HALOXYFOP (193)

EXPLANATION

Haloxyfop was evaluated for the first time by the 1995 JMPR. That Meeting received data on residues in beans and peas but the exact Codex commodities to which the data referred were not clear. The 1995 Meeting agreed not to estimate maximum residue levels until the commodity descriptions were clarified. Supplementary information on the commodity described as "peas" has now been made available.

The 1995 Meeting could not complete the evaluation of the ruminant and poultry metabolism studies in the time available and reviewed the residue data on ruminants and poultry on the assumption that metabolism in these species is essentially the same as indicated in the metabolism studies on rats, mice, dogs, monkeys and humans. The present Meeting completed the evaluation of the studies of ruminant and poultry metabolism.

The 1995 JMPR estimated a number of maximum residue levels, but agreed not to recommend their use as MRLs because of the lack of critical supporting data on the uptake of soil degradation products by crops. Studies on the uptake of haloxyfop or its degradation products from soil treated with haloxyfop have been made available to the present Meeting.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Ruminants. Two lactating goats were dosed with [*phenyl*-U-^{14}C]haloxyfop in gelatin capsules twice daily for ten consecutive days at a rate equivalent to 16 ppm in the diet (Yackovich and Miller, 1983a). Urine and faeces were collected each morning during the test period. Blood samples were taken daily and expired air was collected from one goat for a ten-hour period. Milk was collected each morning and evening. Twelve hours after the final dose the animals were slaughtered and tissue samples and gut contents taken.

The recovery of the administered radioactivity was 90-96%. Most was excreted in the urine (84-92% of total administered dose) as unchanged haloxyfop; milk contained 1.9-3.2% and faeces 1.5-1.9%. No radioactivity was found in the expired air. Radioactivity as haloxyfop equivalents was 0.02 mg/kg in muscle, 0.1 mg/kg in fat, 0.4 mg/kg in liver and 1.3 mg/kg in kidney.

The liver and kidneys contained only haloxyfop, while the fat contained a non-polar conjugate which was easily hydrolysed to yield haloxyfop under alkaline conditions. The residue in muscle was not identified because the radioactivity was too low.

The radioactivity in the milk reached a plateau within two days after the first dose at 0.2 to 0.3 mg/kg haloxyfop equivalent, representing about 2 to 3% of the daily dose. The residue was

primarily in the milk fat fraction in the form of non-polar conjugate(s) which were easily hydrolysed to yield haloxyfop under alkaline conditions. Enzymatic hydrolysis with lipase (triacylglycerol-acyl-hydrolase) indicated that the conjugates were triacylglycerols.

The recovery of radioactivity and its distribution are shown in Table 1.

Table 1. Recovery and distribution of radioactivity following the feeding of [^{14}C]haloxyfop to lactating goats.

Sample	Recovery of radioactivity, % of total dose	Radioactivity as mg/kg haloxyfop equivalent	% of haloxyfop in residual radioactivity
Fat	0.04-0.06	0.06-0.11[1]	>90[3]
Heart	<0.01	0.02-0.07[1]	
Kidneys	0.09-0.11	1.07-1.45[1]	>90[4]
Liver	0.14-0.16	0.31-0.45[1]	>90[4]
Muscle	0.09-0.11	<0.01-0.02[1]	
Gut contents	0.25-0.40	-	
Milk (whole)	1.92-3.18	0.23-0.33[2]	
Milk (fat)	-	1.53-3.41[2]	>93[3]
Urine	84.4-91.7	-	99[5]
Faeces	1.48-1.88	-	
Expired air	<0.01	-	
Total	89.8-96.2	-	

[1] At 12 hours after the final dose
[2] Average concentration from third day
[3] Non-polar conjugate
[4] Free haloxyfop or polar conjugate
[5] Free haloxyfop

Poultry. Four laying hens were dosed with [*phenyl*-U-^{14}C]haloxyfop in gelatin capsules once a day for eleven consecutive days at a rate equivalent to 12 ppm in the diet (Yackovich and Miller, 1983b). Eggs and faeces were collected each morning during the test period. The hens were killed 24 hours after the final dose and tissue samples and gut contents taken.

The recovery of the administered radioactivity was 93-102%, mostly from the excreta with small amounts in eggs (82-90% and 1.1-2.6% of total administered dose respectively). In the tissues the radioactivity as haloxyfop equivalent was 0.12 mg/kg in muscle, 0.99 mg/kg in fat, 1.8 mg/kg in liver and 4.2 mg/kg in the kidneys.

The excreta, liver and kidneys contained mainly haloxyfop with some polar and/or non-polar conjugates, while most of the radioactivity in the fat was due to a non-polar conjugate of haloxyfop. Both the polar and non-polar conjugates were easily hydrolysed to yield haloxyfop under alkaline conditions. The residue in muscle was not identified because its radioactivity was too low.

The residue in the eggs was mainly in the yolk, where its radioactivity reached a plateau within seven days after the first dose at about 3 mg/kg haloxyfop equivalent, representing about 2 to 3% of the daily dose. As in goat milk fat, the residue in the egg yolk was identified as non-polar conjugate(s) of haloxyfop, easily hydrolysed by alkali to yield haloxyfop and shown by enzymatic hydrolysis with lipase to be triacylglycerol(s). The recovery and distribution of radioactivity are shown in Table 2.

Table 2. Recovery and distribution of radioactivity following the feeding of [^{14}C]haloxyfop to laying hens.

Sample	Recovery of radioactivity, % of total dose	Radioactivity as mg/kg haloxyfop equivalent	% of haloxyfop in residual radioactivity
Fat	0.15	0.99[1]	99[(3)]
Heart	-	0.28[1]	
Kidneys	0.23	4.22[1]	90[(4)]
Liver	0.37	1.82[1]	91[(4)]
Muscle	0.25	0.12[1]	
Gut contents	7.5	-	
Egg (whole)	1.58	-	
Egg (white)	-	0.25[2]	
Egg (yolk)	-	2.94[2]	92[(3)]
Developing yolk	0.49	-	
Faeces	86.54	-	97[(5)]
Total	97.95	-	

[1] At 24 hours after final dose
[2] Average concentration from 7th day
[3] Non-polar conjugate
[4] 60-70% as free haloxyfop, non-polar and polar conjugates
[5] Free haloxyfop or polar conjugates

Uptake by crops from treated soil

Because haloxyfop is often applied to soil the 1995 Meeting concluded that information was needed on the uptake by crops of haloxyfop and its degradation products from soil. Appropriate studies were reported to the present Meeting.

An emulsifiable formulation of [*phenyl*-U-^{14}C]haloxyfop-butyl was applied to bare soil at a rate equivalent to 0.56 kg ai/ha (Yackovich and Miller, 1983c). Thirty days later the plot was tilled and planted with spring wheat, soya beans, carrots, turnips and lettuce. The crops were grown to maturity and the uptake of ^{14}C determined by combustion and LSC. The highest residue, 0.07 mg/kg haloxyfop equivalent, was found in immature soya bean foliage sampled 56 days after planting. Residues in the edible portions of the crops were ≤0.01 mg/kg haloxyfop equivalent. Owing to the

low levels of radioactivity, attempts to isolate and identify the residues were unsuccessful. The results are given in Table 3.

Table 3. Uptake of ^{14}C by crops planted 30 days after treatment of soil with [^{14}C]haloxyfop at 0.56 kg ai/ha.

Sample	Days from planting to harvest	^{14}C as mg/kg haloxyfop equivalent
Lettuce	49	0.01
Turnip foliage	64	<0.01
Turnip root	64	<0.01
Wheat grain	110	0.01
Wheat straw	110	0.02
Soya bean	113	<0.01
Soya bean forage	56	0.07
Soya bean straw	113	0.01
Carrot forage	115	<0.01
Carrot root	115	<0.01

An EC formulation of [*phenyl*-U-^{14}C]haloxyfop-butyl was applied to a crop of 37.5 cm soya bean plants at a rate equivalent to 0.56 kg ai/ha (Yackovich and Miller, 1983d). After harvesting the crop 130 days after treatment, the top 5 cm of soil was removed, brought into the laboratory, and sown with spring wheat, soya beans, sugar beet and lettuce. The crops were grown to maturity and the uptake of ^{14}C determined by combustion and LSC. Residues in the edible portions of the commodities were ≤0.04 mg/kg haloxyfop equivalent. Owing to the low levels of radioactivity, the residues could not be identified. The results are shown in Table 4.

Table 4. Uptake of ^{14}C by rotational crops from soil after treatment of original crop with [^{14}C]haloxyfop at 0.56 kg ai/ha. Rotational crops planted 130 days after treatment of primary crop.

Rotational crop sample	Days from planting to harvest	^{14}C as mg/kg haloxyfop equivalent
Lettuce	37	0.01
Lettuce	50	0.01
Lettuce	67	0.01
Lettuce	95	0.01
Soya bean	117	0.04
Soya bean forage	50	0.02
Soya bean straw	117	0.05
Wheat grain	104	0.02
Wheat straw	104	0.02
Sugar beet	132	0.01
Sugar beet forage	132	0.02

An emulsifiable formulation of [*phenyl*-U-^{14}C]haloxyfop-butyl was applied to a crop of soya bean plants at a rate equivalent to 0.28 or 0.56 kg ai/ha. Carrots, sugar beet, lettuce, wheat and soya beans were planted in the same plot the following year, grown to maturity and analyzed to determine the uptake of ^{14}C (Yackovich and Miller, 1984). The ^{14}C was determined by combustion and LSC. The highest residues, 0.007 and 0.01 mg/kg haloxyfop equivalent, were seen in soya bean grain and straw after treatment of the original crop at the higher application rate. Residues in the edible portions of the other crops were <0.005 mg/kg haloxyfop equivalent. The residues were too low for isolation and identification. The results are given in Table 5.

Table 5. Uptake of ^{14}C by rotational crops from soil after treatment of original crop with [^{14}C]haloxyfop. All rotational crops planted 240 days after original application.

Rotational crop sample	Application to original crop, kg ai/ha	Days from planting to harvest	^{14}C as mg/kg haloxyfop equivalent
Wheat grain	0.28	95	<0.005
Wheat straw	0.28	95	0.005
Soya bean	0.28	161	<0.005
Soya bean forage	0.28	75	<0.005
Soya bean straw	0.28	161	<0.005
Soya bean	0.56	161	0.007
Soya bean forage	0.56	75	<0.005
Soya bean straw	0.56	161	0.01
Lettuce	0.56	39	<0.005
Lettuce	0.56	47	<0.005
Lettuce	0.56	69	<0.005
Carrot root	0.56	153	<0.005
Carrot forage	0.56	153	<0.005
Sugar beet	0.56	139	<0.005
Sugar beet forage	0.56	139	<0.005

Unlabelled haloxyfop-methyl was applied to soya beans at a rate of 0.28 kg ai/ha or to cotton at a rate of 0.28 or 0.56 kg ai/ha (Bjerke *et al*., 1985). At 25-34 or 92-148 days after application, the primary crop was harvested and wheat, lettuce or sugar beet planted as rotational crops in the same plots. The crops were grown to maturity and analyzed by gas chromatography to determine the uptake of haloxyfop. The highest residues were found in immature wheat forage grown on plots where the primary crop had been harvested 25 days after treatment, at a level of 0.01 mg/kg. Residues in the edible portions of the rotational crops were <0.01 mg/kg haloxyfop. The validated LOD was 0.01 mg/kg for all substrates except wheat straw, for which it was 0.02 mg/kg.

USE PATTERN

Information on use patterns was submitted to the 1995 JMPR and reported in the 1995 monograph. The use pattern on beans and peas was recorded in Table 8 (p. 422).

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials data on peas were submitted to the 1995 JMPR and reported in the 1995 monograph (Table 18, p. 432). The present Meeting was informed that all commodities referred to as "peas" were *Pisum sativum*. Because it is now possible to determine which residues resulted from treatments according to GAP, the Table is repeated below as Table 6.

Peas (pods and succulent seeds). Four supervised trials in France with racemic haloxyfop-etotyl were at 0.1 or 0.21 kg ai/ha with PHIs of 36-68 days. Three others in France and four in Germany were with haloxyfop-R-methyl at 0.052 or 0.1 kg ai/ha with PHIs of 36-60 days (Table 6).

Table 6. Residues of haloxyfop in peas (pods and succulent seeds). All single EC applications.

Country Year	Application			PHI, days	Growth stage at last treatment	Residues, mg/kg	Reference
	Compound/Form.	Kg ai/ha	Kg ai/hl				
France 1984	SR-EE EC	0.1	0.016	68	15-20 cm height	0.07	GHE-P-1671 (N66)
France 1988	SR-EE EC	0.1 0.21	N.S.[1] N.S.	39 39	8-9 leaves	<0.05 0.11	GHE-P-1956 (N30(R))
France 1989	SR-EE EC	0.1 0.21	0.021 0.042	36 36	5-6 leaves	0.03 0.06	GHE-P-2057 (N31(R))
France 1989	SR-EE EC	0.1 0.21	0.042 0.084	36 36	flower buds hidden by top leaves	0.04 0.07	GHE-P-2057 (N31(R))
France 1988	R-Me EC	0.052 0.1	N.S. N.S.	39 39	8-9 leaves	<0.05 0.06	GHE-P-1956 (N30(R))
France 1989	R-Me EC	0.052 0.1	0.01 0.021	36 36	5-6 leaves	0.03 0.04	GHE-P-2057 (N31(R))
France 1989	R-Me EC	0.052 0.1	0.021 0.042	36 36	flower buds hidden by top leaves	0.03 0.05	GHE-P-2057 (N31(R))
Germany 1989	R-Me EC	0.1	0.035	43 56	3 leaves	<0.02[2] ≤0.02	GHE-P-2154 (N36(R))
Germany 1989	R-Me EC	0.1	0.035	38 53	4 leaves	<0.02[2] ≤0.02	GHE-P-2154 (N36(R))
Germany 1989	R-Me	0.1	0.026	42	10 leaves	0.07[2]	GHE-P-2154 (N36(R))
Germany 1989	R-Me EC	0.1	0.026	42 60	6-7 leaves	0.03[2] ≤0.02	GHE-P-2154 (N36(R))

[1] Not specified in report
[2] Pod

APPRAISAL

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clear. The 1995 Meeting agreed not to estimate maximum residue levels until the commodity descriptions were clarified. Supplementary information on the commodity described as "peas" has now been made available.

The 1995 Meeting could not complete the evaluation of the ruminant and poultry metabolism studies in the time available and reviewed the residue data on ruminants and poultry on the assumption that metabolism in these species is essentially the same as indicated in the metabolism studies on rats, mice, dogs, monkeys and humans. The present Meeting completed the evaluation of the studies of ruminant and poultry metabolism.

The 1995 JMPR estimated a number of maximum residue levels, but agreed not to recommend their use as MRLs because of the lack of critical supporting data on the uptake of soil degradation products by crops. Studies on the uptake of haloxyfop or its degradation products from soil treated with haloxyfop have been made available to the present Meeting.

Metabolism in lactating goats and laying hens

Metabolic studies on lactating goats and laying hens with 10 or 11 days consecutive oral dosing at rates equivalent to 12-16 ppm in the feed were reported.

In goats, the administered haloxyfop was rapidly absorbed from the gastrointestinal tract and mainly excreted in the urine unchanged (84-92%). The radioactivity remaining in the body was relatively low, at levels of 0.02, 0.1, 0.4 and 1.3 mg/kg haloxyfop equivalent in the muscle, body fat, liver and kidneys respectively 12 hours after the last dose. The liver and kidneys contained only haloxyfop or its polar conjugates; the radioactivity in muscle was too low for identification of the residues. The predominant metabolites in the body fat and milk fat were identified as non-polar conjugates of haloxyfop and were evidently triacylglycerols because they were hydrolysed by lipase to produce haloxyfop.

Laying hens eliminated 82-90% of the dose as intact haloxyfop in their excreta. Radioactive residues in tissues were 0.12, 0.99, 1.8 and 4.2 mg/kg haloxyfop equivalent in the muscle, body fat, liver and kidneys respectively 24 hours after the final administration. Polar and non-polar conjugates were found as major metabolites in the tissues. The nature of metabolites in the body fat and egg yolk was same as in goat body and milk fat. They were easily hydrolysed by lipase to yield haloxyfop.

In conclusion, the metabolism of haloxyfop in poultry is similar to that in ruminants, which is essentially the same as in the other mammalian species studied: rats, mice, dogs, monkeys and humans.

These studies show that the definition of the residue for products of animal origin should be the same as for plant products.

Residue evaluations

Peas (pods and succulent seeds). Four supervised trials were carried out with racemic haloxyfop-etotyl in France at application rates of 0.1 or 0.21 kg ai/ha. There was no information on French GAP but conditions in three of the trials were according to Spanish Gap for legumes (0.1-0.21 kg ai/ha applied after weed emergence at 2-4-leaf stage). In the other trial application was at the early budding stage, which is not recommended practice. The residues in the trials according to GAP were ≤0.05-0.11 mg/kg.

Three supervised trials were carried out in France and four in Germany with applications of haloxyfop-R-methyl at 0.052-0.1 kg ai/ha. No information on GAP was available from these countries, but the trial conditions complied with the GAP of some East European countries (0.052-0.13 kg ai/ha, 60-day PHI, or 0.052-0.16 kg ai/ha, up to closing of canopy). The residues were <0.02-0.07 mg/kg. The trials in which the application rates were more than 30% below the maximum registered rate were not used in estimating the STMR.

Two French trials with racemic haloxyfop were according to maximum Spanish GAP; the residues were 0.06 and 0.11 mg/kg. Three German trials with 0.1 kg ai/ha of haloxyfop-R and 53-60 days PHI were comparable with maximum GAP in Poland (0.13 kg ai/ha, 60-day PHI). The residues in all three were <0.02 mg/kg.

The residues from the 5 trials in rank order were <0.02 (3), 0.06 and 0.11 mg/kg.

The Meeting estimated a maximum residue level 0.2 mg/kg and an STMR of 0.02 mg/kg for haloxyfop in peas (pods and succulent seeds).

Pea hay or fodder (dry). Six supervised trials on field peas in France and four in Germany complied with French GAP for fodder peas (0.052-0.1 kg ai/ha of haloxyfop-R applied up to early tillering); the residues were <0.02-0.1 mg/kg. The same data were evaluated by the 1995 Meeting in estimating a maximum residue level for dry pulses. The residues in the peas under the use pattern for fodder peas were below the maximum residue level estimated for dry pulses (0.2 mg/kg). Data were submitted to the 1995 Meeting on residues in pea haulms from four supervised trials in Germany and in whole plants of pigeon peas from two trials in Australia. However as the moisture content of the samples was not known the residues could not be referred to a dry weight basis, as required for residues in animal feeds (see report of 1980 JMPR, Section 2.8).

The Meeting could not estimate a maximum residue level for pea hay or fodder (dry).

Estimation of STMR and STMR-P levels for commodities for which maximum residue levels were estimated at the 1995 JMPR

The residue definition for the estimation of STMR or STMR-P levels should be the same as for the estimation of maximum residue levels (haloxyfop esters, haloxyfop and its conjugates expressed as haloxyfop), because no other metabolites were found in plant or animal metabolic studies.

Orchard crops. Haloxyfop is used in orchards to control grass weeds. Since the application is directed at the weeds growing at the base of the trees, residues in fruits will be caused only by drift contamination or translocation after the uptake of residues from soil by the roots. The Meeting therefore concluded that orchard crops should be evaluated as a single group.

Although bananas are not an orchard crop, they can be regarded as orchard crops for evaluation since the purpose and method of application is the same.

Eight supervised trials on citrus fruits, three on apples, nine on grapes and two on bananas were carried out in Australia, Brazil, France, Italy and New Zealand. The residues were below the limit of determination (<0.01- <0.1 mg/kg) in all the trials, except one in Australia on grapes which showed 0.03 mg/kg, even in trials carried out at excessive application rates.

The Meeting estimated a nil residue for orchard crops and bananas taking into consideration the use pattern and the fact that practically no uptake of residues from soil was observed.

The Meeting estimated an STMR of 0 mg/kg for haloxyfop in citrus fruits, apples, grapes and bananas.

Pulses (dry). Haloxyfop is registered for use on several pulses. The Meeting concluded that the supervised trials on pulses could be evaluated together because of the similarities in the use patterns and residue behaviour.

Broad bean (dry). Conditions in two supervised trials (0.1 and 0.16 kg ai/ha, 103 and 171-day PHIs respectively) were comparable with maximum Australian GAP (0.1 kg ai/ha, 147-day PHI); the residues in both were <0.05 mg/kg. One trial in France was according to maximum Spanish GAP (0.21 kg ai/ha, with weeds at 2-4-leaf stage) and the residue was 0.03 mg/kg.

Chick-pea (dry). Three supervised trials in Australia (0.1 kg ai/ha, 78-99 days PHI) approximated maximum Australian GAP (0.1 kg ai/ha, 98-day PHI). The residues were <0.03, 0.03 and 0.04 mg/kg.

Common bean (dry). One Australian trial (0.16 kg ai/ha, 75-day PHI, 6-leaf stage) was comparable with maximum Australian GAP (0.16 kg ai/ha, 91-day PHI). The residue was 0.03 mg/kg. The Meeting considered that conditions in another Australian trial with applications at early budding were unpractical.

Field pea (dry). Two Australian supervised trials (0.21 kg ai/ha, 93-94-day PHI) could be used in the estimation of an STMR although the doses were higher than the maximum Australian rate (0.16 kg ai/ha, 91-day PHI) because the residues were below the limit of determination of 0.01 mg/kg.

Six supervised trials were carried out with racemic haloxyfop in France according to maximum Spanish GAP (0.21 kg ai/ha, with weeds at 2-4-leaf stage). The residues were <0.02 (3), <0.05, 0.06 and 0.14 mg/kg.

Two supervised trials in Australia with haloxyfop-R (0.1 kg ai/ha, 93-94-day PHI) were comparable with maximum Australian GAP (0.078 kg ai/ha, 91-day PHI). The residues in both were <0.01 mg/kg.

One trial in France (0.1 kg ai/ha, 68-day PHI) and one in Germany (0.1 kg ai/ha, 60-day PHI) were comparable with maximum GAP in East European countries (0.13 kg ai/ha, 60-day PHI or 0.16 kg ai/ha, up to closing of canopy). The residues were 0.06 and 0.03 mg/kg.

Lupin (dry). Four Australian supervised trials with racemic haloxyfop-etotyl at 0.078-0.12 kg ai/ha, with 92-115 days PHI were comparable with maximum Australian GAP (0.1 kg ai/ha, 119-day PHI). The residues were <0.05 (2), 0.03 and 0.11 mg/kg. One supervised trial with haloxyfop-R-methyl in Australia at 0.052 kg ai/ha, 92-day PHI, approximated maximum Australian GAP (0.052 kg ai/ha, 119-day PHI). The residue was 0.05 mg/kg.

Soya bean (dry). Three supervised trials (0.16 kg ai/ha, 102-122-day PHI) with racemic haloxyfop in Australia complied with maximum GAP (0.16 kg ai/ha, 119-day PHI). The residues were all <0.03 mg/kg.

Five supervised trials (0.12 kg ai/ha, 97-110-day PHI) in Brazil with racemic haloxyfop were according to maximum GAP conditions (0.12 kg ai/ha, 98-day PHI). The residues were <0.05 (4) and 0.06 mg/kg.

Four French and three Italian trials with haloxyfop-R at 0.1 kg ai/ha with PHIs of 76-133 days were according to maximum French GAP (0.1 kg ai/ha, up to early tillering). The residues were <0.02 (5), 0.07 and 0.09 mg/kg.

The haloxyfop residues in pulses from the 39 trials in rank order were <0.01 (4), <0.02 (8), <0.03 (4), 0.03 (5), 0.04, <0.05 (9), 0.05, 0.06 (3), 0.07, 0.09, 0.11 and 0.14 mg/kg. The Meeting estimated an STMR of 0.03 mg/kg for haloxyfop in pulses (dry).

Potatoes. Nineteen supervised trials were carried out with racemic haloxyfop in Belgium, Germany, The Netherlands, Norway, Sweden and the UK according to maximum Irish GAP (0.21 kg ai/ha, application up to 60 cm height of crop).

The residues from the 19 trials in rank order were <0.01 (3), 0.01, 0.02, 0.03 (3), 0.04 (5), 0.05, 0.06, 0.07 (3) and 0.1 mg/kg. The Meeting estimated an STMR of 0.04 mg/kg for haloxyfop in potatoes.

Sugar beet, fodder beet and sugar beet leaves or tops

The Meeting concluded that supervised trials on sugar beet and fodder beet could be evaluated together because the use pattern of haloxyfop on these crops is the same and the residue behaviour is expected to be similar.

Sugar beet. Twelve supervised trials were carried out in France and 13 in the UK with racemic haloxyfop according to maximum French GAP (0.21 kg ai/ha, up to early weed tillering). In the French trials the residues in the roots were <0.01, <0.02 (3), 0.02, <0.03 (3), 0.03 (2), 0.05 and 0.1 mg/kg. In the UK trials the residues in the roots were <0.01(3), 0.01 (2), 0.02 (2), <0.03, 0.03 (2), 0.05 (2) and 0.23 mg/kg and in the leaves or tops <0.02 (3), 0.02, <0.03 (3), 0.03, 0.04 (2), 0.09, 0.11 and 0.28 mg/kg.

Eight supervised trials were carried out with racemic haloxyfop in Germany according to maximum GAP (0.21 kg ai/ha, 90-day PHI). The residues in the roots were <0.005, 0.01 (3), 0.02, 0.04, 0.14 and 0.16 mg/kg and in the leaves or tops <0.01, <0.02 (2), 0.03, 0.04, 0.08, 0.28 and 0.3 mg/kg.

In seven supervised trials with haloxyfop-R in France, Germany and Italy according to maximum French GAP (0.1 kg ai/ha, up to early weed tillering) the residues in the roots were 0.01, <0.02 (3), 0.02, 0.03 and 0.06 mg/kg. Residues in the leaves or tops in four of the trials were <0.02, 0.09 (2) and 0.14 mg/kg.

Fodder beet. In five supervised trials with racemic haloxyfop in Germany according to maximum GAP (0.21 kg ai/ha, 90-day PHI) the residues in the roots were <0.01 (2), 0.01, 0.03 and 0.04 mg/kg and in the leaves or tops <0.02 (3), 0.03 and 0.05 mg/kg.

The residues in the roots from the 45 trials in rank order were <0.005, <0.01 (6), 0.01 (6), <0.02 (6), 0.02 (5), <0.03 (4), 0.03 (6), 0.04 (2), 0.05 (3), 0.06, 0.1, 0.14, 0.16 and 0.23 mg/kg.

The residues in the leaves or tops from 30 of the trials in rank order were <0.01, <0.02 (9),

0.02, <0.03 (3), 0.03 (3), 0.04 (3), 0.05, 0.08, 0.09 (3), 0.11, 0.14, 0.28 (2) and 0.3 mg/kg. However, because the Meeting did not estimate a maximum residue level for the leaves or tops, no STMR was estimated.

The Meeting estimated an STMR of 0.02 mg/kg for haloxyfop in sugar beet and fodder beet.

Rice. In nine supervised trials on rice with racemic haloxyfop in Brazil, Colombia, Mexico and Costa Rica the application rates were comparable with the maximum rates in some South American countries of 0.11 kg ai/ha. All the residues were <0.01 mg/kg.

The Meeting estimated an STMR of 0 mg/kg for haloxyfop in husked and polished rice taking into consideration that no residue was found even in trials carried out at an excessive dose rate.

Cotton seed. Four supervised trials with racemic haloxyfop in Australia according to maximum Australian GAP (0.16 kg ai/ha, 119-day PHI). The residues were <0.05 (2), 0.06 and 0.08 mg/kg. Conditions in four supervised trials carried out with racemic haloxyfop in Brazil (0.24 kg ai/ha applied 22-40 days after planting, 93-112-day PHI) were comparable with maximum GAP in Paraguay (0.18 kg ai/ha, applied with weeds at 2-4 leaf stage). The residues were <0.1 (2), 0.1 and 0.2 mg/kg.

The residues from the 8 trials in rank order were <0.05 (2), 0.06, 0.08, <0.1 (2), 0.1 and 0.2 mg/kg. The Meeting estimated an STMR of 0.09 mg/kg for haloxyfop in cotton seed.

Peanuts. Two trials with racemic haloxyfop in Argentina were at dose rates of 0.24 and 0.4 kg ai/ha, comparable to maximum GAP (0.3 kg ai/ha, weeds at 2-4-leaf stage). The residues were 0.03 and <0.05 mg/kg.

Three supervised trials with racemic haloxyfop in Australia were at 0.12-0.16 kg ai/ha, 98-117 days PHI, similar to maximum GAP (0.16 kg ai/ha, 119 days PHI). The residues were <0.03 and 0.03 (2) mg/kg.

The residues from the 5 trials were <0.03, 0.03 (3) and <0.05 mg/kg.

The Meeting estimated an STMR of 0.03 mg/kg for haloxyfop in peanuts.

Rape seed and rape fodder. Two supervised trials with racemic haloxyfop in Australia at a rate of 0.16 kg ai/ha, slightly higher than maximum GAP (0.1 kg ai/ha, 119-day PHI). In this case the influence of the dose rate on the residue of haloxyfop is assumed to be little, since the application was made at an early growth stage (2-6 leaves) causing less direct exposure of the crops to haloxyfop. The Meeting therefore concluded that the trials were comparable with GAP. The residues in the rape seed were <0.03 and 0.07 mg/kg.

Thirteen supervised trials were carried out with racemic haloxyfop in France according to maximum French GAP (0.21 kg ai/ha, up to early tillering). The residues in the rape seed were <0.05 (7), 0.05, 0.09, 0.14, 0.145, 0.37 and 0.66 mg/kg. Seven of the French trials also included treatments comparable with maximum Spanish GAP (0.42 kg ai/ha, applied with weeds at 2-4 leaf stage). The residues in the seed were <0.05, 0.05 (2), 0.17, 0.315, 0.32 and 1.68 mg/kg.

Six supervised trials with racemic haloxyfop in Germany were according to maximum GAP (0.21 kg ai/ha, post weed emergence). The residues were <0.05, 0.1, 0.13 (2), 0.15 and 0.77 mg/kg in

the seed and <0.05 (2), 0.09 and 0.12 mg/kg in four samples of fodder.

Eighteen supervised trials (three trials in 1984 were counted as two trials each, because the applications were made in the year before harvest or the year of harvest) with racemic haloxyfop in the UK were according to maximum French or German GAP (0.21 kg ai/ha, up to early tillering or post weed emergence). The residues were <0.05 (11), 0.05, 0.06, 0.09, 0.1, 0.11, 0.44 and 0.64 mg/kg in the seed and <0.05 (9), 0.05, 0.07 and 0.08 mg/kg in twelve samples of fodder.

Three supervised trials with haloxyfop-R in France and two in Germany which complied with maximum French GAP (0.1 kg ai/ha, up to early tillering) showed residues in the seed of <0.05 (4) and 0.07 mg/kg. The residues in rape fodder in the German trials were both <0.05 mg/kg.

The residues in the rape seed from the 51 trials in rank order were <0.03, <0.05 (24), 0.05 (4), 0.06, 0.07 (2), 0.09 (2), 0.1 (2), 0.11, 0.13 (2), 0.14, 0.145, 0.15, 0.17, 0.315, 0.32, 0.37, 0.44, 0.64, 0.66, 0.77 and 1.68 mg/kg. However the residues in the seven French trials in which application was at the maximum Spanish GAP rate (0.42 kg ai/ha) seem to be from a different population from the others. The Meeting concluded that an STMR for haloxyfop in rape seed should be estimated form this higher population. The residues in rape seed from these 7 trials in rank order were <0.05, 0.05 (2), 0.17, 0.315, 0.32 and 1.68 mg/kg.

The residues in rape fodder from 18 trials in rank order were <0.05 (13), 0.05, 0.07, 0.08, 0.09 and 0.12 mg/kg. The Meeting estimated an STMR of 0.17 mg/kg for haloxyfop in rape seed.

Sunflower seed. Eight supervised trials were carried out with racemic haloxyfop in Argentina, Australia and France at the relevant maximum GAP (rates of 0.3, 0.16 and 0.21 kg ai/ha respectively). The residues were <0.03 (2), 0.03, 0.04, <0.05 (2), 0.143 and 0.16 mg/kg.

One supervised trial with haloxyfop-R in France at the maximum GAP rate of 0.1 kg ai/ha gave a residue of 0.07 mg/kg.

The residues from the 9 trials in rank order were <0.03 (2), 0.03, 0.04, <0.05 (2), 0.07, 0.143 and 0.16 mg/kg. The Meeting estimated an STMR of 0.05 mg/kg for haloxyfop in sunflower seed.

Alfalfa. In two supervised trials with racemic haloxyfop in Australia the conditions (0.21 kg ai/ha, 21-22 days PHI) were comparable with maximum GAP (0.16 kg ai/ha, 21-day PHI). The residues were 2.45 and 3.11 mg/kg. In two further trials with haloxyfop-R in Australia the conditions (0.1 kg ai/ha, 22-day PHI) were again comparable with maximum GAP (0.078 kg ai/ha, 21-day PHI) and the residues were 1.8 and 2.21 mg/kg.

The residues from the 4 trials in rank order were 1.8, 2.21, 2.45 and 3.11 mg/kg.

Pasture. Four supervised trials with racemic haloxyfop and two with haloxyfop-R in Australia were according to maximum GAP (0.1 kg ai/ha racemic haloxyfop, 0.052 kg ai/ha haloxyfop-R, 7-day PHI in both cases). The residues from the 6 trials in rank order were 0.49, 0.99, 1.47, 1.71, 2.04 and 3.35 mg/kg.

Processing

Sugar beet. Two processing studies were carried out and no residues of haloxyfop (<0.01 mg/kg)

were found in sugar derived from sugar beet containing 0.07 and 0.11 mg/kg.

The Meeting estimated an STMR-P of 0.002 mg/kg for haloxyfop in sugar.

The concentration factors for pressed pulp were 0.36 and 0.43. The Meeting estimated an STMR-P of 0.008 mg/kg for haloxyfop in pressed pulp by applying the mean concentration factor (0.4) to the sugar beet STMR of 0.02 mg/kg.

Soya beans. Concentration factors from 4 trials were 0.75, 1.19, 1.25 and 1.31 for meal, 0.375, 0.41, 0.79 and 1.25 for crude oil and 0.33, 0.375, 0.75 and 1.22 for refined oil, giving mean factors of 1.13, 0.71 and 0.67 respectively. The Meeting estimated STMR-P levels of 0.03, 0.02 and 0.02 mg/kg for meal, crude oil and refined oil respectively by calculation from the STMR for pulses (0.03 mg/kg).

Rice. The residues in rice bran from normally treated rice were <0.02 mg/kg, and the Meeting estimated an STMR-P of 0.02 mg/kg for rice bran, unprocessed.

Cotton seed. Concentration factors for crude oil from 3 trials were 0.88, 1.0 and 1.6, giving a mean of 1.16. The Meeting estimated an STMR-P of 0.10 mg/kg for crude oil from the STMR for cotton seed of 0.09 mg/kg.

Rape seed. Concentration factors from 4 trials were 0.72, 0.89, 0.92 and 0.93 for meal or cake and 1.43, 1.97, 2.34 and 2.79 for crude oil (residues in pressed oil were not used for calculation of the concentration factors because the process is not current commercial practice). The factors for refined oil were 1.07 and 2.19. The Meeting estimated STMR-P levels of 0.15, 0.36 and 0.28 mg/kg for meal, crude oil and refined oil from the mean concentration factors of 0.87, 2.13 and 1.63 respectively and the STMR for rape seed of 0.17 mg/kg.

Note - Correction to report of 1995 JMPR

The concentration factors of 1.7 for crude oil and 2.1 for refined oil should be replaced by 2.13 for crude and 1.63 for refined oil.

Products of animal origin

Cattle. The Meeting was aware that the dosing levels in the feeding studies evaluated by the 1995 JMPR were expressed on a dry-weight basis, whereas the provisional maximum residue levels for the feed items were estimated on a wet-weight basis. The Meeting therefore reconsidered the conclusions of the 1995 Meeting with respect to residues in cattle products.

Fodder beet, alfalfa, pasture, sugar beet tops, pulses, rape fodder and processed fractions of oil seed and sugar beet can be used as feed for beef and dairy cattle, but the maximum haloxyfop intake would result from consuming 100% of pasture. The maximum residue found in pasture was 3.35 mg/kg (1995 Residue Evaluations, p.488), and with an assumed 80% moisture content this would be equivalent to 16.75 ppm in the feed on a dry-weight basis.

Since this feed level is higher than the highest level in the feeding studies (beef calves 10 ppm; lactating cows 2.5 ppm), the Meeting could not confirm the maximum residue levels for cattle products that were estimated by the 1995 JMPR and agreed to withdraw the provisional estimates for these commodities.

Poultry. Pulses and processed fractions of pulses and oil seed can be used as feed for poultry. Cereals are the main feed items, but the feed could contain up to 50% of pulses, 7% of rape seed meal and 30% of soya bean meal, and this composition would provide the maximum haloxyfop intake. The median intake level for this feed composition was calculated from the STMR for each feed item (pulses 0.03 mg/kg, rape seed meal 0.15 mg/kg and soya bean meal 0.03 mg/kg) to be 0.035 ppm (dry weight basis).

The residues in the muscle, liver, fat and eggs at a feeding level of 0.035 ppm were estimated from control residues (<0.01 mg/kg in each product) and the highest residues found in each product in the feeding study at 0.25 ppm by extrapolation to be <0.01, 0.01, 0.01 and <0.01 mg/kg respectively.

The Meeting confirmed the 1995 estimates of maximum residue levels in poultry products and estimated an STMR of 0.01 mg/kg for haloxyfop in chicken meat, chicken edible offal and chicken eggs.

Residues in rotational crops

Comprehensive studies were conducted with six rotational crops, using labelled or unlabelled haloxyfop. When lettuce, sugar beet and wheat were planted as rotational crops 25-148 days after treating soya beans or cotton as primary crops with unlabelled haloxyfop at a rate of 0.28 or 0.56 kg ai/ha, no residues were found in any of the mature rotational crops except green wheat forage at the LOD of 0.01 mg/kg, 110 days after treatment with 0.28 kg ai/ha. The limit of determination was 0.01 mg/kg for all substrates except wheat straw, for which it was 0.02 mg/kg.

When lettuce, wheat, soya beans, carrots or turnips were grown to maturity in soil which had been treated with phenyl-ring-labelled haloxyfop at 0.56 kg ai/ha 30 days before planting, the highest radioactive residues in the edible portions were found in lettuce and wheat grain and were 0.01 mg/kg haloxyfop equivalent. The radioactivity was too low for identification of the residue. 130 days after treatment of soya bean plants with phenyl-ring-labelled haloxyfop, the top 5 cm of soil was transferred to pots and sown with lettuce, soya bean, wheat and sugar beet in the laboratory. The total radioactivity was 0.01, 0.04, 0.02 and 0.01 mg/kg haloxyfop equivalent in lettuce, soya bean, wheat and sugar beet respectively and 0.02, 0.05, 0.02 and 0.02 mg/kg in soya bean forage, soya bean straw, wheat straw and sugar beet forage respectively. Again, the residue could not be identified owing to the low level of radioactivity.

The pyridinol 3-chloro-5-trifluoromethylpyridin-2-ol was found in soil as a major terminal degradation product under aerobic conditions (1995 Residue Evaluations, p.415) but it was not detected in the plants at harvest in any of the plant metabolism studies, although these included experiments with pyridinol-labelled haloxyfop.

The submitted data indicated that haloxyfop and its soil degradation products would not be absorbed or accumulate in plants to any significant extent.

The Meeting noted that the residues found in supervised trials on fodder crops reviewed by the 1995 JMPR were expressed on a wet-weight basis, although the Codex Classification of Food and Feeds indicates that MRLs for fodder and forage should preferably be set and expressed on a dry-weight basis. As the Meeting did not have information on the moisture content of the fodder crops for which the 1995 JMPR estimated provisional maximum residue levels, it agreed to withdraw the provisional estimates for fodder crops.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels, STMR levels and STMR-P levels listed in the Tables below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue, for compliance with MRLs and for estimation of dietary intake: haloxyfop esters, haloxyfop and its conjugates expressed as haloxyfop.

Commodity		Recommended MRL, mg/kg		PHI, days	Estimated STMR, for dietary intake estimation, mg/kg
CCN	Name	New	Previous max. residue level[1]		
AL 1021	Alfalfa forage (green)	W	5	21	
FI 0327	Banana	0.05*	0.05*	-	0
FC 0001	Citrus fruits	0.05*	0.05*	-	0
SO 0691	Cotton seed	0.2	0.2	-	0.09
OC 0691	Cotton seed oil, crude	0.5	0.5	-	0.1 (STMR-P)
AM 1051	Fodder beet	0.3	0.3	90	0.02
AV 1051	Fodder beet leaves or tops	W	0.3	90	
FB 0269	Grapes	0.05*	0.05*	-	0
SO 0697	Peanut	0.05	0.05	-	0.03
VP 0063	Peas (pods and succulent seeds)	0.2	-	-	0.02
FP 0009	Pome fruits	0.05*	0.05*	-	0
VD 0070	Pulses (dry)	0.2	0.2	-	0.03
VR 0589	Potato	0.1	0.1	-	0.04
SO 0495	Rape seed	2	2	-	0.17
OC 0495	Rape seed oil, crude	5	5	-	0.36 (STMR-P)
OR 0495	Rape seed oil, edible	5	5	-	0.28 (STMR-P)
CM 1206	Rice bran, unprocessed	0.02*	0.02*	-	0.02 (STMR-P)
CM 0649	Rice, husked	0.02*	0.02*	-	0
CM 1205	Rice, polished	0.02*	0.02*	-	0
OC 0541	Soya bean oil, crude	0.2	0.2	-	0.02 (STMR-P)
OR 0541	Soya bean oil, refined	0.2	0.2	-	0.02 (STMR-P)
VR 0596	Sugar beet	0.3	0.3	-	0.02
AV 0596	Sugar beet leaves or tops	W	0.3	-	
SO 0702	Sunflower seed	0.2	0.2	-	0.05
MM 0812	Cattle meat	W	0.01	-	
MO 0812	Cattle, Edible offal of	W	0.5	-	
MF 0812	Cattle fat	W	0.1	-	

Commodity		Recommended MRL, mg/kg		PHI, days	Estimated STMR, for dietary intake estimation, mg/kg
CCN	Name	New	Previous max. residue level[1]		
ML 0812	Cattle milk	W	0.05	-	
FM 0812	Cattle milk fat	W	0.5	-	
PM 0840	Chicken meat	0.01*	0.01*	-	0.01
PO 0840	Chicken, Edible offal of	0.1	0.1	-	0.01
PE 0840	Chicken eggs	0.01*	0.01*	-	0.01

[1] Provisional estimates of maximum residue levels made by the 1995 JMPR but not recommended for use as MRLs.

Raw agricultural commodity	STMR, mg/kg	Processed commodity	STMR-P, mg/kg
Sugar beet	0.02	Refined sugar	0.002
		Sugar beet pressed pulp	0.008
Rape seed	0.17	Rape seed meal	0.15
Soya bean	0.03 (Pulses (dry))	Soya bean meal	0.03

FURTHER WORK ON INFORMATION

Desirable

1. Information on the moisture content of fodder crops.

2. Ruminant feeding studies at a feeding level comparable to the maximum residue level found in fodder crops.

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(All unpublished)

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