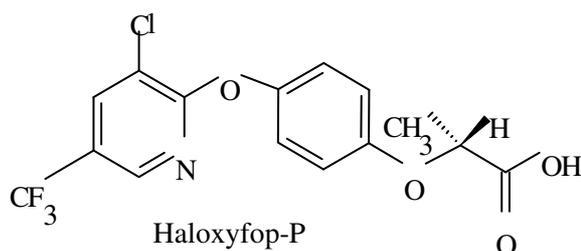


### 5.13 HALOXYFOP (194) AND HALOXYFOP-P

#### RESIDUE AND ANALYTICAL ASPECTS

Residue and analytical aspects of haloxifop 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 haloxifop, haloxifop-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.



Haloxifop 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 haloxifop-P. The ISO name for the unresolved isomeric mixture is haloxifop.

#### *Animal metabolism*

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

When two lactating goats were dosed with phenyl ring labelled haloxifop via gelatin capsule twice daily for 10 consecutive days at the equivalent of 16 ppm haloxifop 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 [ $^{14}\text{C}$ ] volatiles or  $^{14}\text{CO}_2$ .

Radiolabel, expressed as haloxifop, 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 haloxifop, but some may have been present as labile conjugates.

Radiolabel levels in milk were 0.25 and 0.20 mg/kg. The  $^{14}\text{C}$  residues in milk fat were nonpolar and were susceptible to alkaline hydrolysis or lipase hydrolysis releasing haloxifop. The behaviour was consistent with haloxifop 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 haloxifop via gelatin capsule for 11 consecutive days at the equivalent of 12 ppm haloxifop 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 haloxifop, 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, haloxifop. Most likely, parent haloxifop 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 7<sup>th</sup> day of dosing. Almost the entire residue in the yolks was present as triacylglycerides. Mild alkaline hydrolysis or lipase hydrolysis produced haloxifop as the single product.

In summary, the metabolism of haloxifop 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. Haloxifop is readily conjugated and incorporated into fats or secreted in the lipid of milk or eggs. The intact haloxifop may be released from its conjugates by mild alkaline or enzymatic hydrolysis.

### *Plant metabolism*

The Meeting received plant metabolism studies with haloxifop-butyl in cotton; haloxifop-methyl, haloxifop-butyl and haloxifop-ethoxyethyl in soya beans; and haloxifop-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 haloxifop 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 haloxifop, 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 haloxifop butyl ester. In the cotton seed, almost all of the <sup>14</sup>C was accounted for as haloxifop free acid (32% at day 105) and haloxifop conjugates (66% at day 105). In the oil, 100% of the <sup>14</sup>C was present as haloxifop conjugates. In the field trash, the radiolabel was present as free acid (39%) and conjugates (55%).

The <sup>14</sup>C in the oil was associated with the triglycerides. Lipase hydrolysis and alkali hydrolysis released 91–99.8% of the <sup>14</sup>C as haloxifop, suggesting that the non-polar residues were triglyceride esters of haloxifop.

In a soya bean metabolism study, the mature second and developing third trifoliolate leaves of 20 day old soya bean plants were treated with [<sup>14</sup>C]labelled haloxifop 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 haloxifop-methyl phenyl label and pyridyl label, suggesting that the haloxifop 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 haloxifop 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 haloxifop accounted for 35–39% and 61–65% of the label respectively in the untreated parts of the plant, i.e., unconjugated haloxifop was the major component of the residue. Mild alkaline hydrolysis of the polar translocated residue released haloxifop, demonstrating that at least part of the polar fraction consisted of haloxifop conjugates.

In a second soya bean metabolism study, plants were treated once with [<sup>14</sup>C]haloxifop-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 haloxifop showed that the haloxifop molecule remained intact and essentially the entire residue contained both the phenyl and pyridyl rings.

Radiolabelled residue levels, expressed as haloxifop, 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 haloxifop 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 haloxifop was incorporated into the oil triglycerides. Most of the polar conjugates also produced haloxifop on hydrolysis.

Polar conjugates (65–66%) were the main component of the residues in treated soya bean forage 15 days after treatment, with unconjugated haloxifop (27–30%) accounting for most of the remainder. In new-growth forage sampled at the same time, unconjugated haloxifop and polar conjugates accounted for 42% and 57% of the residue respectively. In soya bean straw, unconjugated haloxifop 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 haloxifop-P-methyl formulated as an EC. At maturity, 92 days after application,  $^{14}\text{C}$  residues expressed as haloxifop-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% haloxifop-P acid, 19% conjugate 1, 20% haloxifop-P glycoside conjugate 1 and 20% unextracted. The composition of the residue in sugar beet shoots at maturity was: 33% haloxifop-P acid, 24% conjugate 1, 14% haloxifop-P glycoside conjugate 1 and 12% unextracted.

In summary, haloxifop-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 haloxifop-P-methyl formulated as an EC. By day 14, haloxifop-P-methyl had disappeared and haloxifop-P acid was the major component of the residue. At maturity, 29 days after treatment,  $^{14}\text{C}$  residues expressed as haloxifop-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 haloxifop-P acid at 93% of the  $^{14}\text{C}$ , with 5.4% accounted for by various conjugates. The  $^{14}\text{C}$  residue in lettuce outer leaves consisted of: 38% haloxifop-P acid, 24% conjugate 1, 23% glycoside conjugate 2, 10% unextracted and 6.9% glycoside conjugate 1.

Summary of haloxifop in plant metabolism—when applied to a plant, the esters of haloxifop or haloxifop-P are broken down quickly to release free acid which is readily translocated throughout the plant. The haloxifop (or haloxifop-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.

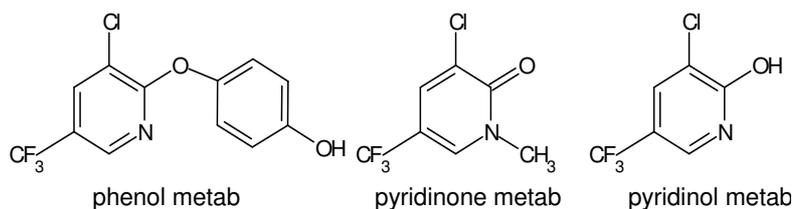
### *Environmental fate in soil*

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

Haloxifop residues are generally not persistent in soils. Haloxifop 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 haloxfop-P-methyl disappeared with a half-life of approximately 0.5 days. Haloxfop-P-methyl was hydrolysed just as quickly in a sterile soil as in a fresh soil, demonstrating that the methyl ester is chemically labile. Haloxfop-P acid was persistent in the sterile soil.

Under aerobic soil incubation, the first metabolite was haloxfop-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 (haloxfop-P-methyl labelled in the pyridyl ring) had been mineralized and 28–46% was unextracted.



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 haloxfop-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 [<sup>14</sup>C]phenyl ring labelled haloxfop-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 <sup>14</sup>C contents of the plant tissues, expressed as haloxfop 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 haloxfop-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. Haloxfop 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 haloxfop in soil metabolism—haloxfop 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 haloxfop residues compared with soil metabolism. Haloxfop 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 haloxfop 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.

Haloxifop residue methods rely on an initial extraction and hydrolysis step, usually with methanolic NaOH to release haloxifop from conjugates. After solvent partition cleanup, the haloxifop is methylated or butylated ready for GC analysis or further cleanup before the GC analysis. Typically, haloxifop 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 haloxifop 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  $^{14}\text{C}$  from the soya beans. HPLC produced a single peak matching haloxifop which accounted for 95% of the  $^{14}\text{C}$  in the extract.

The completeness of extraction of haloxifop, esters and conjugates from goat milk was tested on a sample from a goat dosed with [ $^{14}\text{C}$ ]haloxifop-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  $^{14}\text{C}$  (91%) was extracted and released as haloxifop 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 haloxifop ester such as haloxifop-ethoxyethyl as the spiked analyte, which does check that the extraction conditions quantitatively hydrolyse the spiked ester. Haloxifop 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 haloxifop enantiomers. The methods effectively measure 'total' haloxifop present as acid, salts, esters and conjugates (esters with natural compounds).

Haloxifop residues are not suitable for analysis by multi-residue methods because the extraction step is typically also a base-hydrolysis step designed to release haloxifop 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 haloxifop measure haloxifop present as acid, salts, esters and conjugates, so changes among these different forms during storage would not be detected.

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

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

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

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

Haloxifop 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 haloxifop residues in animal commodities, but from haloxifop 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 haloxifop is: Haloxifop esters, haloxifop and its conjugates expressed as haloxifop.

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

- Goat metabolism study: the  $^{14}\text{C}$  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  $^{14}\text{C}$  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 haloxifop 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 haloxifop 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 haloxifop 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 haloxifop-P.

The Meeting recommended a revised residue definition for haloxifop.

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

The residue is fat-soluble.

### ***Results of supervised trials on crops***

The Meeting received information on the use patterns and labels for haloxifop-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 haloxifop-P-methyl, haloxifop-methyl and haloxifop-ethoxyethyl.

Current GAP relies on haloxifop-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 haloxifop maximum residue level recommendation of 0.1 mg/kg for potatoes.

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

Haloxifop 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 haloxifop 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.

Haloxifop-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 haloxifop-ethoxyethyl (0.42 and 0.83 kg ai/ha, PHI 28 days), haloxifop residues were below LOQ (0.05 mg/kg).

In Uruguay, haloxifop-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), haloxifop-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. Haloxifop residues were all below LOQ (0.1 mg/kg).

Haloxifop-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 haloxifop-ethoxyethyl (0.21 and 0.42 kg ai/ha, PHI 29 days) and six trials on lemons also with haloxifop-ethoxyethyl (0.21–0.83 kg ai/ha, PHI 28 days), haloxifop residues were all below LOQ (0.05 mg/kg).

The Syrian label allows the use of haloxifop-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), haloxifop residues were below LOQ (0.02 mg/kg).

Residues in fruits are not expected with this directed use on the weeds because haloxifop 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.

Haloxifop-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 haloxifop-ethoxyethyl (0.42 and 0.83 kg ai/ha, PHI 24 days), haloxifop residues were below LOQ (0.05 mg/kg).

The Syrian label allows the use of haloxifop-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 haloxifop-ethoxyethyl (0.16 kg ai/ha, PHI 126–132 days), haloxifop residues were below LOQ (0.02 mg/kg).

Haloxifop-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 haloxifop-ethoxyethyl (0.21 kg ai/ha, PHI 29 days), haloxifop residues were below LOQ (0.01 mg/kg).

In eight US trials on apples with directed applications of haloxifop-methyl (0.28 and 0.56 kg ai/ha, PHI 59–60 days), haloxifop 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.

Haloxifop-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 haloxifop-ethoxyethyl (0.42 and 0.83 kg ai/ha, PHI 24 days), haloxifop 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.

Haloxifop-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 haloxifop-ethoxyethyl (0.21, 0.42 and 0.83 kg ai/ha, PHI 21 and 29 days), haloxifop residues were below LOQ (0.05 mg/kg).

The Swiss label allows the use of haloxifop-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 haloxifop-ethoxyethyl (0.10, 0.21, 0.42, 0.83 and 1.7 kg ai/ha, PHI 86-115 days), haloxifop 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.

Haloxifop-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 haloxifop-P-methyl and haloxifop-ethoxyethyl (0.42 and 0.83 kg ai/ha respectively, PHI 14 days), haloxifop 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 haloxifop-P-methyl for weed control in onions at 0.10 kg ai/ha. In two Belgian trials matching Moldovan GAP on onions, haloxifop 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, haloxifop 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 haloxifop-P-methyl for weed control in onions at 0.10 kg ai/ha. In two French trials matching Tunisian GAP on onions, haloxifop residues in the onions (whole plant) were < 0.02 and 0.03 mg/kg 28 days after treatment.

Haloxifop-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 haloxifop 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.

Haloxifop residues from the eight onion trials in rank order, median underlined were: < 0.02, 0.02, 0.03, 0.03, 0.04, 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<sup>34</sup>) 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 haloxifop-P-methyl for weed control in field beans at 0.10 kg ai/ha. In eight French trials matching Tunisian GAP on field beans, haloxifop 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, haloxifop 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, haloxifop residues in beans (whole pods) from the 10 trials in rank order (median underlined) were: < 0.02, < 0.02, 0.03, 0.06, 0.07, 0.10, 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).

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 haloxifop-P-methyl for weed control in peas at 0.10 kg ai/ha. In eight French trials matching Tunisian GAP on peas, haloxifop 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, haloxifop 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, haloxifop residues in the peas in pods were: 0.12 and 0.53 mg/kg, 28 days after treatment.

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

In summary, haloxypop 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 haloxypop-P-methyl for weed control in peas at 0.10 kg ai/ha. In nine French trials matching Tunisian GAP on peas, haloxypop 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, haloxypop 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, haloxypop residues in shelled peas were: 0.12 and 0.75 mg/kg, 28 days after treatment.

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

In summary, haloxypop 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, haloxypop-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  $\pm$  25% tolerance (0.11–0.19 kg ai/ha and PHI range 50–80 days), haloxypop 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  $\pm 25\%$  tolerance (0.11–0.19 kg ai/ha and PHI range 50–80 days), haloxifop residues in beans were: 0.01, 0.06, 0.07, 0.08, 0.08, 0.32 and 0.42 mg/kg.

In Brazil, haloxifop-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  $\pm 25\%$  tolerance (0.036–0.060 kg ai/ha and PHI range 50–80 days), haloxifop 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  $\pm 25\%$  tolerance (0.036–0.060 kg ai/ha and PHI range 61–70 days), haloxifop 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, 0.32, 0.39, 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 haloxifop 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, haloxifop-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, haloxifop 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, haloxifop 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, haloxifop-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, haloxifop 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, haloxifop residues were also all below LOQ (0.01 mg/kg). In six trials with haloxifop-ethoxyethyl at application rates of 0.10 and 0.21 kg ai/ha, but with the same timing, haloxifop residues were also below LOQ (0.01 mg/kg).

The Tunisian label allows the use of haloxifop-P-methyl for weed control in peas at 0.10 kg ai/ha. In eight French trials matching Tunisian GAP on peas, haloxifop 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 haloxifop-ethoxyethyl, haloxifop 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 haloxifop 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, 0.04, < 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, haloxifop-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), haloxifop 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), haloxifop 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, haloxifop-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), haloxifop residues in soya beans were < 0.01 (3), 0.01 and 0.06 mg/kg.

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

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

In Hungary, two trials with haloxifop-P-methyl at 0.10 kg ai/ha (compare with Moldovan GAP) produced haloxifop 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 haloxifop residues in soya beans (rank order, underlined median): < 0.01 (4), 0.02, 0.02, 0.03, 0.03, < 0.05, 0.06, 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

Haloxifop-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 haloxifop-P-methyl (compare with Belarus GAP) produced haloxifop residues in sugar beet roots of 0.03 mg/kg.

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

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

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

In summary, haloxifop 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), 0.02, 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 haloxifop-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 haloxifop residue data on cotton seed, were available from Brazil, Greece, Spain and the USA.

In Brazil, haloxifop-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, haloxifop residues in cotton seed were: < 0.01, < 0.01 and 0.08 mg/kg.

In Argentina, haloxifop-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 haloxifop-P-methyl matching Argentinean GAP ( $\pm 25\%$ ), haloxifop 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 haloxifop-methyl at 0.12 kg ai/ha, haloxifop 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, haloxifop 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, < 0.1 (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 haloxifop-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, haloxifop residues in canola grain were: 0.22 and 0.86 mg/kg.

Haloxifop-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 ( $\pm 25\%$ ) are included.

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

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

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

Summarising, 22 European trials with haloxifop-P-methyl at 0.10 kg ai/ha produced haloxifop residues in rapeseed (rank order, underlined median): < 0.01 (3), < 0.05 (7), 0.07, 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, haloxifop-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 haloxifop-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, haloxifop 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, haloxifop-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, haloxifop residues in sunflower seed were 0.14 mg/kg.

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

In three French trials with haloxifop-ethoxyethyl, but matching the Tunisian GAP application rate on sunflowers, haloxifop 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: < 0.05 (4), 0.05, 0.06, 0.07 and 0.10 mg/kg.

The Serbian label allows the use of haloxifop-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, haloxifop residues in the sunflower seed were: < 0.05, 0.05 and 0.14 mg/kg.

In three French trials with haloxifop-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, haloxifop residues in the sunflower seed were: 0.07, 0.09 and 0.16 mg/kg.

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

In three Spanish trials matching Serbian GAP on sunflowers, haloxifop 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, haloxypop-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 haloxypop-methyl at 0.18 and 0.36 kg ai/ha, haloxypop residues in coffee beans did not exceed the LOQ (0.02 mg/kg).

In 13 trials on coffee in Brazil with directed applications of haloxypop-methyl at 0.12 to 0.96 kg ai/ha, haloxypop 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, haloxypop-P-methyl is registered for weed control uses on alfalfa at 0.078 kg ai/ha. The label allows use from the second trifoliolate 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, haloxypop 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 haloxypop-ethoxyethyl matching the GAP application rate and PHI (0.078 ± 25%, 0.059–0.10 kg ai/ha, PHI 28–32 days) on alfalfa, haloxypop 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, haloxypop 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, haloxypop-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, haloxypop 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.

Haloxypop 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 haloxifop-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, haloxifop residues in peanut forage, dry weight, were: < 0.02, 0.13, 0.28 and 1.1 mg/kg. Haloxifop 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, haloxifop 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.

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

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

In Hungary, two trials with haloxifop-P-methyl at 0.10 kg ai/ha (compare with Moldovan GAP) produced haloxifop 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 haloxifop residues in soya bean forage (rank order, underlined median): < 0.05 (4), 0.10, 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.

Haloxifop-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 haloxifop-P-methyl (compare with Belarus GAP) produced haloxifop residues in sugar beet tops of 0.07 mg/kg.

In Germany, five trials with haloxifop-P-methyl at 0.10 kg ai/ha (compare with Belarus GAP) produced haloxifop 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, haloxifop 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, 0.10, 0.12, 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*

Haloxifop-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 haloxifop-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, haloxifop 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, haloxifop 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.

Haloxifop-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 ( $\pm 25\%$ ) 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 haloxifop-P-methyl application at 0.10 kg ai/ha ( $\pm 25\%$ ), haloxifop 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 haloxifop-P-methyl application at 0.10 kg ai/ha ( $\pm 25\%$ ), haloxifop 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 haloxifop-P-methyl application at 0.10 kg ai/ha ( $\pm 25\%$ ), haloxifop 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 haloxifop 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, 3.9, 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 haloxifop 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 haloxifop residues during the processing of cotton seed. The Meeting withdrew the previous recommendation of 0.5 mg/kg for a haloxifop maximum residue level in crude cotton seed oil.

A processing study was also received for apples, but haloxifop 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, haloxifop-ethoxyethyl was applied to oilseed rape 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. Haloxifop 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 °C under reduced pressure.

The processing factors for haloxifop 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 haloxifop 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 haloxifop 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, haloxifop-methyl was applied at 0.28 kg ai/ha to soya beans in bloom or haloxifop-P-methyl at 0.70 kg ai/ha was applied to soya beans at the 5<sup>th</sup> trifoliolate leaf stage. The soya beans were processed in a laboratory-scale system to produce hulls, meal, crude oil, refined oil and soapstock and haloxifop residue levels were measured on the products.

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

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

The processing factors for haloxifop 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 haloxifop-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.

Haloxifop 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 haloxifop residues for sugar beet → refined sugar were: < 0.09 and 0.15—best estimate < 0.09.

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

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

Beef calves were dosed with haloxifop 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 haloxifop residues in the muscle from the five 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 haloxifop-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 haloxifop triacylglyceride conjugates and could not be used.

Mean haloxifop 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, haloxifop 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 haloxifop 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.

Haloxifop 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 haloxifop 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 haloxifop 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 haloxifop-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 haloxifop by a method that does not include a hydrolysis step and therefore may not have recovered haloxifop residues quantitatively from triacylglyceride conjugates. Residues appeared to plateau at or before 10 days. The average concentrations of haloxifop 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 haloxifop 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 haloxifop 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. 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	

	Livestock dietary burden, haloxfop, 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 <sup>a</sup>	13.0 <sup>b</sup>
Dairy cattle	8.16	3.94	6.53	2.70	14.4 <sup>c</sup>	7.09 <sup>d</sup>
Poultry-broiler	0.11	0.11	0.11	0.11	0.29	0.29
Poultry-layer	0.11	0.11	2.40 <sup>e</sup>	1.41 <sup>f</sup>	0.29	0.29

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

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

<sup>c</sup> Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk.

<sup>d</sup> Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

<sup>e</sup> Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

<sup>f</sup> 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 haloxfop 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. 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, haloxfop, 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 <sup>a</sup>	3.14 <sup>b</sup>
Dairy cattle	0.80	0.59	3.03	1.47	7.31 <sup>c</sup>	2.41 <sup>d</sup>
Poultry—broiler	0.11	0.11	0.11	0.11	0.29	0.29
Poultry—layer	0.11	0.11	2.40 <sup>e</sup>	1.41 <sup>f</sup>	0.29	0.29

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

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

<sup>c</sup> Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk.

<sup>d</sup> Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

<sup>e</sup> Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

<sup>f</sup> 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 haloxifop 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 haloxifop 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 haloxifop 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

Dietary burden (ppm)				
Feeding level [ppm]	Eggs	Muscle + skin	Liver	Fat
STMR broilers and layers (1.41 ppm) [0.75, 2.5 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]

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.

### 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). 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.

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.

