg/kg).

Residues in fruits are not expected with this directed use on the weeds because haloxyfop breaks down reasonably quickly in soils and its residues are not readily taken up from the soil (evidence from the rotational crop studies).

The trials data, many at exaggerated rates, support that expectation that residues would be essentially zero.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg and STMR and HR values of 0 mg/kg for citrus fruits. The previous recommendation of 0.05(*) mg/kg is withdrawn.

Pome fruits

Supervised trials on apples were available from Australia, Italy, New Zealand and the USA.

Haloxyfop-P-methyl is registered in Australia for weed control in orchards, vines and plantations at 0.42 kg ai/ha. In two Australian trials on apples with directed applications of haloxyfop-ethoxyethyl (0.42 and 0.83 kg ai/ha, PHI 24 days), haloxyfop residues were below LOQ (0.05 mg/kg).

The Syrian label allows the use of haloxyfop-P-methyl for weed control in fruit trees and vines at 0.13 kg ai/ha. In six Italian trials (compare with Syrian GAP) on apples with directed applications of haloxyfop-ethoxyethyl (0.16 kg ai/ha, PHI 126–132 days), haloxyfop residues were below LOQ (0.02 mg/kg).

Haloxyfop-P-methyl is registered in New Zealand for weed control around pome fruit trees at 0.15 kg ai/ha. In two NZ trials on apples with directed applications of haloxyfop-ethoxyethyl (0.21 kg ai/ha, PHI 29 days), haloxyfop residues were below LOQ (0.01 mg/kg).

In eight US trials on apples with directed applications of haloxyfop-methyl (0.28 and 0.56 kg ai/ha, PHI 59–60 days), haloxyfop residues were below LOQ (0.05 mg/kg). No GAP is available to evaluate the US trials, but they provide supporting evidence that the directed use around fruit trees is essentially a zero residue situation.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg and STMR and HR values of 0 mg/kg for pome fruits. The previous recommendation of 0.05(*) mg/kg is withdrawn.

Stone fruits

Supervised trials on peaches were available from Australia.

Haloxyfop-P-methyl is registered in Australia for weed control in orchards, vines and plantations at 0.42 kg ai/ha. In two Australian trials on peaches with directed applications of haloxyfop-ethoxyethyl (0.42 and 0.83 kg ai/ha, PHI 24 days), haloxyfop residues were below LOQ (0.05 mg/kg).

Because of the nature of this use, i.e., the pesticide is not applied to the crop, and the expectation of a zero residue, the Meeting agreed to extrapolate from the results on citrus and pome fruits to stone fruits.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg and STMR and HR values of 0 mg/kg for stone fruits.

Grapes

Supervised trials on grapes were available from Australia, France and Italy.

Haloxyfop-P-methyl is registered in Australia for weed control in orchards, vines and plantations at 0.42 kg ai/ha. In six Australian trials on grapes with directed application of haloxyfop-ethoxyethyl (0.21, 0.42 and 0.83 kg ai/ha, PHI 21 and 29 days), haloxyfop residues were below LOQ (0.05 mg/kg).

The Swiss label allows the use of haloxyfop-P-methyl for weed control in grapevines at 0.16 kg ai/ha. In 11 French trials (compare with Swiss GAP) on grapes with directed applications of haloxyfop-ethoxyethyl (0.10, 0.21, 0.42, 0.83 and 1.7 kg ai/ha, PHI 86-115 days), haloxyfop residues were below LOQ (0.01 mg/kg).

The directed use in vineyards is directly comparable with the use in orchards with also the expectation of a zero residue.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg and STMR and HR values of 0 mg/kg for grapes. The previous recommendation of 0.05(*) mg/kg is withdrawn.

Bananas

Supervised trials on bananas were available from Australia.

Haloxyfop-P-methyl is registered in Australia for weed control in orchards, vines and plantations at 0.42 kg ai/ha. In two Australian trials on bananas with directed applications of haloxyfop-P-methyl and haloxyfop-ethoxyethyl (0.42 and 0.83 kg ai/ha respectively, PHI 14 days), haloxyfop residues were below LOQ (0.05 mg/kg).

Because of the nature of this use, i.e., the pesticide is not applied to the crop, and the expectation of a zero residue, the Meeting agreed to extrapolate from the results on orchards and vineyards to banana plantations.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg and STMR and HR values of 0 mg/kg for bananas. The previous recommendation of 0.05(*) mg/kg is withdrawn.

Onions

Supervised trials on onions were available from Belgium, France, Germany and New Zealand.

The Moldovan label allows the use of haloxyfop-P-methyl for weed control in onions at 0.10 kg ai/ha. In two Belgian trials matching Moldovan GAP on onions, haloxyfop residues in the onions (whole plant) were 0.06 and 0.12 mg/kg, 28 days after treatment. In four German trials matching Moldovan GAP on onions, haloxyfop residues in the onions were 0.02, 0.03, 0.04, and 0.09 mg/kg, 26–28 days after treatment.

The Tunisian label allows the use of haloxyfop-P-methyl for weed control in onions at 0.10 kg ai/ha. In two French trials matching Tunisian GAP on onions, haloxyfop residues in the onions (whole plant) were < 0.02 and 0.03 mg/kg 28 days after treatment.

Haloxyfop-P-methyl is registered in New Zealand for weed control in onions at 0.15 kg ai/ha, with harvest permitted 35 days later. The six trials on onions did not match GAP and could not be evaluated.

Plant metabolism studies have shown that haloxyfop is systemic and is quickly distributed throughout a treated plant. The European data on samples described as 'onions' and 'onions (whole plant)' may be combined.

Haloxyfop residues from the eight onion trials in rank order, median underlined were: < 0.02, 0.02, 0.03, <u>0.03</u>, <u>0.04</u>, 0.06, 0.09 and 0.12 mg/kg.

The Meeting estimated an STMR value of 0.035 mg/kg and a maximum residue level of 0.2 mg/kg for onions. The HR was 0.12 mg/kg.

The value derived from use of the NAFTA Calculator (after MLE[3]) was 0.24 mg/kg. The calculated value is in good agreement with the Meeting's estimate. However, the MRL calculation is sensitive to the lowest value.

Beans

Supervised trials on field beans were available from Belgium, France, Germany, Greece and Spain.

The Tunisian label allows the use of haloxyfop-P-methyl for weed control in field beans at 0.10 kg ai/ha. In eight French trials matching Tunisian GAP on field beans, haloxyfop residues in the beans (whole pods) were: < 0.02, < 0.02, 0.03, 0.06, 0.07, 0.10, 0.19 and 0.26 mg/kg, 25–29 days after treatment.

In a Greek trial and a Spanish trial with conditions also matching Tunisian GAP, haloxyfop residues in the beans (whole pods) were 0.18 and 0.22 mg/kg respectively 28 days after treatment.

No suitable GAP was available to evaluate the trials in Belgium and Germany.

In summary, haloxyfop residues in beans (whole pods) from the 10 trials in rank order (median underlined) were: < 0.02, < 0.02, 0.03, 0.06, <u>0.07</u>, <u>0.10</u>, 0.18, 0.19, 0.22 and 0.26 mg/kg.

The Meeting noted that the lowest residues (< 0.02 mg/kg) were associated with applications at growth stage BBCH 14 (fourth true leaf unfolded) and the highest residues (0.19, 0.22 and 0.26 mg/kg) were associated with applications at BBCH 59 (first petals visible) and BBCH 65 (full flowering).

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[3] <u>Note</u>: MLE (Maximum Likelihood Estimate) is the NAFTA process that adjusts the data below LOQ to a lognormal distribution, by applying the distribution based on values at or above the LOQ.

The growth stage timing for application clearly influences the residue level. Application at full flowering may occur while still observing the 28 days PHI. If all the trials were conducted with applications at BBCH 59–65, it is likely that most of the residues would be closer to the upper end of the distribution (0.19–0.26 mg/kg).

The Meeting estimated an STMR value of 0.085 mg/kg and a maximum residue level of 0.5 mg/kg for beans. The HR was 0.26 mg/kg.

The value derived from use of the NAFTA Calculator (after MLE) was 0.54 mg/kg. The calculated value is in good agreement with the Meeting's estimate. However, the lognormal plot extrapolation apparently diverges from the trend of the four highest residues. The calculated value is sensitive to the lowest value of the dataset.

Peas

Supervised trials on peas were available from Belgium, France, Italy and Spain.

The Tunisian label allows the use of haloxyfop-P-methyl for weed control in peas at 0.10 kg ai/ha. In eight French trials matching Tunisian GAP on peas, haloxyfop residues in the peas in pods were: 0.07, 0.08, 0.08, 0.14, 0.21, 0.32, 0.32 and 0.43 mg/kg, 22–60 days after treatment.

In three Italian trials matching Tunisian GAP on peas, haloxyfop residues in the peas in pods were: < 0.05, 0.05 and 0.07 mg/kg, 28–36 days after treatment.

In two Spanish trials matching Tunisian GAP on peas, haloxyfop residues in the peas in pods were: 0.12 and 0.53 mg/kg, 28 days after treatment.

The Belarus label allows the use of haloxyfop-P-methyl for weed control in peas at 0.10 kg ai/ha. In two Belgian trials matching Belarus GAP on peas, haloxyfop residues in the peas in pods were: 0.07 and 0.11 mg/kg, 31–34 days after treatment.

In summary, haloxyfop residues in peas in pods from the 15 trials in rank order (median underlined) were: < 0.05, 0.05, 0.07, 0.07, 0.07, 0.08, 0.08, 0.11, 0.12, 0.14, 0.21, 0.32, 0.32, 0.43 and 0.53 mg/kg.

All crops were treated between growth stages BBCH 50–51 (first flower buds visible) and BBCH 65 (full flowering), i.e., a limited growth stage range.

The Meeting estimated an STMR value of 0.11 mg/kg and a maximum residue level of 0.7 mg/kg for peas in pods. The latter replaces the previous recommendation (0.2 mg/kg). The HR was 0.53 mg/kg.

The value derived from use of the NAFTA Calculator was 0.9 mg/kg. The calculated value appears to be higher than necessary and is influenced by the lowest value in the dataset.

The Tunisian label allows the use of haloxyfop-P-methyl for weed control in peas at 0.10 kg ai/ha. In nine French trials matching Tunisian GAP on peas, haloxyfop residues in shelled peas were: < 0.01, < 0.05, 0.07, 0.07, 0.15, 0.26, 0.29, 0.32 and 0.44 mg/kg, 22–60 days after treatment.

In three Italian trials matching Tunisian GAP on peas, haloxyfop residues in shelled peas were: < 0.05, 0.05 and 0.05 mg/kg, 28–36 days after treatment.

In two Spanish trials matching Tunisian GAP on peas, haloxyfop residues in shelled peas were: 0.12 and 0.75 mg/kg, 28 days after treatment.

The Belarus label allows the use of haloxyfop-P-methyl for weed control in peas at 0.10 kg ai/ha. In two Belgian trials matching Belarus GAP on peas, haloxyfop residues in shelled peas were: 0.04 and 0.09 mg/kg, 31–34 days after treatment.

In summary, haloxyfop residues in shelled peas from the 16 trials in rank order (median underlined) were: < 0.01, 0.04, < 0.05, < 0.05, 0.05, 0.05, 0.07, 0.07, 0.09, 0.12, 0.15, 0.26, 0.29, 0.32, 0.44 and 0.75 mg/kg.

The Meeting estimated an STMR value of 0.08 mg/kg and a maximum residue level of 1 mg/kg for shelled peas. The HR was 0.75 mg/kg.

The value derived from use of the NAFTA Calculator (after MLE) was 1.8 mg/kg. This calculation appears to be higher than necessary and is influenced by the lowest value in the dataset. Different LOQs in the one dataset were probably not considered in the design of the NAFTA Calculator.

Pigeon peas

Supervised trials on pigeon peas were available from Australia, but no suitable GAP was available for evaluation.

Dry beans (pulses)

Supervised trials on beans (pulses) were available from Argentina, Brazil, Costa Rica and Germany.

No suitable GAPs were available for evaluating the data from Costa Rica and Germany.

In Argentina, haloxyfop-P-methyl may be used for weed control in beans at 0.15 kg ai/ha, with a PHI of 65 days. In seven trials in Argentina with an application matching GAP with a ± 25% tolerance (0.11–0.19 kg ai/ha and PHI range 50–80 days), haloxyfop residues in beans were: 0.21, 0.39, 0.86, 1.5, 1.5, 1.8 and 2.0 mg/kg.

In seven trials in Brazil with an application matching Argentinean GAP with a ± 25% tolerance (0.11–0.19 kg ai/ha and PHI range 50–80 days), haloxyfop residues in beans were: 0.01, 0.06, 0.07, 0.08, 0.08, 0.32 and 0.42 mg/kg.

In Brazil, haloxyfop-P-methyl may be used for weed control in beans at 0.048 kg ai/ha, with a PHI of 66 days. In 10 trials in Brazil with an application matching GAP with a ± 25% tolerance (0.036–0.060 kg ai/ha and PHI range 50–80 days), haloxyfop residues in beans were: < 0.01, 0.03, 0.03, 0.04, 0.04, 0.06, 0.06, 0.08, 0.23 and 0.49 mg/kg.

In seven trials in Argentina with an application matching Brazilian GAP with a ± 25% tolerance (0.036–0.060 kg ai/ha and PHI range 61–70 days), haloxyfop residues in beans were: 0.08, 0.26, 0.27, 0.41, 0.70, 0.80 and 1.2 mg/kg.

The data based on the Argentine GAP produced the higher residues and were selected for maximum residue estimation.

In summary, the residues from the 14 trials in line with Argentine GAP, in rank order, median underlined, were: 0.01, 0.06, 0.07, 0.08, 0.08, 0.21, <u>0.32</u>, <u>0.39</u>, 0.42, 0.86, 1.5, 1.5, 1.8 and 2.0 mg/kg.

The Meeting estimated an STMR value of 0.335 mg/kg and a maximum residue level of 3 mg/kg for beans (dry).

The previous recommendation of a group haloxyfop maximum residue level for pulses (0.2 mg/kg) is withdrawn. Insufficient data are available for a group maximum residue level. The group value is replaced by individual commodity recommendations where data are available.

The value derived from use of the NAFTA Calculator was 2.5 mg/kg. The calculated value is in good agreement with the Meeting's estimate.

Chickpeas

Supervised trials on chickpeas were available from Australia.

In Australia, haloxyfop-P-methyl may be used for weed control in chickpeas at 0.052 kg ai/ha from second leaf stage until prior to flowering.

In two trials in Australia with conditions in line with Australian GAP, haloxyfop residues in the chickpea grain were < 0.02 and 0.02 mg/kg. In two trials at double the GAP rate the residues were < 0.02 and 0.04 mg/kg.

The number of chickpea trials is very limited. However, the Australian use pattern for chickpeas is the same as for peas. In six trials matching GAP (see below), and four trials at 0.10 kg ai/ha, haloxyfop residues in peas (pulses) were < 0.01 mg/kg. The meeting used the pea data to support a chickpea maximum residue level.

The Meeting estimated an STMR value of 0.02 mg/kg and a maximum residue level of 0.05 mg/kg for chickpeas.

Peas (pulses)

Supervised trials on peas grown for dry pea production were available from Australia and France.

In Australia, haloxyfop-P-methyl may be used for weed control in peas at 0.052 kg ai/ha from second leaf stage until prior to flowering. In six trials in Australia matching GAP, haloxyfop residues in pea grain were: < 0.01 mg/kg (6). In four trials with the same timing but an application rate of 0.10 kg ai/ha, haloxyfop residues were also all below LOQ (0.01 mg/kg). In six trials with haloxyfop-ethoxyethyl at application rates of 0.10 and 0.21 kg ai/ha, but with the same timing, haloxyfop residues were also below LOQ (0.01 mg/kg).

The Tunisian label allows the use of haloxyfop-P-methyl for weed control in peas at 0.10 kg ai/ha. In eight French trials matching Tunisian GAP on peas, haloxyfop residues in the dry peas were: 0.02, 0.02, 0.04, < 0.05, 0.05, 0.06, 0.06 and 0.10 mg/kg. In nine French trials matching the Tunisian application rate (0.10 kg ai/ha), but using haloxyfop-ethoxyethyl, haloxyfop residues in dry peas were: < 0.02, < 0.02, < 0.02, 0.03, 0.04, 0.04, < 0.05, 0.05 and 0.07 mg/kg.

Residues from the Tunisian GAP were higher than those from Australian GAP and so were chosen for maximum residue evaluation.

In summary, the haloxyfop residues on dry peas from the Tunisian GAP (17 French trials) in rank order, median underlined, were: < 0.02, < 0.02, < 0.02, 0.02, 0.02, 0.03, 0.04, 0.04, <u>0.04</u>, < 0.05, < 0.05, 0.05, 0.05, 0.06, 0.06, 0.07 and 0.10 mg/kg.

The Meeting estimated an STMR value of 0.04 mg/kg and a maximum residue level of 0.2 mg/kg for peas (dry).

The value derived from use of the NAFTA Calculator (after MLE) was 0.17 mg/kg. The calculated value is in good agreement with the Meeting's estimate. The NAFTA Calculator is little influenced by the low values. However, the number of < LOQ values (5 in 17 trials, i.e., 29%) reduces the reliability of the calculated result. Different LOQs in the one dataset were probably not considered in the design of the NAFTA Calculator.

Soya beans

Supervised trials on soya beans were available from Argentina, Brazil, France, Germany, Hungary, Italy, Spain and the USA.

In Argentina, haloxyfop-P-methyl may be used for weed control in soya beans at 0.15 kg ai/ha. In two trials in Argentina with an application rate of 0.18 kg ai/ha (within 25% of 0.15 kg ai/ha), haloxyfop residues in soya beans were 0.03 and 0.11 mg/kg.

In 16 trials in Brazil with an application rate of 0.12 kg ai/ha (within 25% of the Argentinean GAP rate, 0.15 kg ai/ha), haloxyfop residues in soya beans were < 0.01 (4), 0.02, 0.02, 0.03, < 0.05, 0.06, 0.06, 0.08, 0.15, 0.19, 0.45, 0.90 and 1.8 mg/kg.

In Brazil, haloxyfop-P-methyl may be used for weed control in soya beans at 0.060 kg ai/ha with a PHI of 98 days. In five trials in Brazil in line with Brazilian GAP (accept tolerance on PHI of 90–110 days), haloxyfop residues in soya beans were < 0.01 (3), 0.01 and 0.06 mg/kg.

Haloxyfop-P-methyl is registered for use for weed control in soya beans in Moldova and Russian Federation at 0.10 kg ai/ha. No restraints on timing or crop growth stage are available.

In France, four trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Moldovan GAP) produced haloxyfop residues in soya beans of < 0.05 (2), 0.31 and 0.99 mg/kg.

In Germany, two trials with haloxyfop-P-methyl at 0.11 kg ai/ha (compare with Moldovan GAP) produced haloxyfop residues in soya beans of < 0.05 and 0.23 mg/kg.

In Hungary, two trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Moldovan GAP) produced haloxyfop residues in soya beans of < 0.05 and 0.11 mg/kg.

No suitable GAP was available to evaluate the US trials on soya beans.

Trials matching the conditions of Argentinean GAP produced the higher residues, so were used for maximum residue evaluation.

Summarising, 18 trials matching Argentinean GAP produced haloxyfop residues in soya beans (rank order, underlined median): < 0.01 (4), 0.02, 0.02, 0.03, 0.03, < <u>0.05</u>, <u>0.06</u>, 0.06, 0.08, 0.11, 0.15, 0.19, 0.45, 0.90 and 1.8 mg/kg.

The Meeting estimated an STMR value of 0.055 mg/kg and a maximum residue level of 2 mg/kg for soya beans.

The value derived from use of the NAFTA Calculator (after MLE) was 2.7 mg/kg. The number of < LOQ values (five in 18 trials, i.e., 28%) reduces the reliability of the calculated result. Different LOQs in the one dataset were probably not considered in the design of the NAFTA Calculator.

Sugar beet

Supervised trials on sugar beet were available from Belgium, France, Germany, Italy and Spain

Haloxyfop-P-methyl is registered for weed control in sugar beet in Belarus, Moldova, the Russian Federation and the Ukraine at 0.10 kg ai/ha.

In Belgium, a trial at 0.10 kg ai/ha of haloxyfop-P-methyl (compare with Belarus GAP) produced haloxyfop residues in sugar beet roots of 0.03 mg/kg.

In France, three trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Belarus GAP) produced haloxyfop residues in sugar beet roots of < 0.02, < 0.02 and < 0.02 mg/kg.

In France, three trials with haloxyfop-ethoxyethyl at 0.10 kg ai/ha (compare with Belarus GAP) produced haloxyfop residues in sugar beet roots of < 0.02, < 0.02 and < 0.02 mg/kg.

In Germany, five trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Belarus GAP) produced haloxyfop residues in sugar beet roots of 0.02, 0.04, 0.09, 0.11 and 0.30 mg/kg.

In summary, haloxyfop residues in sugar beet roots from 12 trials matching Belarus, Moldovan, Russian Federation and Ukrainian GAP were, in rank order, median underlined: < 0.02 (6), <u>0.02</u>, 0.03, 0.04, 0.09, 0.11 and 0.30 mg/kg.

The Meeting estimated an STMR value of 0.02 mg/kg, an HR value of 0.30 mg/kg and a maximum residue level of 0.4 mg/kg for sugar beet. The latter replaces the previous recommendation (0.3 mg/kg).

The value derived from use of the NAFTA Calculator (after MLE) was 0.11 mg/kg. The number of < LOQ values (6 in 12 trials, i.e., 50%) reduces the reliability of the calculated result.

Rice

Supervised trials with haloxyfop-methyl on rice were available from the USA.

No suitable GAP was available, so the trials could not be evaluated for estimation of a maximum residue level.

The Meeting withdrew its recommendations for polished rice of 0.02(*) mg/kg, husked rice of 0.02(*) mg/kg) and unprocessed rice bran of 0.02(*) mg/kg.

Cotton seed

Supervised trials on cotton, generating haloxyfop residue data on cotton seed, were available from Brazil, Greece, Spain and the USA.

In Brazil, haloxyfop-P-methyl may be used for weed control in cotton at 0.060 kg ai/ha, with a PHI of 123 days. In three trials in Brazil with an application matching GAP, haloxyfop residues in cotton seed were: < 0.01, < 0.01 and 0.08 mg/kg.

In Argentina, haloxyfop-P-methyl may be used for weed control in cotton at 0.15 kg ai/ha. In five trials in Brazil with an application of haloxyfop-P-methyl matching Argentinean GAP (± 25%), haloxyfop residues in cotton seed were: < 0.01, 0.02, 0.03, 0.09 and 0.52 mg/kg. In four trials in Brazil with an application of haloxyfop-methyl at 0.12 kg ai/ha, haloxyfop residues in cotton seed were < 0.1 (3) and 0.15 mg/kg.

No suitable GAP was available for evaluating cotton trials in Greece, Spain and the USA.

The Brazilian trials in line with Argentinean GAP were used for the maximum residue level estimation.

In summary, haloxyfop residues in cotton seed from the nine residue trials matching Argentinean GAP, in rank order, median underlined were: < 0.01, 0.02, 0.03, 0.09, < <u>0.1</u> (3), 0.15 and 0.52 mg/kg.

The Meeting estimated an STMR value of 0.1 mg/kg and a maximum residue level of 0.7 mg/kg for cotton seed. The latter replaces the previous recommendation (0.2 mg/kg).

The value derived from use of the NAFTA Calculator (after MLE) was 0.20 mg/kg. The lognormal plot extrapolation apparently diverges from the trend of the five highest residues. The number of < LOQ values (four in nine trials, i.e., 44%) reduces the reliability of the calculated result. Different LOQs in the one dataset were probably not considered in the design of the NAFTA Calculator.

Oilseed rape (canola)

Supervised trials on oilseed rape were available from Australia, France, Germany, Greece, Italy, Poland and Spain.

The Australian label allows application of haloxyfop-P-methyl for weed control in canola at 0.052 kg ai/ha at growth stages from second leaf to prior to bud formation and stem elongation. In two trials in Australia matching GAP, haloxyfop residues in canola grain were: 0.22 and 0.86 mg/kg.

Haloxyfop-P-methyl is registered for weed control in oilseed rape in Belarus, Moldova, Russian Federation and Ukraine at 0.10 kg ai/ha. No restraints on timing or crop growth stage are available, so all the European trials that have an application rate of 0.10 kg ai/ha (± 25%) are included.

In France, eight trials with haloxyfop-P-methyl at 0.10 kg ai/ha produced haloxyfop residues in rapeseed of < 0.01 (2), < 0.05 (3), 1.1, 1.5 and 1.9 mg/kg.

In France, three trials with haloxyfop-ethoxyethyl at 0.10 kg ai/ha produced haloxyfop residues in rapeseed of < 0.05 mg/kg (3).

In Germany, eight trials with haloxyfop-P-methyl at 0.10 kg ai/ha produced haloxyfop residues in rapeseed of < 0.01, < 0.05, 0.07, 0.10, 0.11, 0.37, 0.43 and 0.57 mg/kg.

In Poland, three trials with haloxyfop-P-methyl at 0.10 kg ai/ha produced haloxyfop residues in rapeseed of 0.33, 0.42 and 0.62 mg/kg.

Summarising, 22 European trials with haloxyfop-P-methyl at 0.10 kg ai/ha produced haloxyfop residues in rapeseed (rank order, underlined median): < 0.01 (3), < 0.05 (7), <u>0.07</u>, 0.10, 0.11, 0.33, 0.37, 0.42, 0.43, 0.57, 0.62, 1.1, 1.5 and 1.9 mg/kg.

The Meeting estimated an STMR value of 0.07 mg/kg and a maximum residue level of 3 mg/kg for rape seed. The latter replaces the previous recommendation (2 mg/kg).

The value derived from use of the NAFTA Calculator (after MLE) was 5.9 mg/kg. The number of < LOQ values (10 in 22 trials, i.e., 45%) reduces the reliability of the calculated result. Different LOQs in the one dataset were probably not considered in the design of the NAFTA Calculator.

Peanuts

Supervised trials on peanuts were available from Argentina and Australia.

In Argentina, haloxyfop-P-methyl may be used for weed control in peanuts at 0.15 kg ai/ha. The application rates in the trials were 0.045 and 0.090 kg ai/ha, so Argentine GAP could not be used for evaluation of the trials.

The Australian label allows application of haloxyfop-P-methyl for weed control in peanuts at 0.078 kg ai/ha at crop growth stages from second leaf to pegging. In four trials in Australia matching GAP, haloxyfop residues in peanuts were: < 0.02, < 0.02, 0.02 and 0.02 mg/kg.

The number of trials was too few to support a recommendation.

The Meeting agreed to withdraw its previous recommendations for peanuts (0.05 mg/kg).

Sunflowers

Supervised trials on sunflowers were available from Argentina, France, Germany, Greece and Spain.

In Argentina, haloxyfop-P-methyl may be used for weed control in sunflowers at 0.15 kg ai/ha. In one trial at 0.18 kg ai/ha, haloxyfop residues in sunflower seed were 0.14 mg/kg.

The Tunisian label allows the use of haloxyfop-P-methyl for weed control in sunflowers at 0.10 kg ai/ha. In five French trials matching Tunisian GAP on sunflowers, haloxyfop residues in the sunflower seed were: < 0.05 (2), 0.06, 0.07 and 0.10 mg/kg.

In three French trials with haloxyfop-ethoxyethyl, but matching the Tunisian GAP application rate on sunflowers, haloxyfop residues in the sunflower seed were: < 0.05 (2) and 0.05 mg/kg.

Summary of European sunflower seed data from eight trials matching Tunisian GAP: < <u>0.05</u> (4), <u>0.05</u>, 0.06, 0.07 and 0.10 mg/kg.

The Serbian label allows the use of haloxyfop-P-methyl for weed control in sunflowers at 0.16 kg ai/ha. In three French trials matching Serbian GAP (0.16 ± 25%, 0.12–0.20 kg ai/ha) (all 3 done at 0.15 kg ai/ha) on sunflowers, haloxyfop residues in the sunflower seed were: < 0.05, 0.05 and 0.14 mg/kg.

In three French trials with haloxyfop-ethoxyethyl, but matching Serbian GAP (0.16 ± 25%, 0.12–0.20 kg ai/ha) (all 3 done at 0.20 kg ai/ha) on sunflowers, haloxyfop residues in the sunflower seed were: 0.07, 0.09 and 0.16 mg/kg.

In two Greek trials matching Serbian GAP on sunflowers, haloxyfop residues in the sunflower seed were: < 0.05 mg/kg (2).

In three Spanish trials matching Serbian GAP on sunflowers, haloxyfop residues in the sunflower seed were: < 0.05 (2) and 0.17 mg/kg.

Summary of European sunflower seed data from 11 trials matching Serbian GAP: < 0.05 (5), 0.05, 0.07, 0.09, 0.14, 0.16 and 0.17 mg/kg.

The Meeting relied on the data from the higher application rate, i.e., the second set, for estimating the maximum residue level.

The Meeting estimated an STMR value of 0.05 mg/kg and a maximum residue level of 0.3 mg/kg for sunflower seed. The latter replaces the previous recommendation (0.2 mg/kg).

The value derived from use of the NAFTA Calculator (after MLE) was 0.31 mg/kg. The calculated MRL is in good agreement with the Meeting's estimate. The MLE process converted the distribution from non-lognormal to one where the lognormal presumption was not rejected. The number of < LOQ values (5 in 11 trials, i.e., 44%) reduces the reliability of the calculated result.

Coffee

Supervised trials on coffee were available from Brazil and Colombia.

In Colombia, haloxyfop-P-methyl is allowed as a directed application for control of weeds in coffee at a maximum rate of 0.36 kg ai/ha.

In two trials on coffee in Colombia with directed applications of haloxyfop-methyl at 0.18 and 0.36 kg ai/ha, haloxyfop residues in coffee beans did not exceed the LOQ (0.02 mg/kg).

In 13 trials on coffee in Brazil with directed applications of haloxyfop-methyl at 0.12 to 0.96 kg ai/ha, haloxyfop residues in coffee beans did not exceed the LOQ (0.02 mg/kg).

Residues in coffee beans are not expected from such a use where the trees are not sprayed. The trials data, some at exaggerated rates, support that expectation that residues would be essentially zero.

The Meeting estimated an STMR value of 0 mg/kg and a maximum residue level of 0.02(*) mg/kg for coffee beans. The HR was 0 mg/kg.

Legume animal feeds—alfalfa

Supervised trials on alfalfa were available from Australia, France, Germany and Poland.

In Australia, haloxyfop-P-methyl is registered for weed control uses on alfalfa at 0.078 kg ai/ha. The label allows use from the second trifoliate leaf onwards and imposes a 28 days interval between application and grazing or cutting for livestock.

In five Australian trials matching GAP (0.078 ± 25%, 0.059–0.10 kg ai/ha, PHI 28–32 days) on alfalfa, haloxyfop residues in the alfalfa forage (fresh weight) were: 0.10, 0.76, 1.0, 1.9 and 3.1 mg/kg.

In two Australian trials with haloxyfop-ethoxyethyl matching the GAP application rate and PHI (0.078 ± 25%, 0.059–0.10 kg ai/ha, PHI 28–32 days) on alfalfa, haloxyfop residues in the alfalfa forage (fresh weight) were: 1.1 and 1.9 mg/kg.

No suitable GAP was available to evaluate the alfalfa trials from France, Germany and Poland.

In summary, haloxyfop residues in alfalfa forage, fresh weight, from the seven Australian trials in rank order, median underlined, were: 0.10, 0.76, 1.0, 1.1, 1.9, 1.9 and 3.1 mg/kg.

The Meeting estimated STMR and high residue values for alfalfa forage (fresh weight) of 1.1 and 3.1 mg/kg, respectively.

The previous maximum residue level recommendation (5 mg/kg) for alfalfa forage is withdrawn because the policy is now to use information on forage in dietary burden calculations, but not to propose maximum residue levels for fresh forage commodities, which are understood not to be traded internationally.

Legume animal feeds—chickpea forage and straw

Supervised trials on chickpeas were available from Australia with data on forage and straw.

In Australia, haloxyfop-P-methyl may be applied for weed control in chickpeas at 0.052 kg ai/ha from second leaf stage until prior to flowering. The label imposes a 28 days interval between application and grazing or cutting for livestock.

In two trials in Australia with conditions in line with Australian GAP, haloxyfop residues in the chickpea forage (dry weight) were 2.9 and 4.3 mg/kg. In two trials at double the GAP rate the residues were 6.7 and 10.2 mg/kg.

Haloxyfop residues in chickpea straw (dry weight) from the four Australian trials were 0.13 and < 0.05 mg/kg for the label rate and 0.28 and < 0.05 mg/kg for the double rate.

The data were insufficient to support a recommendation.

Legume animal feeds—peanut forage and fodder

Supervised trials on peanuts were available from Australia with data on forage and fodder.

The Australian label allows application of haloxyfop-P-methyl for weed control in peanuts at 0.078 kg ai/ha at crop growth stages from second leaf to pegging. The label imposes a 28 days interval between application and grazing or cutting for livestock.

In four trials in Australia matching GAP, haloxyfop residues in peanut forage, dry weight, were: < 0.02, 0.13, 0.28 and 1.1 mg/kg. Haloxyfop residues in peanut straw (dry weight) from the same four Australian trials were: 0.42, 1.2, 2.9 and 3.0 mg/kg. Peanut forage data are not currently used in dietary burden calculations.

In four trials in Australia at 0.16 kg ai/ha (double the GAP application rate) but matching GAP for timing of application, haloxyfop residues in peanut straw (dry weight) were: 1.1, 1.9, 3.8 and 5.4 mg/kg, i.e., double the application rate produced approximately double the residue level. The data from the double rate trials provide support for the GAP trials.

The Meeting estimated an STMR of 2.1 mg/kg and a maximum residue level of 5 mg/kg for peanut fodder. The high residue was 3.0 mg/kg.

Legume animal feeds—soya bean forage

Supervised trials on soya beans were available from France, Germany, Hungary, Italy and Spain with data on forage.

Haloxyfop-P-methyl is registered for use for weed control in soya beans in Moldova and the Russian Federation at 0.10 kg ai/ha. No restraints on timing or crop growth stage are available.

In France, four trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Moldovan GAP) produced haloxyfop residues in soya bean plants, i.e., forage, of < 0.05 (2), 0.12 and 0.13 mg/kg.

In Germany, two trials with haloxyfop-P-methyl at 0.11 kg ai/ha (compare with Moldovan GAP) produced haloxyfop residues in soya bean plants, i.e., forage, of < 0.05 and 0.10 mg/kg.

In Hungary, two trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Moldovan GAP) produced haloxyfop residues in soya bean plants, i.e., forage, of < 0.05 and 0.18 mg/kg.

Summarising soya bean forage data—eight trials from Europe matching Moldova and Russian Federation GAP produced haloxyfop residues in soya bean forage (rank order, underlined median): < <u>0.05</u> (4), <u>0.10</u>, 0.12, 0.13 and 0.18 mg/kg.

The Meeting estimated STMR and high residue values for soya bean forage (fresh weight) of 0.075 and 0.18 mg/kg, respectively.

Sugar beet leaves or tops

Supervised trials on sugar beets were available from Germany, Italy and Spain with data on leaves and tops.

Haloxyfop-P-methyl is registered for weed control in sugar beet in Belarus, Moldova, the Russian Federation and the Ukraine at 0.10 kg ai/ha.

In Belgium, a trial at 0.10 kg ai/ha of haloxyfop-P-methyl (compare with Belarus GAP) produced haloxyfop residues in sugar beet tops of 0.07 mg/kg.

In Germany, five trials with haloxyfop-P-methyl at 0.10 kg ai/ha (compare with Belarus GAP) produced haloxyfop residues in sugar beet leaves of 0.10, 0.17 and 0.38 mg/kg and residues of 0.08 and 0.12 mg/kg in beet tops.

In summary, haloxyfop residues in sugar beet leaves or tops from six trials matching Belarus, Moldovan, Russian Federation and Ukrainian GAP were, in rank order median underlined: 0.07, 0.08, <u>0.10</u>, <u>0.12</u>, 0.17 and 0.38 mg/kg.

The Meeting estimated STMR and HR values of 0.11 and 0.38 mg/kg for sugar beet leaves or tops.

The previous maximum residue level recommendations (0.3 mg/kg) for sugar beet leaves or tops and fodder beet leaves or tops are withdrawn because the policy is now to use information on forage in dietary burden calculations, but not to propose maximum residue levels for fresh forage commodities, which are understood not to be traded internationally.

Fodder beet

Haloxyfop-P-methyl is registered for weed control in beets in Iraq at 0.12 kg ai/ha. Therefore, the data on sugar beet at 0.10 kg ai/ha can be used to support a fodder beet recommendation.

The Meeting extrapolated the estimate for sugar beet to fodder beet: an STMR value of 0.02 mg/kg, an HR value of 0.30 mg/kg and a maximum residue level of 0.4 mg/kg for fodder beet. The latter replaces the previous recommendation (0.3 mg/kg).

Rapeseed forage

Supervised trials on oilseed rape were available from Australia, France, Germany, Greece, Italy, Poland and Spain with data on forage.

The Australian label allows application of haloxyfop-P-methyl for weed control in canola (oilseed rape) at 0.052 kg ai/ha at growth stages from second leaf to prior to bud formation and stem elongation. The label imposes a 28 days interval between application and grazing or cutting for livestock.

In three trials in Australia matching GAP, haloxyfop residues in canola forage, expressed on dry weight, were: 0.32, 1.3 and 5.0 mg/kg.

In two trials in Australia matching GAP, haloxyfop residues in canola fodder were: 0.06 and 0.22 mg/kg.

The Meeting estimated STMR and high residue values for oilseed rape forage (dry weight) of 1.3 and 5.0 mg/kg, respectively for Australian uses.

Haloxyfop-P-methyl is registered for weed control in oilseed rape in Belarus, Moldova, the Russian Federation and the Ukraine at 0.10 kg ai/ha. No restraints on timing or crop growth stage are available, so all the European trials that have an application rate of 0.10 kg ai/ha (± 25%) could be included.

However, residues in forage decline quickly and some time limits are needed to produce a residue population suitable for STMR estimation. In practice, forage could be grazed or cut immediately after treatment. The Meeting decided to use forage data from samples taken on the same day as the treatment or 1 day later.

In three trials in France with haloxyfop-P-methyl application at 0.10 kg ai/ha (± 25%), haloxyfop residues in oilseed rape plants harvested on the day of application were: 1.5, 3.1 and 5.4 mg/kg.

In six trials in Germany with haloxyfop-P-methyl application at 0.10 kg ai/ha (± 25%), haloxyfop residues in oilseed rape plants harvested on the day of application or one day later were: 1.6, 3.9, 4.3, 5.6, 5.7 and 6.8 mg/kg.

In two trials in Poland with haloxyfop-P-methyl application at 0.10 kg ai/ha (± 25%), haloxyfop residues in oilseed rape plants harvested on the day of application were: 2.2 and 3.4 mg/kg.

In summary, 11 trials from Europe with the application rate 0.10 kg ai/ha (GAP of Belarus, Moldova, the Russian Federation and the Ukraine) produced haloxyfop residues in oilseed rape plant 0 or 1 day after treatment (rank order, median underlined): 1.5, 1.6, 2.2, 3.1, 3.4, <u>3.9</u>, 4.3, 5.4, 5.6, 5.7 and 6.8 mg/kg

The Meeting estimated STMR and high residue values for oilseed rape forage (fresh weight) of 3.9 and 6.8 mg/kg, respectively for European uses.

Fate of residues during processing

The Meeting received information on the fate of haloxyfop residues during the processing of oilseed rape for oil and meal, soya beans for oil and meal and sugar beet for sugar.

No information was available on the fate of haloxyfop residues during the processing of cotton seed. The Meeting withdrew the previous recommendation of 0.5 mg/kg for a haloxyfop maximum residue level in crude cotton seed oil.

A processing study was also received for <u>apples</u>, but haloxyfop uses as a directed spray on weeds around apple trees did not produce detectable residues in the apples or processed commodities. No processing factors could be calculated.

In a series of trials in France, haloxyfop-ethoxyethyl was applied to <u>oilseed rape</u> at 0.10, 0.21 and 0.63 kg ai/ha at one of two growth stages, 5–6 leaves and beginning of flowering. The harvested rapeseed was processed at laboratory scale to crude oil, refined and deodorized oil and meal. Haloxyfop residues in the rapeseed were below LOQ (0.05 mg/kg) for low application rates and early growth-stage treatments and were not included in the processing factor calculations.

The laboratory process was designed to simulate the commercial process. Rapeseed was coarsely ground and extracted with hot hexane. The extracted solid material was the meal. Crude oil was degummed, alkali was added and the soap was allowed to settle. The oil was decanted and filtered and then bleached with a Fuller's earth treatment and deodorized by steam distillation at 240 ºC under reduced pressure.

The processing factors for haloxyfop residues for rapeseed → crude oil were: 1.1, 1.2, 1.4, 1.7, 1.8 and 2.0—median 1.6.

The processing factors for haloxyfop residues for rapeseed → refined and deodorized oil were: 0.93, 1.1, 1.3, 1.7, 1.9 and 2.2—median 1.5.

The processing factors for haloxyfop residues for rapeseed → meal were: 0.73, 0.88, 0.89, 0.92, 0.93 and 1.7—median 0.91.

The processing factors for crude rape seed oil (1.6), refined rape seed oil (1.5) and meal (0.91) were applied to the estimated STMR for rape seed (0.07 mg/kg) to produce STMR-P values for crude rape seed oil (0.17 mg/kg), refined rape seed oil (0.16 mg/kg) and rapeseed meal (0.10 mg/kg). These concentrations fall below the estimated maximum residue level for rape seed (3 mg/kg), so maximum residue levels for the oils and meal are not needed.

The maximum residue level recommendations for crude rape seed oil (5 mg/kg) and refined rape seed oil (5 mg/kg) are withdrawn.

In soya bean trials in the USA, haloxyfop-methyl was applied at 0.28 kg ai/ha to soya beans in bloom or haloxyfop-P-methyl at 0.70 kg ai/ha was applied to soya beans at the 5th trifoliate leaf stage. The soya beans were processed in a laboratory-scale system to produce hulls, meal, crude oil, refined oil and soapstock and haloxyfop residue levels were measured on the products.

The processing factors for haloxyfop residues for soya beans → crude oil were: 0.40, 0.79 and 1.3—median 0.79.

The processing factors for haloxyfop residues for soya beans → refined oil were: 0.33, 0.75 and 1.2—median 0.75.

The processing factors for haloxyfop residues for soya beans → meal were: 1.19, 1.25 and 1.29—median 1.25.

The processing factors for crude soya bean oil (0.79), refined soya bean oil (0.75) and soya bean meal (1.25) were applied to the estimated STMR for soya beans (0.055 mg/kg) to produce STMR-P values for crude soya bean oil (0.044 mg/kg), refined soya bean oil (0.041 mg/kg) and soya bean meal (0.069 mg/kg). These concentrations fall below the estimated maximum residue level for soya beans (2 mg/kg), so maximum residue levels for the oils and meal are not needed.

The maximum residue level recommendations for crude soya bean oil (0.2 mg/kg) and refined soya bean oil (0.2 mg/kg) are withdrawn.

In UK trials, sugar beet were treated with haloxyfop-ethoxyethyl at 0.25 or 0.50 kg ai/ha at the 6–8 leaf growth stage. After harvest, beets were processed to juice, pressed pulp, refined sugar and green syrup. The process was pilot scale and consisted of washing, slicing, water extraction, pressing, filtration, calcium carbonate precipitation-filtration, boiling and centrifuging.

Haloxyfop residue levels in the refined sugar did not exceed the analytical method LOQ (0.01 mg/kg). Processing factors were calculated for the refined sugar, the green syrup and the pressed pulp. Green syrup is the liquor from the second last crystallizer, comparable with molasses, the liquor from the final crystallizer.

The processing factors for haloxyfop residues for sugar beet → refined sugar were: < 0.09 and 0.15—best estimate < 0.09.

The processing factors for haloxyfop residues for sugar beet → green syrup were: 2.95 and 3.31—mean 3.1.

The processing factors for haloxyfop residues for sugar beet → pressed pulp were: 0.36 and 0.46—mean 0.41.

The processing factor for refined sugar (< 0.09) was applied to the estimated STMR for sugar beet (0.02 mg/kg) to produce an STMR-P value for refined sugar (0.002 mg/kg). This concentration falls below the estimated maximum residue level for sugar beet (0.4 mg/kg), so a maximum residue level for haloxyfop residues in raw sugar is not needed.

The processing factor for green syrup (3.1) was applied to the estimated STMR for sugar beet (0.02 mg/kg) to produce an STMR-P value for green syrup (0.063 mg/kg).

The processing factor for pressed pulp (0.41) was applied to the estimated STMR for sugar beet (0.02 mg/kg) to produce an STMR-P value for pressed pulp (0.008 mg/kg).

Residues in animal commodities

The meeting received beef cattle feeding studies with haloxyfop and haloxyfop-P, dairy cattle studies with haloxyfop and haloxyfop-P and a laying hen study with haloxyfop. These livestock feeding studies provided information on likely haloxyfop residues resulting in bovine tissues and milk and poultry tissues and eggs from haloxyfop residues in the livestock diets.

Beef calves were dosed with haloxyfop via gelatin capsule at rates equivalent to 0.25, 0.5, 1, 5 and 10 ppm in the dry-weight diet for 28 consecutive days. Animals were slaughtered 18 to 21 hours after the final dose for tissue collection. Additional groups of animals at the highest dose were kept for 7 and 14 days after the final dose to observe declines in residue levels.

Mean haloxyfop residues in the muscle from the fivr dose rates (equivalent to 0.25, 0.5, 1, 5 and 10 ppm of dry weight diet) were: < 0.01, < 0.01, < 0.01, 0.01, and 0.03 mg/kg, respectively. Similarly for liver: 0.02, 0.02, 0.05, 0.13 and 0.54 mg/kg, respectively; kidney: 0.06, 0.07, 0.14, 0.39 and 1.3 mg/kg, respectively; and fat: 0.02, 0.01, 0.01, 0.057, and 0.27 mg/kg, respectively.

After 7 and 14 days on a residue-free diet, residues had declined, but residue levels were widely variable between animals.

For animals dosed at 5 and 10 ppm, residues in muscle equalled or exceeded LOQ and ratios between residue levels in fat and muscle were calculated: mean = 7.8, range 3.6–18.5, n = 5, suggesting a fat-soluble residue.

Beef cattle were dosed with haloxyfop-P via gelatin capsule at rates equivalent 10, 20 and 30 ppm in the dry-weight diet for 28 consecutive days and were slaughtered on day 28 for tissue collection. Additional animals at the highest dose were kept for 7, 14, 21 and 28 days after the final dose to observe declines in residue levels. The analytical method did not include a hydrolysis step, so the residue data for fat were unlikely to include haloxyfop triacylglyceride conjugates and could not be used.

Mean haloxyfop residues in the muscle from the three dose rates (equivalent to 10, 20 and 30 ppm of dry weight diet) were: 0.03, 0.05 and 0.04 mg/kg, respectively. Similarly for liver: 0.25, 0.38 and 0.28 mg/kg, respectively; and kidney: 0.58, 1.0 and 1.2 mg/kg, respectively.

After 7 days on a residue-free diet, haloxyfop residues in muscle had fallen below LOQ (0.01 mg/kg) while residues in liver and kidney had fallen by approximately 70% and 90% respectively. Residues continued to decline during the next 21 days, but at a slower rate.

Holstein dairy cows were dosed through the feed with haloxyfop at nominal concentrations of 0.25, 0.75 and 2.5 ppm in the dry-weight diet for 28 consecutive days. Milk was collected twice daily. Milk from morning milking was put through a separator to produce cream.

Haloxyfop residues in milk from the low-dose (0.25 ppm) cows did not exceed the LOQ (0.01 mg/kg) except for one case (0.01 mg/kg). Residues in the milk from the middle dose group (0.75 ppm) were in the range < 0.01 to 0.026 mg/kg from days 5 to 28. Residues in the milk from the high dose group (2.5 ppm) were in the range < 0.01 to 0.055 mg/kg (mean 0.033 mg/kg) from days 5 to 28, the approximate plateau of residue levels.

The range of haloxyfop residue levels in cream from days 10 and 17 were 0.043–0.051 mg/kg for the low dose (0.25 ppm), 0.11–0.22 mg/kg for the middle dose group (0.75 ppm) and 0.28–0.42 mg/kg the high dose group (2.5 ppm).

Residue data were available for cream and milk on an individual animal basis for days 3 and 10. Average (and range) of haloxyfop residue levels were: cream 0.316 mg/kg (0.24–0.42 mg/kg) and milk 0.019 (0.01–0.11 mg/kg). The average for 'milk residues ÷ cream residues' was 0.059.

Friesian dairy cows were dosed with haloxyfop-P via gelatin capsule at rates equivalent to 10, 20 and 30 ppm in the dry-weight diet for 28 consecutive days. Milk was collected twice daily. Milk was analysed for haloxyfop by a method that does not include a hydrolysis step and therefore may not have recovered haloxyfop residues quantitatively from triacylglyceride conjugates. Residues appeared to plateau at or before 10 days. The average concentrations of haloxyfop measured in the milk from days 10 to 26 were 0.317, 0.558 and 0.804 mg/kg for dosing levels equivalent to 10, 20 and 30 ppm, respectively.

White Leghorn laying hens were dosed through the feed with haloxyfop at nominal concentrations of 0.25, 0.75, and 2.5 ppm in the diet, for 28 consecutive days. Eggs were collected twice daily. Birds were slaughtered approximately 24 hours after the final dose for tissue collection.

Additional groups of birds at the highest dose were kept for 7 and 14 days after the final dose to observe declines in residue levels.

Mean haloxyfop residues in the muscle + skin from the three dose rates (equivalent to 0.25, 0.75 and 2.5 ppm of dry weight diet) were: < 0.01, 0.014 and 0.063 mg/kg, respectively. Similarly for liver: 0.033, 0.12 and 0.36 mg/kg, respectively; fat: 0.013, 0.045 and 0.26 mg/kg, respectively; and eggs (day 4 to day 28, 11 sampling days): < 0.01, 0.014 and 0.036 mg/kg, respectively.

Residues depleted quickly in muscle and liver for birds placed on a haloxyfop residue-free diet, but were quite persistent in fat. Mean haloxyfop residues in the fat (dose rate equivalent to 2.5 ppm of the dry weight diet) were 0.26 mg/kg (day 28, final dose), 0.17 mg/kg (day 35, 7 days later) and 0.16 mg/kg (day 42, 14 days after the final dose).

Haloxyfop residue levels in fat were approximately 4–5 times as high as in the muscle for the 2.5 ppm dosing group on day 28 and an average 14 times on day 35 for cases where residues in muscle exceeded the LOQ (0.01 mg/kg).

Livestock dietary burden

The Meeting estimated the dietary burden of haloxyfop in livestock on the basis of the diets listed in Annex 6 of the 2006 JMPR Report (OECD Feedstuffs Derived from Field Crops). Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities.

Some processed and forage commodities do not appear in the Recommendations Table (because no maximum residue level is needed) but they are used in estimating livestock dietary burdens. Those commodities are listed here. Also, the terminology for commodities in the OECD feed tables is not always identical to descriptions in the original studies or Codex descriptions and some clarification is needed.

Commodity	STMR or STMR-P, mg/kg	High residue, mg/kg
Alfalfa forage = Alfalfa forage (Australia)	1.1	3.1
Fodder beet = Beet, mangel, fodder	see Recommendations Table	
Oilseed rape forage = Rape forage (Europe)	3.9	6.8
Oilseed rape forage = Rape forage (Australia)	1.3 dry wt	5.0 dry wt
Peanut fodder = Peanut hay	see Recommendations Table	
Rape seed meal = Canola meal	0.10	
Soya bean forage (green) = Soya bean forage (Europe)	0.075	0.18
Soya bean meal	0.069	
Sugar beet green syrup = Beet sugar, molasses	0.063	
Sugar beet leaves or tops = Beet, sugar tops (Europe)	0.11	0.38
Sugar beet pressed pulp = Beet, sugar, dried pulp	0.008	

Estimated maximum and mean dietary burdens of livestock

Tier 1

In a Tier 1 assessment, livestock from US-Canada, EU and Australia are assumed to be exposed to residues on all feed commodities irrespective of where they are produced.

Tier 1 dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6 of the 2009 JMPR Report. The calculations were made according to the livestock diets from US-Canada, EU and Australia in Appendix IX of the 2009 FAO Manual.

	Livestock dietary burden, haloxyfop, ppm of dry matter diet					
	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	9.91	4.55	8.87	3.59	22.7[a]	13.0[b]
Dairy cattle	8.16	3.94	6.53	2.70	14.4[c]	7.09[d]
Poultry-broiler	0.11	0.11	0.11	0.11	0.29	0.29
Poultry-layer	0.11	0.11	2.40[e]	1.41[f]	0.29	0.29

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

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

[c] Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk.

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

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

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

Tier 2

A Tier 2 refinement was considered because the estimated IEDI exceeded the ADI for some diets (see below).

In a Tier 2 assessment, livestock from US-Canada, EU and Australia are assumed to be exposed to residues on all feed commodities that are traded internationally. Fresh forages are not traded internationally, so the dietary burden from fresh forage arises only where the relevant GAP produces residues on that fresh forage.

For example, a registered haloxyfop use in Australia produces residues on fresh alfalfa forage. In a Tier 2 assessment, the residues on fresh alfalfa forage would add to the dietary burden of Australian livestock, but not to livestock in US-Canada and EU.

Tier 2 dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6 of the 2009 JMPR Report. The calculations were made according to the livestock diets from US-Canada, EU and Australia in Appendix IX of the 2009 FAO Manual.

	Livestock dietary burden, haloxyfop, ppm of dry matter diet					
	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	0.98	0.71	3.12	1.51	8.86[a]	3.14[b]
Dairy cattle	0.80	0.59	3.03	1.47	7.31[c]	2.41[d]
Poultry—broiler	0.11	0.11	0.11	0.11	0.29	0.29
Poultry—layer	0.11	0.11	2.40[e]	1.41[f]	0.29	0.29

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

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

c Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk.

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

e Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

f Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Animal commodities, maximum residue level estimation

Cattle

Tier 1

Residue levels in milk appeared to be critical for chronic dietary exposure.

The STMR for milk was calculated from the STMR dairy cow dietary burden (7.09 ppm) by interpolating between the 0 and the 10 ppm feeding levels of the Friesian dairy cow study.

The Meeting estimated an STMR value of 0.22 mg/kg for milks.

With milk STMR of 0.22 mg/kg, the IEDI for haloxyfop in the 13 diets was 60–190% of the ADI. In an IEDI calculation for milk only, the intake was estimated as 15–63 µg/person for the 13 diets, which exceeded the ADI (equivalent to 42 µg/person) in some diets.

The Meeting examined how the assessment may be refined in a Tier 2 assessment.

Tier 2

Fresh forages are not traded internationally, so the livestock dietary burdens were recalculated assuming that fresh forages (with locally generated residues) are consumed only by livestock where the relevant GAP produces residues on that fresh forage.

For MRL estimation, the high residues in the tissues were calculated by interpolating the maximum beef cattle dietary burden (8.86 ppm) between the relevant feeding levels (5 and 10 ppm) from the beef calf feeding study and using the highest tissue concentrations from individual animals within those feeding groups.

The STMR values for the tissues were calculated by interpolating the STMR beef cattle dietary burden (3.14 ppm) between the relevant feeding levels (1 and 5 ppm) from the haloxyfop beef calf feeding study and using the mean tissue concentrations from those feeding groups. For muscle, residues were below LOQ at the 1 ppm feeding level, so the STMR for muscle was calculated by taking the dietary burden (3.14 ppm) as a proportion of the 5 ppm feeding level.

For milk, the high residues were calculated from the maximum dairy cow dietary burden (7.31 ppm) as a proportion of the 10 ppm feeding level and using the mean milk residues from the Friesian dairy cow feeding study. The STMR for milk was calculated from the STMR dairy cow dietary burden (2.41 ppm) by interpolating between the 0.75 and 2.5 ppm feeding levels of the Holstein dairy cow study.

The Holstein dairy cow study provided some information on the relative concentrations of haloxyfop residues in milk and cream. The ratio between residue concentrations in milk and in cream was quite variable.

In the table, dietary burdens are shown in round brackets (), feeding levels and residue concentrations from the feeding study are shown in square brackets [] and estimated concentrations related to the dietary burdens are shown without brackets.

Dietary burden (ppm)					
Feeding level [ppm]	Milk	Muscle	Liver	Kidney	Fat
MRL					
	mean	highest	highest	highest	highest
MRL beef cattle (8.86 ppm) [5, 10 ppm]		0.041 mg/kg [0.01, 0.05]	0.53 mg/kg [0.14, 0.65]	1.42 mg/kg [0.46, 1.7]	0.33 mg/kg [0.068, 0.41]
MRL dairy cattle (7.31 ppm) [0, 10 ppm]	0.23 mg/kg [0, 0.317]				

Dietary burden (ppm)					
Feeding level [ppm]	Milk	Muscle	Liver	Kidney	Fat
	mean	mean	mean	mean	mean
STMR beef cattle (3.14 ppm) [0, 1, 5 ppm]		0.006 mg/kg [0, < 0.01, 0.01]	0.093 mg/kg [0, 0.05, 0.13]	0.27 mg/kg [0, 0.14, 0.39]	0.035 mg/kg [0, 0.01, 0.057]
STMR dairy cattle (2.41 ppm) [0.75, 2.5 ppm]	0.033 mg/kg [0.01, 0.034]				

The data from the cattle feeding studies were used to support the estimation of maximum residue levels for haloxyfop in mammalian meat, edible offal and milk based on the residues in liver and kidney.

The Meeting estimated an STMR value of 0.27 mg/kg and a maximum residue level of 2 mg/kg for mammalian edible offal, based on liver and kidney data. The HR was 1.42 mg/kg.

The Meeting estimated an STMR value of 0.033 mg/kg and a maximum residue level of 0.3 mg/kg for milks.

The average for 'milk residues ÷ cream residues' for the 2.5 ppm dosing group (day 10 data) was 0.076. The STMR and high residue for milk fat may be calculated from the values for milk (HR = 0.23 mg/kg, STMR = 0.033 mg/kg), the 'milk residues ÷ cream residues' factor and taking cream as 50% milk fat.

The Meeting estimated an STMR value of 0.87 mg/kg and a high residue level of 6.1 mg/kg for milk fat. The Meeting estimated a maximum residue level of 7 mg/kg for milk fat.

The Meeting estimated STMR values of 0.006 mg/kg for mammalian muscle and 0.035 mg/kg for mammalian fat, and a maximum residue level of 0.5 (fat) for mammalian meat. The HRs were 0.041 and 0.33 mg/kg for muscle and fat respectively.

Previous recommendations for cattle meat (0.05 mg/kg), cattle liver (0.5 mg/kg), cattle kidney (1 mg/kg) and cattle milk (0.3 mg/kg) are withdrawn.

Poultry

For MRL estimation, the high residues in the tissues and eggs were calculated by interpolating the maximum dietary burden (2.4 ppm) between the relevant feeding levels (0.75 and 2.5 ppm) from the haloxyfop laying hen feeding study and using the highest tissue concentrations of the group.

The STMR values for the poultry tissues and eggs were calculated by interpolating the STMR dietary burden (1.41 ppm) between the relevant feeding levels (0.75 and 2.5 ppm) from the haloxyfop laying hen feeding study and using the mean tissue and egg concentrations from those feeding groups.

In the table, dietary burdens are shown in round brackets (), feeding levels and residue concentrations from the feeding study are shown in square brackets [] and estimated concentrations related to the dietary burdens are shown without brackets.

Dietary burden (ppm)				
Feeding level [ppm]	Eggs	Muscle + skin	Liver	Fat
MRL	highest	highest	highest	highest
MRL broilers and layers (2.4 ppm) [0.75, 2.5 ppm]	0.050 mg/kg [0.02, 0.052 mg/kg]	0.105 mg/kg [0.02, 0.11 mg/kg]	0.61 mg/kg [0.19, 0.64 mg/kg]	0.52 mg/kg [0.11, 0.54 mg/kg]
STMR	mean	mean	mean	mean
STMR broilers and layers (1.41 ppm)	0.022 mg/kg [0.014, 0.036 mg/kg]	0.032 mg/kg [0.014, 0.063]	0.21 mg/kg [0.12, 0.36 mg/kg]	0.13 mg/kg [0.045, 0.26 mg/kg]

Dietary burden (ppm)				
Feeding level [ppm]	Eggs	Muscle + skin	Liver	Fat
[0.75, 2.5 ppm]				

The data from the laying hen feeding studies were used to support the estimation of maximum residue levels for haloxyfop in poultry tissues and eggs.

The Meeting estimated a maximum residue level for poultry meat (fat) of 0.7 mg/kg. The STMR values were: 0.13 mg/kg (fat) and 0.032 mg/kg (muscle). The recommendation for chicken meat (0.01(*) mg/kg) is withdrawn. The HR values were: 0.52 mg/kg (fat) and 0.11 mg/kg (muscle).

The Meeting estimated an STMR value of 0.21 mg/kg and a maximum residue level of 0.7 mg/kg for edible offal of poultry. The recommendation for edible offal of chicken (0.05 mg/kg) is withdrawn. The HR for poultry edible offal was 0.61 mg/kg.

The Meeting estimated an STMR value of 0.022 mg/kg and a maximum residue level of 0.1 mg/kg for eggs. The recommendation for chicken eggs (0.01(*) mg/kg) is withdrawn. The HR for eggs was 0.05 mg/kg.

RECOMMENDATIONS

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

For plants and animals: Definition of the residue (for compliance with the MRL and for estimation of dietary intake): *sum of haloxyfop (including haloxyfop-P), its esters and its conjugates expressed as haloxyfop.*

The residue is fat soluble.

	Commodity	MRL, mg/kg		STMR or STMR-P	HR or HR-P
CCN	Name	New	previous	mg/kg	mg/kg
AL 1021	Alfalfa forage (green)	W [a]	5 [4]		
FI 0327	Banana	0.02 *	0.05 *	0	0
VD 0071	Beans (dry)	3		0.335	
VP 0061	Beans, except broad bean and soya bean (green pods and immature seeds)	0.5		0.085	0.26
MO 1280	Cattle, Kidney	W [b]	1		
MO 1281	Cattle, Liver	W [b]	0.5		
MM 0812	Cattle meat	W [b]	0.05		
ML 0812	Cattle milk	W [b]	0.3		
PE 0840	Chicken eggs	W [c]	0.01 *		
PM 0840	Chicken meat	W [c]	0.01 * [5]		
PO 0840	Chicken, Edible offal of	W [c]	0.05		
VD 0524	Chick-pea (dry)	0.05		0.02	

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[4] Fresh weight basis

[5] With adhering skin

	Commodity	MRL, mg/kg		STMR or STMR-P	HR or HR-P
CCN	Name	New	previous	mg/kg	mg/kg
FC 0001	Citrus fruits	0.02 *	0.05 *	0	0
SB 0716	Coffee beans	0.02*		0	0
SO 0691	Cotton seed	0.7	0.2	0.1	
OC 0691	Cotton seed oil, crude	W	0.5		
MO 0105	Edible offal (Mammalian)	2		0.27	1.42
PE 0112	Eggs	0.1		0.022	0.05
AM 1051	Fodder beet	0.4	0.3	0.02	0.30
AV 1051	Fodder beet leaves or tops	W [a]	0.3[b]		
FB 0269	Grapes	0.02 *	0.05 *	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.5 (fat)		0.035 (fat) 0.006 (muscle)	0.33 (f) 0.041 (m)
FM 0183	Milk fats	7		0.87	
ML 0106	Milks	0.3		0.033	
VA 0385	Onion, Bulb	0.2		0.035	0.12
SO 0697	Peanut	W	0.05		
AL 0697	Peanut fodder	5		2.1	3.0
VD 0072	Peas (dry)	0.2		0.04	
VP 0063	Peas (pods and succulent = immature seeds)	0.7	0.2	0.11	0.53
VP 0064	Peas, shelled (succulent seeds)	1		0.08	0.75
FP 0009	Pome fruits	0.02 *	0.05 *	0	0
VR 0589	Potato	W	0.1		
PM 0110	Poultry meat	0.7 (fat)		0.13 (fat) 0.032 (muscle)	0.52 (f) 0.11 (m)
PO 0111	Poultry, Edible offal of	0.7		0.21	0.61
VD 0070	Pulses	W [d]	0.2		
SO 0495	Rape seed	3	2	0.07	
OC 0495	Rape seed oil, crude	W [e]	5	0.17	
OR 0495	Rape seed oil, edible	W [e]	5	0.16	
CM 1206	Rice bran, unprocessed	W	0.02 *		
CM 0649	Rice, husked	W	0.02 *		
CM 1205	Rice, polished	W	0.02 *		
VD 0541	Soya bean (dry)	2		0.055	
OC 0541	Soya bean oil, crude	W [f]	0.2	0.044	
OR 0541	Soya bean oil, refined	W [f]	0.2	0.041	
FS 0012	Stone fruits	0.02 *		0	0
VR 0596	Sugar beet	0.4	0.3	0.02	0.30
AV 0596	Sugar beet leaves or tops	W [a]	0.3		
SO 0702	Sunflower seed	0.3	0.2	0.05	

W: the recommendation is withdrawn

* At or about the limit of quantification.

[a] The current policy is not to recommend maximum residue levels for fresh animal forages, but to use the data in livestock dietary burden calculations.

[b] Recommendations for cattle kidney and cattle liver are withdrawn, to be replaced by a recommendation for mammalian edible offal. Recommendations for cattle meat and cattle milk are withdrawn and replaced by recommendations for mammalian meat and milks.

[c] Recommendations for chicken eggs, meat and edible offal are withdrawn, to be replaced by recommendations for poultry commodities.

[d] The recommendation for pulses is withdrawn to be replaced by recommendations for individual commodities.

[e] The recommendations for maximum residue levels for rape seed oils are withdrawn, because they are covered by the recommendation for rape seed.

[f] The recommendations for maximum residue levels for soya bean oils are withdrawn, because they are covered by the recommendation for soya bean (dry).

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes of haloxyfop, based on the STMRs estimated for 22 commodities, for the GEMS/Food Consumption Cluster Diets were in the range of 20 to 80% of the maximum ADI (0.0007 mg/kg bw/day)(Annex 3 of the 2009 JMPR Report). The Meeting concluded that the long-term intake of residues of haloxyfop resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for haloxyfop was calculated for food commodities and their processed fractions for which maximum residue levels were estimated and for which consumption data were available. The results are shown in Annex 4 of the 2009 JMPR Report.

The IESTI represented 0–10% of the ARfD for the general population and 0–10% of the ARfD for children. The Meeting concluded that the short-term intake of residues of haloxyfop, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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GHB-P-024	Braga AM and Bontempi Filho O	1985	Residues of haloxyfop in soya beans following postemergence application of Verdict* herbicide. Dow Chemical, Brazil. Report GHB-P-024. Includes RT/12/84, RT/13/84, RT/14/84, RT/15/84, RT/16/84, RT/17/84, RT/25/84, RT/26/84, RT/28/84, RT/29/84, RT/32/84, RT/33/84, RT/34/84, RT/35/84, RT/36/84 and RT/37/84. Unpublished.
GHB-P-034	Rodrigues MA	1986	Residues of haloxyfop in cottonseed following post- emergence of Verdict* herbicide—Brazil. Dow Chemical, Brazil. Report GHB-P-034. Unpublished.
GHB-P-9783	Devine HC	2002	Residues of DE-535 in vegetable bean at harvest following a single application of DE-535 (EF-1400), EU southern zone—2001. Dow AgroSciences, UK. Report GHB-P-9783. Unpublished
GH-C 1507	Bauriedel WR and Miller JH	1982	A greenhouse study of the early metabolic fate of three esters of DOWCO 453 applied to soya beans. Report GH-C 1507. Dow Chemical Company, USA. Unpublished.
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GH-C 1618	Yackovich PR and Miller JH	1983	The fate of DOWCO 453 herbicide applied as the butyl ester to soya bean plants. Study GH-C 1618. Dow Chemical Company, USA. Unpublished.
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GH-C 1624	Yackovich PR and Miller JH	1983	The fate of DOWCO 453 fed to lactating goats. Dow Chemical, USA. Report GH-C 1624. Unpublished.
GH-C 1625	Gardner RC	1983	Residues of DOWCO 453 in soya beans (revised). Dow Chemicals, USA. Report GH-C 1625. Unpublished.
GH-C 1634	Stafford LE and Miller JH	1983	A study of the metabolism of DOWCO 453 in cotton. Study GH-C 1634. Dow Chemical Company, USA. Unpublished.
GH-C 1635	Yackovich PR and Miller JH	1983	The fate of DOWCO 453 fed to laying hens, Study GH-C 1635. Dow Chemical Company, USA. Unpublished.
GH-C 1680	Kutschinski AH and Bjerke EL	1984	Residues in tissues from beef calves fed haloxyfop. Dow Chemical, USA. Report GH-C 1680. Unpublished
GH-C 1701	Kutschinski AH and Bjerke EL	1984	Residues in tissues and eggs from chickens fed haloxyfop. Dow Chemical, USA. Report GH-C 1701. Unpublished.

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GH-C 1758	Bjerke EL, Kutschinski AH, Gardner RC and Majorski SA	1985	A residue study in rotational crops. Dow Chemical, USA. Report GH-C 1758. Unpublished
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GH-C 2037	Gardner RC	1990	Determination of haloxyfop in apple fractions after treatment of the orchard floor with haloxyfop-methyl herbicide. Dow Chemical, USA. Report GH-C 2037. Unpublished.
GH-C 2162	Cobb JD; Swayze KM and Stuart MP	1989	Solubility of XRD-535 methyl ester in organic solvents. Report GH-C 2162. Dow Chemical USA. Unpublished.
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GHE P 2140	Dawson J	1990	The determinations of physico chemical properties of XRD 535 methyl ester according to OECD/EEC guidelines. Report GHE P 2140. Dow Elanco, UK. Unpublished.
GHE-P-10092	Rawle NW	2002	Residues of DE-535 in oilseed rape at harvest following a single application of DE-535 (EF-1400), EU southern zone—2001. Dow AgroSciences, UK. Report GHE-P-10092. Unpublished.
GHE-P-11071	Devine HC	2007	Residues of DE-535 in alfalfa at intervals following a single application of DE-535 (EF-1400), EU northern zone—2004. Dow AgroSciences, UK. Report GHE-P-11071. Study CEMS-2337. Unpublished.
GHE-P-11073	Devine HC	2007	Residues of DE-535 in vegetable peas at harvest following a single application of DE-535 (EF-1400) EU southern zone 2004. Study CEMS-2239. Dow AgroSciences, UK. Report GHE-P-11073. Unpublished.
GHE-P-1125	Yon DA	1984	Determination of residues in haloxyfop in selected sugar process fractions obtained from sugar beet treated with an EC Formulation Containing 125 g haloxyfop ethoxy ethyl (EE) ester/litre—UK trials 1983. Dow Chemical, UK. Report GHE-P-1125. Unpublished.
GHE-P-1148	Yon DA	1984	Determination of residues of haloxyfop in grapes treated with a 125 g haloxyfop ethoxyethyl (EE) Ester/litre EC formulation in France (1982 and 1983). Dow Chemical, UK. Report GHE-P-1148. Includes RT 277-7/83. Unpublished.
GHE-P-11656	Balluff M	2007	Residues of haloxyfop-R in oilseed rape at harvest following a single spring application of EF-1400, southern European zone (France, Spain, Italy, Greece)—2006. GAB Study 20064009/E3-FPRA. Dow AgroSciences, UK. Report GHE-P-11656. Includes F06W028R, S06W027R, I06W023R and GR06W013R. Unpublished,
GHE-P-11657	Balluff M	2007	Residues of haloxyfop-R in oilseed rape at interval or at harvest following a single spring application of EF-1400, northern European zone (Germany, France, Poland)—2006. Dow AgroSciences, UK. Report GHE-P-11657. Includes F06W080R, PL06W009R, G06WO17R and G06WO18R. Unpublished.
GHE-P-1313R	Yon DA	1987	Residues of haloxyfop in selected process fractions obtained from the refining of winter oilseed rape treated with a 125g Haloxyfop ethoxyethyl ester/litre formulation, France 1982/83. Dow Chemical Co., UK. Report GHE-P-1313R. Unpublished.
GHE-P-1523	Dawson J	1986	Residues of haloxyfop in grapes following application of Gallant*—France 1985. Dow Chemical, UK. Report GHE-P-1523. Includes RT 304/85, RT 310/85, RT 311/85. Unpublished.

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GHE-P-1965	Perkins JM	1988	Residues of haloxyfop in apples following basal application of Gallant 125 Herbicide—Italy 1987. Dow Chemical, UK. Report GHE-P-1965. Unpublished.
GHE-P-1966	Perkins JM and Veal P	1990	Residues of haloxyfop in spring field peas (*pois proteagineux*) following application of Gallant (EF-687) or DowCo 535 (EF 1020) herbicides—France 1988. Study R88-39. DowElanco, UK. Report GHE-P-1966. Unpublished.
GHE-P-1972	Perkins JM and Veal P	1990	Residues of Haloxyfop in sugarbeet roots following application of Gallant (EF 687) or Dowco 534 (EF 1020) herbicide—France 1988. Study R88-36. DowElanco, UK. Report GHE-P-1972. Unpublished.
GHE-P-1973	Perkins JM and Harrison C	1990	Residues of haloxyfop in oilseed rape seed following an autumn application of Gallant (EF 687) or DOWCO 534 (EF 1020) herbicides—France 1988. Study R89-5. DowElanco, UK. Report GHE-P-1973. Unpublished.
GHE-P-2036	Perkins J, Teasdale R and Veal P	1990	Residues of haloxyfop in sugarbeet (whole plant, roots and leaves) after the application of Gallant (EF 687) and Dowco 535 (EF 1020)—Germany 1988. Project R 88-22. DowElanco, UK. Report GHE-P-2036. Unpublished.
GHE-P-2055	Perkins JM and Harrison C	1990	Residues of haloxyfop in spring field peas (*pois proteagineux*) following application of Gallant (EF-687) or DowCo 535 (EF 1020) herbicides—France 1989. Study R89-6. DowElanco, UK. Report GHE-P-2055. Unpublished.
GHE-P-2057	Perkins JM and Harrison C	1990	Residues of haloxyfop in peas (*pois conserve*) following application of Gallant (EF-687) or DOWCO 535 (EF-1020) herbicides—France 1989. Study R89-7. DowElanco, UK. Report GHE-P-2057. Unpublished.
GHE-P-2058	Perkins JM and Harrison C	1990	Residues of haloxyfop in winter field peas (*pois proteagineux*) following application of Gallant (EF 687) or DowCo 535 (EF 1020) herbicides—France 1989. Study R89-4. DowElanco, UK. Report GHE-P-2058. Unpublished.
GHE-P-2059	Perkins JM and Harrison C	1990	Residues of haloxyfop in sunflower seed following application of Gallant (EF-687) or Dowco 535 (EF 1020) herbicides—France 1988 and 1989. Studies R88-37 and R89-8. DowElanco, UK. Report GHE-P-2059. Unpublished.
GHE-P-2115	Perkins JM, MacDonald I, Gillis N, Taylor L and Flatt S	1990	Residues of haloxyfop in grapes following application of DOWCO 535 (EF-1020) herbicide—Italy 1989. DowElanco, UK. Report GHE-P-2115. Study R89-31. Unpublished.
GHE-P-2144	Perkins JM, MacDonald I, Gillis N and Flatt S	1990	Residues of haloxyfop in oilseed rape (whole plant, seed and straw) after the autumn application of Gallant (EF 687) and DOWCO 535 (EF 1020)—Germany 1988–89. Project R88-21. DowElanco, UK. Report GHE-P-2144. Unpublished.
GHE-P-2444	Butcher S	1991	Residues of haloxyfop in field beans following a single application of EF 1020 Herbicide—Germany 1990. DowElanco, UK. Report GHE-P-2444. Unpublished.
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GHE-P-2802	Butcher S and Long T	1992	Residues of haloxyfop in cotton following a single application of DE-535 (EF-1020) herbicide—Spain 1991. DowElanco, UK. Report GHE-P-2802. Study R91-31. Unpublished.
GHE-P-3078	Hastings M, Rawle N and Bolton A	1993	Residues of haloxyfop in sugar beet at harvest following a single application of DE 535 (EF 1020)—Italy 1992. Study R92-45. DowElanco, UK. Report GHE-P-3078. Includes CEMS-185. Unpublished.
GHE-P-3157	Hastings M and Butcher S	1993	The stability of haloxyfop in green peas stored under frozen conditions. Study ST91-9. DowElanco, UK. Report GHE-P-3157. Unpublished.
GHE-P-3158	Hastings M and Butcher S	1993	The stability of haloxyfop in cabbage stored under frozen conditions. Study ST91-10. DowElanco, UK. Report GHE-P-3158. Unpublished.
GHE-P-3594	Hale K and Trigg R	1994	Investigations into the metabolism of DE-535 methyl ester (haloxyfop-R methyl ester) in soil according to BBA guidelines IV 4.1. DowElanco Europe, UK. Study 12G. Report GHE-P-3594. Unpublished.

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GHE-P-8559	Nicholson A	2000	Residues of DE-535 acid in vegetable bean at harvest following a single application of EF-1400, European Union, northern zone—1999. Dow AgroSciences, UK. Report GHE-P-8559. Unpublished.
GHE-P-8573	Nicholson A	2000	Residues of DE-535 acid in vegetable pea at harvest following a single application of EF-1400, EU northern zone—1999. Trials R99-133A, R99-133B, R99-133C and R99-133D. Dow AgroSciences, UK. Report GHE-P-8573. Unpublished.
GHE-P-8577	Nicholson A	2000	Residues of DE-535 acid in vegetable bean at harvest following a single application of EF-1400, European Union, southern zone—1999. Dow AgroSciences, UK. Report GHE-P-8577. Unpublished.
GHE-P-8578	Nicholson A	2000	Residues of DE-535 acid in vegetable pea at harvest following a single application of EF-1400, EU southern zone—1999. Trials R99-168A and R99-168B. Dow AgroSciences, UK. Report GHE-P-8578. Unpublished.
GHE-P-8849	Rawle NW	2001	Residues of DE-535 in vegetable bean at harvest following a single application of DE-535 (EF-1400, EU northern zone—2000. Dow AgroSciences, UK. Report GHE-P-8849. Unpublished.
GHE-P-8851	Rawle NW	2001	Residues of DE-535 in vegetable peas at harvest following a single application of DE-535 (EF-1400), EU northern zone—2000. Trials CEMS-1248A, CEMS-1248B, CEMS-1248C and CEMS-1248D. Dow AgroSciences, UK. Report GHE-P-8851. Unpublished.
GHE-P-8852	Rawle NW	2002	Residues of DE-535 in vegetable peas at harvest following a single application of DE-535 (EF-1400), EU southern zone—2000. Trials CEMS-1249A and CEMS-1249B. Dow AgroSciences, UK. Report GHE-P-8852. Unpublished.
GHE-P-8854	Rawle NW	2001	Residues of DE-535 in cotton at harvest following a single application of DE-535 (EF-1400), EU southern zone—2000. Dow AgroSciences, UK. Report GHE-P-8854. Includes CEMS-1251. Unpublished.
GHE-P-8857	Rawle NW	2002	Residues of DE-535 in oilseed rape at harvest following a single application of DE-535 (EF-1400, EU northern zone—2000. Dow AgroSciences, UK. Report GHE-P-8857. Unpublished.
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GHE-P-8859	Rawle NW	2002	Residues of DE-535 in soya beans at harvest following a single application of DE-535 (EF-1400), EU southern zone—2000. Study CEMS-1257. Dow AgroSciences, UK. Report GHE-P-8859. Unpublished.
GHE-P-8860	Rawle NW	2001	Residues of DE-535 in sunflower at harvest following a single application of DE-535 (EF-1400), EU northern zone—2000. Study CEMS-1258. Dow AgroSciences, UK. Report GHE-P-8860. Unpublished.
GHE-P-8861	Rawle NW	2001	Residues of DE-535 in sunflower seeds at harvest following a single application of DE-535 (EF-1400) EU southern zone—2000. Study CEMS-1259. Dow AgroSciences, UK. Report GHE-P-8861. Unpublished.
GHE-P-8867	Rawle NW	2001	Residues of DE-535 in sugarbeet at harvest following a single application of DE-535 (EF 1400), EU northern zone—2000. Trials CEMAS-1266A, CEMAS-1266B, CEMAS-1266C. Dow AgroSciences, UK. Report GHE-P-8867. Unpublished.
GHE-P-8868	Rawle NW	2002	Residues of DE-535 in sugarbeet at harvest following a single application of the DE-535 (EF-1400), EU southern zone—2000. Study CEMS-1267. Dow AgroSciences, UK. Report GHE-P-8868. Unpublished.
GHE-P-8870	Kang J	2001	Residues of DE-535 in onions at harvest following a single application of DE-535 (EF-1400) EU northern zone—2000. Study CEMS-1269. Dow AgroSciences, UK. Report GHE-P-8870. Unpublished.
GHE-P-8871	Rawle NW	2001	Residues of DE-535 in onions at harvest following a single application of DE-535 (EF-1400) EU southern zone—2000. Study CEMS-1270. Dow AgroSciences, UK. Report GHE-P-8871. Unpublished.

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GHE-P-9337	Rawle NW	2001	Residues of DE-535 acid in cotton at harvest following a single application of EF-1400, European Union, southern zone—1999. Dow AgroSciences, UK. Report GHE-P-9337. Includes R99-134A, R99-134B, R99-134C, R99-134D. Unpublished.
GHE-P-9782	Devine HC	2002	Residues of DE-535 in soya beans at harvest following a single application of DE-535 (EF-1400), Hungary and Germany—2001. Study CEMS-1569. Dow AgroSciences, UK. Report GHE-P-9782. Unpublished.
GHE-P-9784	Devine, HC	2002	Residues of DE-535 in sunflower seed at harvest following a single application of DE-535 (EF-1400), EU southern zone—2001. Study CEMS-1571. Dow AgroSciences, UK. Report GHE-P-9784. Unpublished.
GHF-P 1003	Wilson BI, Tidswell JN, Downard P and Nott P	1990	Residues of haloxyfop in field peas after application of EF 1020 or Verdict 104 selective herbicide in South Australia and Victoria 1989. DowElanco, NZ. Report GHF-P 1003. Unpublished. [*Note: data are included in later version- GHF-P 1392*]
GHF-P 1110	Tidswell JN, Cowles J and Plater C	1991	Residues of haloxyfop in lemons after applications of Verdict 104 selective herbicide in New South Wales 1990. DowElanco, NZ. Report GHF-P 1110. Unpublished.
GHF-P 1122	Cowles J and Wall H	1991	Residues of haloxyfop in stone fruit after the application of Verdict 104 selective herbicide in Victoria, Australia in 1991. DowElanco, NZ. Report GHF-P 1122. Unpublished.
GHF-P 1147	Tidswell JN, Cowles J and Lobb P	1991	Residues of haloxyfop in lemons after application of Verdict 104 selective herbicide in New Zealand 1991. DowElanco, NZ. Report GHF-P 1147. Unpublished.
GHF-P 1149	Wilson BI and Wells GA	1991	Residues of haloxyfop in bananas after application of Verdict 104 selective herbicide and IWD-4180 in Queensland 1991. DowElanco, NZ. Report GHF-P 1149. Unpublished.
GHF-P 1150	Cowles J and Plater C	1991	Residues of haloxyfop in grapes after the application of Verdict 104 selective herbicide in New South Wales, Australia in 1990. DowElanco, NZ. Report GHF-P 1150. Unpublished.
GHF-P 1355	Wilson BI and Tidswell JN	1994	Effect of oil on residues of haloxyfop in lucerne, Australia 1989. DowElanco, NZ. Report GHF-P 1355. Unpublished.
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GHF-P 1700	Cowles RJ, Utting A, Williamson W and Taylor J	1998	Residues of haloxyfop-R in canola forage, fodder and grain after application of Verdict herbicide in Australia 1997. Dow AgroSciences, NZ. Report GHF-P 1700. Unpublished.
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GHF-P 1943	Cowles J	2001	Haloxyfop residues in Australian lucerne after one application of Verdict 520 herbicide in the 1998–1999 season. Dow AgroSciences, Australia. Report GHF-P 1943. Unpublished.
GHF-P 1989	Wilson BI and Tidswell JN	1989	Residues of haloxyfop in pigeon peas after application of Verdict 104 selective herbicide or XRM 5045 in Australia. Ivon-Watkins Dow, NZ. Report GHF-P-1989. Unpublished.
GHF-P 925	Wilson BI and Tidswell JN	1990	Residues of haloxyfop in lucerne after applications of Verdict 104 selective herbicides in New South Wales Australia 1989. Ivon-Watkins-Dow Ltd, NZ. Report GHF-P 925. Unpublished.

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GHF-P-1121	Cowles J and Wall H	1991	Residues of haloxyfop in apples after the application of Verdict 104 selective herbicide in Victoria, Australia in 1991. DowElanco, NZ. Report GHF-P-1121. Unpublished.
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GHF-P-584	Wilson BI	1987	Residues of haloxyfop in apples after basal application of haloxyfop EE thirty days before harvest in New Zealand, 1986. Ivon Watkins-Dow, NZ. Report GHF-P-584. Unpublished.
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