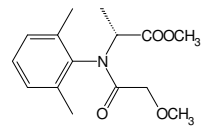
APPRAISAL

The toxicology of metalaxyl-M was evaluated by the 2002 JMPR, which established a group ADI of 0–0.08 mg/kg bw for metalaxyl and metalaxyl-M. Residue and analytical aspects were considered for the first time by the present Meeting. Metalaxyl-M is the biologically active enantiomer (R-enantiomer) of the racemic compound metalaxyl. Metalaxyl was first evaluated by the JMPR in 1982, and Codex MRLs for metalaxyl have been established.

Metalaxyl-M is registered for use on fruit, nut and vegetable crops for the control of various fungal diseases such as those caused by *Phytophthora* and *Pythium* spp. It is applied to foliage, soil or seed and also as a post-harvest fruit treatment.



IUPAC name	(R)-2-[(2,6-dimethylphenyl)-methoxyacetylamino]-propionic acid methyl ester
Chemical Abstracts name	*N*-(2,6-dimethylphenyl)-*N*-(methoxyacetyl)-D-alanine methyl ester

The Meeting received information on the metabolism and environmental fate of metalaxyl-M and on methods of residue analysis, stability in freezer storage, national registered use patterns, the results of supervised trials and farm animal feeding studies, the fate of residues in processing and national MRLs.

As metalaxyl-M constitutes 50% of metalaxyl, investigations into the metabolism and fate of metalaxyl can legitimately be accepted as supporting the metabolism and fate of metalaxyl-M. When the metabolism of metalaxyl and metalaxyl-M was compared directly, it was found to be similar.

In the studies of animal and plant metabolism and environmental fate, metalaxyl or metalaxyl-M uniformly ^{14}C labelled in the aromatic ring was used.

Metabolism

In the list below, the numbering is preserved from the 2002 JMPR toxicology evaluation.

Metabolite 1: *N*-(2,6-dimethylphenyl)-*N*-(methoxyacetyl)alanine

Metabolite 3: *N*-(2,6-dimethylphenyl)-*N*-(hydroxyacetyl)alanine methyl ester

Metabolite 6: *N*-(2,6-dimethylphenyl)-*N*-(hydroxyacetyl)alanine

Metabolite 7: *N*-(2,6-dimethyl-5-hydroxyphenyl)-*N*-(methoxyacetyl)alanine methyl ester

Metabolite 8: *N*-(2-hydroxymethyl-6-methylphenyl)-*N*-(methoxyacetyl)alanine methyl ester (occurs as two isomers)

Metabolite P: *N*-[(2-hydroxymethyl)-6-methylphenyl]-*N*-(hydroxyacetyl)alanine (occurs as two isomers)

Animals

The Meeting received the results of studies on metabolism in rats, lactating goats and laying hens. When animals were dosed orally with radiolabelled metalaxyl, most of the radiolabel was excreted in the urine within a short time, with a small amount in the faeces. Numerous metabolites resulting from hydrolysis, oxidation and demethylation of metalaxyl and subsequent conjugate formation were identified. In a study in goats, metalaxyl itself was not detected as a component of the residue in

tissues or milk. In a study in laying hens, low levels of metalaxyl were present in liver and eggs. The metabolic pathway for metalaxyl was similar in rats, goats and hens.

The absorption, distribution, metabolism and excretion of radiolabel were similar in *rats* dosed orally with ^{14}C-metalaxyl or ^{14}C-metalaxyl-M. Detailed information on metabolism in this species is reported in the 2002 JMPR toxicological evaluations.

Very little radiolabel was found in milk (0.003 mg/kg) or tissues (0.057 mg/kg in liver) from a *goat* dosed with ^{14}C-metalaxyl at the equivalent of 7 ppm in the feed for 10 days.

When two lactating dairy goats were dosed orally once daily for 4 consecutive days by gelatin capsule with ^{14}C-metalaxyl, equivalent to 77 ppm in the diet, the radiolabel was excreted rapidly: within 24 h of administration, 67% of the daily dose appeared in urine, 9% in faeces and 0.1% in milk. Metalaxyl was not detected as a component of the residue. Metabolite 6 was the main component of the residue in liver (0.19 mg/kg), leg muscle (0.014 mg/kg) and perirenal fat (0.065 mg/kg); metabolite 8 was the main residue component in kidney. The main metabolites in milk were C-10 and C-8 fatty acid conjugates of metabolite 3 (0.058 mg/kg). These fatty acids are conjugated through the hydroxyacetyl group of metabolite 3.

When five laying *hens* were dosed orally once daily for 4 consecutive days by gelatin capsule with ^{14}C-metalaxyl, equivalent to approximately 100 ppm metalaxyl in the diet, radiolabel recovered in edible tissues and eggs represented 0.97% of the administered dose; the remainder was recovered in excreta. Metabolite P (consisting of P1 and P2, steric isomers) was the main metabolite in egg white (0.056 mg/kg), egg yolk (0.072 mg/kg) and thigh muscle (0.31 mg/kg). Metalaxyl parent was identified in egg white (0.013 mg/kg), egg yolk (0.010 mg/kg) and liver (0.018 mg/kg), but not in thigh muscle or fat (< 0.001 mg/kg).

Plants

The Meeting received the results of studies on the metabolism of metalaxyl in grape, lettuce and potato and of metalaxyl-M in lettuce. No metabolites were identified in plants which had not already been identified in animals. Parent metalaxyl was the main component of the residue in grapes and in juice produced from the grapes when metalaxyl was used on grape vines. In treated lettuce, parent metalaxyl and metabolite 8 were each present at approximately 20% of the total residue. Metabolite 8 was the main residue component in lettuce in both cases in which metalaxyl and metalaxyl-M were compared. When metalaxyl was used on potato plants, some residue reached the tubers, where parent metalaxyl was the main residue component.

Grapevines in Switzerland were sprayed to runoff seven times at 14-day intervals with a ^{14}C-metalaxyl spray at a concentration of 0.050 kg ai/hl and were harvested 52 days after the last application. Parent metalaxyl (2.0 mg/kg) constituted 64% of the total residues in *grapes*.

When two grapevines in Switzerland were sprayed to runoff six times at approximately 14-day intervals with a ^{14}C-metalaxyl spray at a concentration of 0.030 kg ai/hl and harvested 68 days after the last application, parent metalaxyl (0.83 mg/kg) comprised 64% of total residues in the grapes. Metabolite 8 accounted for 20% of the residue, and metabolites 1, 6 and 7 were minor components (1.8–4.3%). When the grapes were separated into juice and presscake, metalaxyl was still the main part of the residue (62% and 57%, respectively).

Metalaxyl was the main identified component of the residue in *lettuce* (18.6% of the total ^{14}C residue) after seedlings in a greenhouse were sprayed twice, 2 weeks apart, with ^{14}C-metalaxyl at a rate equivalent to 0.25 kg ai/ha and harvested 2 weeks later. The identified metabolites (including glucose conjugates) were metabolite 8 (22% of the total ^{14}C residue), metabolite 6 (10%), metabolite 3 (8.9%), metabolite 7 (6.2%) and metabolite 1 (6.0%).

The metabolic pathways of metalaxyl-M and metalaxyl were compared in lettuce in a field in Switzerland treated three times at 10-day intervals with labelled compounds. The levels of total applied residue and parent compounds in the residue were generally comparable. Metabolite 8 (free and conjugated) was the main identified component of the residue in samples taken 14 and 21 days

after treatment. Enantiomeric ratio measurements suggested similar disappearance rates for the two enantiomers and little interconversion.

Potato plants in the field in Switzerland received five foliar treatments of ^{14}C-metalaxyl at 0.2 kg ai/ha at 10-day intervals and were harvested at maturity 5 weeks after the last treatment. Little residue reached the tubers (0.02 mg/kg ^{14}C as metalaxyl). No parent metalaxyl was detected in tubers. In a second experiment, the level of ^{14}C as metalaxyl in tubers was < 0.0001 mg/kg after ^{14}C-metalaxyl was applied to the soil (residues in soil, approximately 0.5 mg/kg), indicating that metalaxyl is not taken up by the tubers directly from the soil.

Potato plants in the field in the USA received six foliar treatments, about 2 weeks apart, of ^{14}C-metalaxyl at 1.3 kg ai/ha. Tubers harvested at maturity, 1 week after the last treatment, contained 0.5 mg/kg ^{14}C as metalaxyl, of which 50–60% was parent metalaxyl. A number of metabolites were identified, but only the concentration of metabolite 8 exceeded 5% of the residue.

Environmental fate

Soil

The Meeting received information on the behaviour and fate of metalaxyl and metalaxyl-M during aerobic metabolism in a number of soils. The rate of degradation is strongly influenced by the properties of the soil, including its biological activity and the conditions of temperature, moisture and concentration of the residue, with recorded half-lives in the range of 5–180 days. In direct comparisons of metalaxyl and metalaxyl-M under aerobic conditions, metalaxyl-M was the more persistent in one case and less persistent in two others. The main soil metabolite is metabolite 1 or, in the case of metalaxyl-M, the specific enantiomer of metabolite 1.

Field dissipation studies for metalaxyl-M were provided from France, Italy, Spain and Switzerland. Metalaxyl-M residues disappeared from the soil with half-lives ranging from 5 to 35 days. The residues occurred mostly in the top 10 cm of soil, but some reached lower levels. The enantiomer of metabolite 1 was produced in all cases, and its level sometimes exceeded that of the parent metalaxyl-M. A comparison of enantiomeric ratios in metalaxyl residues in soil suggested that the R-enantiomer (i.e. the metalaxyl-M enantiomer) disappeared more quickly than the S-enantiomer. This resulted in a preponderance of S-enantiomer in the metalaxyl residue and a preponderance of R-enantiomer in metabolite 1.

The studies of dissipation in the field suggest that, after use of metalaxyl-M for seed treatment or at the time of sowing, little or none will remain as a soil residue when the crop is harvested.

Rotational crops

Information on the fate of radiolabelled metalaxyl in confined crop rotational studies and of unlabelled metalaxyl-M in field rotational crops was made available to the Meeting. The studies with radiolabel showed that parent metalaxyl was usually a minor part of the residue that reached the rotational crop. The identifiable metabolites were also usually minor, but metabolite 8 as glucose conjugates was detected in spring wheat stalks at 2.3 mg/kg. Metalaxyl-M residues were not detected in unconfined field rotational crops in Switzerland or the United Kingdom, but levels of 0.11 mg/kg were present in broccoli and 0.03 mg/kg in lettuce leaves from crops sown 29 days after treatment of the first crop in a study in Italy. The short interval was used in order to simulate the ploughing-in of a failed crop and the sowing of a new one.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of metalaxyl in plant material, animal tissues, milk and eggs.

Common moiety methods rely on the 2,6-dimethylaniline moiety of metalaxyl and many of its metabolites, and these methods have been used to identify metalaxyl residues in animal commodities. The typical LOQs are 0.05 mg/kg for tissues and 0.01 mg/kg for milk. Metabolite 8, containing the 2-hydroxymethyl-6-methylaniline moiety, is apparently partially converted to 2,6-dimethylaniline, resulting in low and variable recoveries.

With gas–liquid chromatography and nitrogen–phosphorus detection and HPLC with mass spectrometry detection procedures for identifying metalaxyl or metalaxyl-M after a simple extraction and limited clean-up, the LOQs are 0.02–0.04 mg/kg for many crop substrates. A modification to the method (method REM 181.06), with the introduction of an HPLC chiral separation step before determination, allows for the analysis of specific enantiomers.

A multi-residue regulatory method (DFG S19) is available for metalaxyl.

Method REM 181.06 (gas–liquid chromatography with mass spectrometry detection) is not a multi-residue method, but it is enantioselective and suitable as a regulatory method for metalaxyl-M.

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of metalaxyl-M in crops (orange, potato, rape-seed, tomato, wheat) and animal commodities (beef muscle, beef liver, milk, eggs) during storage of analytical samples. Metalaxyl-M residues were stable in these substrates and under the conditions and intervals of storage (2 years). There was no evidence of epimerization during freezer storage. As a common moiety method was used for the animal commodity samples, storage stability refers to the total residue rather than to parent metalaxyl-M. As the common moiety method, which relies on the 2,6-dimethylaniline moiety, is less suitable for metabolite 8, the freezer stability of this metabolite during storage is not demonstrated.

Definition of the residue

Parent metalaxyl is the main identifiable component of the residue in crops resulting from use of metalaxyl, although metabolite 8 can occur at approximately the same levels. Metabolite 8 was not considered to be toxicologically significant.

The current residue definition of metalaxyl is metalaxyl. As metalaxyl-M is one enantiomer of metalaxyl, it is covered by the current residue definition. Non-enantioselective methods cannot distinguish metalaxyl-M from metalaxyl, but an enantioselective method is available. While metalaxyl-M and metalaxyl are both registered for crop uses, it is preferable, for enforcement purposes, to maintain a single residue definition. As the 2002 JMPR recommended a group ADI for metalaxyl and metalaxyl-M, the inclusive residue definition is also suitable for risk assessment purposes. The Meeting recommended that metalaxyl-M be contained within the metalaxyl residue definition and recommended amendment of the metalaxyl residue definition to provide definitions for enforcement and risk assessment purposes.

For plant commodities: Metalaxyl, including metalaxyl-M. Definition of the residue (for compliance with MRL and for estimation of dietary intake): metalaxyl. Note: Metalaxyl is a racemic mixture of an R-enantiomer and an S-enantiomer. Metalaxyl-M is the R-enantiomer.

In animals dosed with metalaxyl, parent metalaxyl was either a minor part of the residue or was not detected. Analytical methods for metalaxyl are based on a common moiety method, and residues in the farm animal feeding studies were measured by this method. Common moiety residues are acceptable for estimation of dietary intake when the parent compound is a minor part of the residue. The log P_{OW} for metalaxyl-M is 1.7, and the studies of animal metabolism confirm that metalaxyl is not fat-soluble.

For animal commodities: Metalaxyl including metalaxyl-M. Definition of the residue (for compliance with MRL and for estimation of dietary intake): metalaxyl and metabolites containing the 2,6-dimethylaniline moiety, expressed as metalaxyl.

Results of supervised trials on crops

The Meeting received data from supervised trials with metalaxyl-M used on citrus fruit, apple, grape, onion, tomato, pepper, lettuce, spinach, potato, sunflower and cacao. In some trials, residues were measured on samples taken just before and just after ('zero-day' residue) the last application. The residue level measured just before the last application, expressed as a percentage of zero-day residue, provides a measure of the contribution of previous applications to the final residue in the use pattern used in the trial. For grapes, the average carryover of residue was 32% in 12 trials in Australia, 57% in three trials in Germany and 46% in three trials in Italy. In lettuce, the average carryover was 1.7% in six trials in France, Germany and Italy. In spinach, the average carryover was 1.1% in eight trials in France.

Residue data were evaluated only when labels (or translations of labels) describing the relevant GAP were available to the Meeting.

Citrus fruit

Metalaxyl-M is registered for use as a post-harvest treatment on citrus in Israel. It is applied as a 0.1 kg ai/hl spray.

In the trials, the formulation of metalaxyl-M was mixed with a commercial wax to produce a spray solution, which was applied at a rate of 200 l /90 t of fruit (theoretical concentration of residue, 2.1 g/t). The residue levels in three trials on *oranges* were 1.2, 1.3 and 1.6 mg/kg in whole fruit and < 0.02 mg/kg in pulp. This method of post-harvest application includes control of the application rate in terms of the amount of metalaxyl-M per unit weight of fruit. The residue levels agreed substantially with expectations.

The Meeting noted that three supervised trials is generally an insufficient number for a major commodity such as oranges.

The residue levels of metalaxyl-M in the trials conducted in line with Israeli GAP did not exceed the current metalaxyl MRL of 5 mg/kg for citrus fruit.

Apple

Metalaxyl-M is registered in Spain for soil treatment around apple trees at 0.5–1.0 g ai/tree and in Italy at 0.5–4 g ai/tree. In two trials each in France, Italy and Spain at application rates of 0.78–10 kg ai/ha, no residues were detected in apples (< 0.02 mg/kg). For an assumed 500–1000 trees per ha, the rate of 10 kg ai/ha appears to be exaggerated.

The Meeting estimated a maximum residue level for metalaxyl-M in apples of 0.02* and an STMR value of 0 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.02* mg/kg would not exceed the current metalaxyl MRL of 1 mg/kg for pome fruits.

Grape

In Australia, metalaxyl-M is registered for a maximum four applications on grapes at 0.11 kg ai/ha, with a PHI of 7 days. The residue levels in grapes in five Australian trials matching GAP, but with six applications instead of four, were: < 0.02, 0.03, 0.06, 0.14 and 0.52 mg/kg. As the final residue level should not be influenced by earlier applications, residue levels after six applications are acceptable as equivalent to residues levels in GAP trials.

No GAP was available to evaluate the data for grapes treated in Germany and Switzerland.

In Greece, grapes may be treated four times with metalaxyl-M at 0.1 kg ai/ha, with harvest 15 days after the last application. The residue levels in grapes in six trials in Italy and Portugal, conducted substantially according to Greek GAP, were: 0.04, 0.06, 0.18, 0.19, 0.21 and 0.55 mg/kg.

The residue levels in the Australian and European trials appear to be similar and can be combined. In summary, the residue levels in the 11 trials, in ranked order, were: < 0.02, 0.03, 0.04, 0.06, 0.06, 0.14, 0.18, 0.19, 0.21, 0.52 and 0.55 mg/kg

The Meeting estimated a maximum residue level for metalaxyl-M in grapes of 1 mg/kg and an STMR value of 0.14 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 1 mg/kg would not exceed the current metalaxyl MRL of 1 mg/kg for grapes.

Onion

In Ecuador and Uruguay, metalaxyl-M is registered for a maximum of three applications on onions at 0.1 and 0.12 kg ai/ha, with a PHI of 7 days. Metalaxyl-M residue levels in bulb onions in three Brazilian trials matching Uruguayan GAP, but with four applications instead of three, were < 0.02 (two) and 0.02 mg/kg.

Metalaxyl-M is registered in Germany for a maximum of three applications on onions at 0.097 kg ai/ha, with a PHI of 21 days. In four trials in Switzerland with conditions matching German GAP, the residue levels were all below the LOQ (0.02 mg/kg).

Data on residues in trials in onions in Italy and Spain could not be evaluated because no relevant GAP was available.

In summary, the residue levels in the seven trials, in ranked order, were < 0.02 (six) and 0.02 mg/kg.

The Meeting estimated a maximum residue level for metalaxyl-M in onions of 0.03 mg/kg and an STMR value of 0.02 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.02 mg/kg would not exceed the current metalaxyl MRL of 2 mg/kg for bulb onions.

Tomato

Metalaxyl-M is registered for foliar application on tomatoes in Algeria, Chile, Ecuador, Greece, Israel and Morocco at 0.10–-0.14 kg ai/ha, with a PHI of 3 days and a maximum of three or four treatments.

Residue levels in tomatoes in six greenhouse trials in France, two in Spain and four in Switzerland at 0.15 kg ai/ha, with harvest 3 days after treatment (equivalent to the stated GAP) were: 0.02 (two), 0.02, 0.03, 0.04, 0.04, 0.05, 0.05, 0.08, 0.09, 0.12 and 0.18 mg/kg.

The Meeting estimated a maximum residue level for metalaxyl-M in tomatoes of 0.2 mg/kg and an STMR value of 0.045 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.2 mg/kg would not exceed the current metalaxyl MRL of 0.5 mg/kg for tomatoes.

Pepper

GAP for use of metalaxyl-M in Italy allows three soil applications of 1 kg ai/ha with a 15-day PHI. Data on residues from Italian and Spanish trials approximating Italian GAP were provided. In some of the trials, residues were measured 10 and 20 days after the last application instead of 15 days, but these trials were considered valid because the residue levels were relatively unchanged. The residue levels in the seven greenhouse trials were: < 0.02 (two), 0.02, 0.03, 0.08, 0.10 and 0.36 mg/kg; and those in the three outdoor trials in Italy were: < 0.02 (two) and 0.02 mg/kg.

The Meeting decided to use the data from the greenhouse trials: < 0.02 (two), 0.02, 0.03, 0.08, 0.10 and 0.36 mg/kg.

The Meeting estimated a maximum residue level for metalaxyl-M in sweet peppers of 0.5 mg/kg and an STMR value of 0.03 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.5 mg/kg would not exceed the current metalaxyl MRL of 1 mg/kg for peppers.

Lettuce

Metalaxyl-M is registered in Spain for use on lettuce at 0.10 kg ai/ha with a PHI of 14 days. The residue levels in lettuce were < 0.02 mg/kg in an Italian trial matching Spanish GAP. The residue levels in lettuce in four French trials matching Spanish GAP were: < 0.02 (two), 0.02 and 0.03 mg/kg.

Metalaxyl-M is registered in Germany for a maximum of two applications on lettuce at 0.097 kg ai/ha, with a PHI of 21 days. In six trials on head lettuce in Germany under conditions matching GAP, but with three applications instead of two, the residue levels were: < 0.02 (four), 0.02 and 0.03 mg/kg. In two German greenhouse trials at 0.10 kg ai/ha with harvest 21 days after the second application, the residue levels were < 0.02 and 0.41 mg/kg.

In two trials in The Netherlands matching German GAP, the residue levels were < 0.02 mg/kg.

Trials in Spain and Switzerland could not be evaluated because there was no matching GAP.

In summary, the residue levels in lettuce in the 15 trials, in ranked order, were: < 0.02 (10), 0.02 (two), 0.03 (two) and 0.41 mg/kg.

The Meeting estimated a maximum residue level for metalaxyl-M in head lettuce of 0.5 mg/kg and an STMR value of 0.02 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.5 mg/kg would not exceed the current metalaxyl MRL of 2 mg/kg for head lettuce.

Spinach

Metalaxyl-M is registered in Switzerland for a maximum of six applications on spinach at 0.10 kg ai/ha with a PHI of 14 days. In three trials on spinach in Switzerland at 0.10 kg ai/ha and three at 0.14 kg ai/ha, with intervals before harvest of 10 or 14 days, the residue levels were all < 0.02 kg/ha. The levels were also < 0.02 mg/kg in two trials in Germany matching the conditions of GAP in Switzerland.

In a number of trials in France in which the application rate was 0.10 or 0.14 kg ai/ha, spinach was sampled for analysis 10 and 20 days after treatment. The Meeting noted that the residue levels generally changed slowly between 10 and 20 days post-treatment and decided to accept the residue levels at 10 days as sufficiently close to those expected at 14 days. The residue levels in the 10 French trials were: < 0.02 (two), 0.02 (four), 0.03, 0.04 (two) and 0.05 mg/kg.

In summary, metalaxyl-M residue levels in the 18 trials were < 0.02 (10), 0.02 (four), 0.03, 0.04 (two) and 0.05 mg/kg.

The Meeting estimated a maximum residue level for metalaxyl-M in spinach of 0.1 mg/kg and an STMR value of 0.02 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.1 mg/kg would not exceed the current metalaxyl MRL of 2 mg/kg for spinach.

Potato

Labels were available from Algeria, Australia, Austria, Chile, Ecuador, Greece, Israel and Morocco from formulations for foliar application of metalaxyl-M to potatoes at 0.1–0.12 kg ai/ha. The

information on GAP suggests that the recommended foliar application rate on potatoes is 0.1 kg ai/ha in many situations.

The results of supervised trials were available from Brazil (three at 0.1 kg ai/ha and three at 0.2 kg ai/ha), Germany (six at 0.1 kg ai/ha), Switzerland (three at 0.075 kg ai/ha) and the United Kingdom (four at 0.1 kg ai/ha). The residue levels in all 19 trials, measured at intervals of 0–28 days after the last treatment, were below the LOQ (0.02 mg/kg).

As residues were found in potato tubers in the metabolism studies after high application rates, the median residue values cannot be assumed to be nil. The Meeting estimated a maximum residue level for metalaxyl-M in potato of 0.02* mg/kg and an STMR value of 0.02 mg/kg. Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.02* mg/kg would not exceed the current metalaxyl MRL of 0.05* mg/kg for potato.

Sunflower seed

Metalaxyl-M is registered in China and Serbia and Montenegro for use as a seed treatment at 0.105 kg ai/100 kg sunflower seed. The Meeting agreed that the results of trials from other countries could be evaluated with respect to this seed treatment GAP.

In six trials in France and two in Spain, metalaxyl-M was used as seed treatment at a nominal rate of 105 g ai/100 kg seed (measured, 61–80 g ai/100 kg seed). The residue levels in harvested sunflower seed 125–151 days after sowing were all below the LOQ (0.01 and 0.02 mg/kg). Because of the long interval between sowing and harvest and the solubility of metalaxyl-M, residues would not be expected in harvested sunflower seed.

The Meeting estimated a maximum residue level for metalaxyl-M in sunflower seed of 0.02* mg/kg and an STMR value of 0 mg/kg. Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.02* mg/kg would not exceed the current metalaxyl MRL of 0.05* mg/kg for sunflower seed.

Cacao beans

Metalaxyl-M is registered in Côte d'Ivoire for use on cacao at 0.012 kg ai/ha. In eight trials in Côte d'Ivoire in which metalaxyl-M was applied as foliar treatment four times at 0.09 kg ai/ha (an exaggerated rate), with harvest 29–30 days after the last treatment, the residue levels in the cacao beans were: < 0.02 (four) and 0.02 (four) mg/kg. The cacao beans were fermented and dried before analysis. The Meeting agreed that the residue levels after application at the label rate would not exceed 0.02 mg/kg.

The Meeting estimated a maximum residue level for metalaxyl-M in cacao beans of 0.02 mg/kg and an STMR value of 0.02 mg/kg.

Metalaxyl-M residue levels complying with the estimated maximum residue level of 0.02 mg/kg would not exceed the current metalaxyl MRL of 0.2 mg/kg for cacao beans.

Fate of residues during processing

The Meeting received information on the fate of metalaxyl-M residues during the production of fruit juices and vinification. The Meeting also received information that metalaxyl-M is hydrolytically stable under hydrolysis conditions that simulate those occurring during food processing.

The following processing factors were calculated from the data from the trials. The factors are mean values, excluding those calculated in cases of undetectable residues.

Commodity	Processed product	Processing factor	No. of trials
Orange	Washed fruit	0.97	2
	Juice, pasteurized	0.060	4

	Oil	9.0	4
	Pomace, wet	1.1	4
	Pomace, dry	4.1	4
	Peel	2.5	3
	Pulp	0.091	1
	Marmalade	0.39	4
Grapes	Juice	0.36	6
	Young wine	0.87	8
	Wine	0.66	13

The Meeting used the processing factors to estimate STMR-Ps for processed commodities. The processing factor for wine (0.66) was applied to the grape STMR (0.14 mg/kg) to calculate an STMR-P of 0.092 mg/kg for wine. The processing factor for grape juice (0.36) was applied to the grape STMR (0.14 mg/kg) to calculate an STMR-P of 0.050 mg/kg for grape juice

Residues in animal commodities

Feeding studies

The Meeting received the results of studies of feeding metalaxyl to lactating dairy cows and laying hens, which provided information on probable residue levels in tissues, milk and eggs from residues in animal feeds.

A group of three lactating dairy cows were dosed daily with metalaxyl, equivalent to 75 ppm in their diet, and were slaughtered for tissue collection on days 14, 21 and 28. Liver, kidney, fat and muscle were analysed by a dimethylaniline common moiety method. The residues were transitory and did not accumulate, and the interval between last dose and slaughter (4 and 23.5 h) influenced the residue levels more than the duration of dosing. The level of residue in milk was 0.02 mg/kg. The residue levels in the tissues collected on day 28 from the animal slaughtered 23.5 h after the last dose were 0.11 mg/kg in kidney, 0.12 mg/kg in liver, < 0.05 mg/kg in fat and 0.06–0.08 mg/kg in muscle.

Groups of 15 laying hens were dosed daily for 28 days with metalaxyl at levels equivalent to 10, 30 and 100 ppm in the feed. Tissue and egg samples were analysed by a dimethylaniline common moiety method. No residues appeared in the eggs (< 0.05 mg/kg) at any dose. The residue levels in the tissues of hens fed 10 ppm were generally below the LOQ (< 0.05 mg/kg) or, in a few cases, just above the LOQ.

Maximum residue levels

The farm animal feeding studies suggest that residues would generally be undetected or transitory in meat, milk and eggs if metalaxyl was present in animal feeds.

Farm animals are therefore not exposed to residues in their feed from commodities in this evaluation, and no MRLs have been established for metalaxyl in animal commodities. Consequently, the Meeting agreed not to recommend animal commodity maximum residue levels.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the metalaxyl-M residue levels listed below are already covered by existing metalaxyl MRLs.

Definition of the residue

For plant commodities.

Metalaxyl including metalaxyl-M: for compliance with MRL and for estimation of dietary intake: metalaxyl.

For animal commodities. (Note that no metalaxyl MRLs are currently recommended for animal commodities).

Metalaxyl including metalaxyl-M: for compliance with MRL and for estimation of dietary intake: metalaxyl and metabolites containing the 2,6-dimethylaniline moiety, expressed as metalaxyl.

Note: Metalaxyl is a racemic mixture of an R-enantiomer and an S-enantiomer. Metalaxyl-M is the R-enantiomer.

CCN	Commodity Name	Metalaxyl-M		Metalaxyl
		Estimated maximum residue level, mg/kg	STMR or STMR-P mg/kg	Existing MRL mg/kg
FP 0009	Apple	0.02(*)	0	Pome fruits 1
FB 0269	Grapes	1	0.14	1
VL 0482	Lettuce, Head	0.5	0.02	2
VA 0385	Onion, Bulb	0.03	0.02	2
VO 0445	Peppers, Sweet	0.5	0.03	Peppers 1
VO 0448	Tomato	0.2	0.045	0.5
VR 0589	Potato	0.02(*)	0.02	0.05(*)
VL 0502	Spinach	0.1	0.02	2
SO 0702	Sunflower seed	0.02(*)	0	0.05(*)
SB 0715	Cacao beans	0.02	0.02	0.2
	Grape juice		0.050	
	Wine		0.092	

* at or about the LOQ

DIETARY RISK ASSESSMENT

Long-term intake

Estimated Theoretical Maximum Daily Intakes for the five GEMS/Food regional diets, based on recommended MRLs for metalaxyl, were in the range of 2-10% of the ADI (Annex 3 of the Report). The Meeting concluded that the long-term intake of residues of metalaxyl and metalaxyl-M resulting from their uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The 2002 JMPR decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of metalaxyl and metalaxyl-M residues is unlikely to present a public health concern.

REFERENCES

Note. References are listed by company reference number, not by author

00DA05. Adam D. 2000. Hydrolysis of ^{14}C-labelled CGA 329351 under processing conditions. Novartis Crop Protection, Switzerland. Report 00DA05. Syngenta File N° CGA329351/1386. Unpublished.

01/95. Tack T. 1995. CGA 329351 + Mancozeb (A-9407A 68WP), potato, UK. Ciba Agriculture, United Kingdom. Residue report 01/95. Syngenta File N° CGA329351/0150. Unpublished.

0111301. Pointurier R. 2002. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on tomatoes in France (south). ADME - Bioanalyses, France. Report 0111301. Syngenta File N° CGA329351/1644. Unpublished.

0111401. Pointurier R. 2002. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on tomatoes in France (south). ADME - Bioanalyses, France. Report 0111401. Syngenta File N° CGA329351/1645. Unpublished.

02/95. Tack T. 1995. CGA 329351 + Mancozeb (A-9407A 68WP), potato, UK. Ciba Agriculture, United Kingdom. Residue report 02/95. Syngenta File N° CGA329351/0151. Unpublished.

06/79. Gross D. 1979. Identification of metabolites of CGA 48988 (Ridomil®) in grapevine. Ciba-Geigy, Switzerland. Report 06/79. Syngenta File N° CGA48988/2018. Unpublished.

08/78. Ellgehausen H. 1978. Degradation of CGA 48988 (Ridomil) in soil under aerobic, aerobic/anaerobic and sterile/aerobic conditions. Ciba-Geigy Ltd. Basel, Switzerland. Report 08/78. Syngenta File N° CGA48988/0193. Unpublished.

11/78. Gross D. 1978. Metabolism of CGA 48988 in grapevine. Ciba-Geigy, Switzerland. Report 11/78. Syngenta File N° CGA48988/2017. Unpublished.

121/97. Kühne RO. 1998. Magnitude of residues of metalaxyl-M (CGA 329351) in sweet peppers applied as formulation GR 2.5 in Italy. Novartis Crop Protection, Switzerland. Report 121/97, includes 2127/97, 2128/97, 2129/97 and 2130/97. Syngenta File N° CGA329351/0915. Unpublished.

124/97. Kühne RO. 1998. Magnitude of residues of metalaxyl-M (CGA 329351) in apples applied as formulation GR 2.5 in France (south). Novartis Crop Protection, Switzerland. Report 124/97, includes 2139/97 and 2140/97. Syngenta File N° CGA329351/1023. Unpublished.

125/97. Kühne RO. 1998. Magnitude of residues of metalaxyl-M (CGA 329351) and copper in spinach applied as formulations ES 350 and WP 42.5 in sise-by-side trials in France (south). Novartis Crop Protection, Switzerland. Report 125/97, includes 2141/97, 2142/97, 2143/97, 2144/97, 2145/97, 2146/97, 2147/97, 2148/97, 2149/97, 2150/97 and 2151/97. Syngenta File N° CGA329351/0931. Unpublished.

19/85. Guth JA. 1985. Degradationof metalaxyl (CGA 48988) in aerobic soils at a temperature of 15°C. Ciba-Geigy Ltd. Basel, Switzerland. Report 19/85. Syngenta File N° CGA48988/0197. Unpublished.

19/97. Muller T. 1997. Comparison of the adsorption, distribution, metabolism and excretion of [phenyl-U-^{14}C]CGA 329351 and [phenyl-U-^{14}C]CGA 48988 in the rat. Project 034AM03. Report 19/97. Novartis, Switzerland. Unpublished.

2000/00. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (north). Syngenta AG, Switzerland. Report 2000/00. Syngenta File N° CGA245704/0762. Unpublished.

2001/00. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (south). Syngenta AG, Switzerland. Report 2001/00. Syngenta File N° CGA245704/0758. Unpublished.

2002/00. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (south). Syngenta AG, Switzerland. Report 2002/00. Syngenta File N° CGA245704/0759. Unpublished.

2007/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Spain. Novartis Crop Protection, Switzerland. Report 2007/97. Syngenta File N° CGA329351/0946. Unpublished.

2008/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Spain. Novartis Crop Protection, Switzerland. Report 2008/97. Syngenta File N° CGA329351/0947. Unpublished.

2009/99. Sack S. 2000. Residue study with acibenzolar-S-Methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Spain. Novartis Crop Protection AG, Switzerland. Report 2009/99. Syngenta File N° CGA245704/0705. Unpublished.

201/01. Kühne RO. 2003. Stability of residues of metalaxyl-M (CGA 329351) in deep freeze stored analytical specimens of oranges (fruit), potatoes (tuber), rape seed (seed), tomatoes (fruit) and wheat (grain). Syngenta Crop Protection, Switzerland. Report 201/01. Syngenta File N° CGA329351/1737. Unpublished.

2010/99. Sack S. 2000. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Spain. Novartis Crop Protection AG, Switzerland. Report 2010/99. Syngenta File N° CGA245704/0706. Unpublished.

2011/01. Kühne RO. 2002. Residue study with mancozeb (ASF21) and metalaxyl-M (CGA 329351) in or on tomatoes in Switzerland. Syngenta Crop Protection, Switzerland. Report 2011/01. Syngenta File N° CGA329351/1624. Unpublished.

2011/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in Spain. Novartis Crop Protection, Switzerland. Report 2011/97. Syngenta File N° CGA329351/0912. Unpublished.

2012/01. Kühne RO. 2002. Residue study with mancozeb (ASF21) and metalaxyl-M (CGA 329351) in or on tomatoes in Switzerland. Syngenta Crop Protection, Switzerland. Report 2012/01. Syngenta File N° CGA329351/1623. Unpublished.

2012/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in Spain. Novartis Crop Protection, Switzerland. Report 2012/97. Syngenta File N° CGA329351/0911. Unpublished.

2013/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on onions in Spain. Novartis Crop Protection, Switzerland. Report 2013/97. Syngenta File N° CGA329351/0925. Unpublished.

2014/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on onions in Spain. Novartis Crop Protection, Switzerland. Report 2014/97. Syngenta File N° CGA329351/0924. Unpublished.

2015/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on apples in Spain. Novartis Crop Protection, Switzerland. Report 2015/97. Syngenta File N° CGA329351/1035. Unpublished.

2016/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on apples in Spain. Novartis Crop Protection, Switzerland. Report 2016/97. Syngenta File N° CGA329351/1036. Unpublished.

2023/00. Kühne RO. 2001. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Spain. Syngenta Crop Protection, Switzerland. Report 2023/00. Syngenta File N° CGA329351/1531. Unpublished.

2024/00. Kühne RO. 2001. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Spain. Syngenta Crop Protection, Switzerland. Report 2024/00. Syngenta File N° CGA329351/1530. Unpublished.

2025/98.Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) + copper as copper oxychloride (ASF 45) and metalaxyl-M (CGA 329351) + chlorothalonil (ASF 41) in or on onions in Italy. Novartis Crop Protection, Switzerland. Report 2025/98. Syngenta File N° CGA329351/1149. Unpublished.

2027/99. Kühne RO. 2003. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on soil in Italy. Syngenta Crop Protection, Switzerland. Report 2027/99. Syngenta File N° CGA329351/1729. Unpublished.

2028/98. Sack S. 1999. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Italy. Novartis Crop Protection AG, Switzerland. Report 2028/98. Syngenta File N° CGA245704/0638. Unpublished.

2028/99. Kühne RO. 2003. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on soil in Switzerland. Syngenta Crop Protection, Switzerland. Report 2028/99. Syngenta File N° CGA329351/1731. Unpublished.

2029/99. Kühne RO. 2003. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on soil in Switzerland. Syngenta Crop Protection, Switzerland. Report 2029/99. Syngenta File N° CGA329351/1732. Unpublished.

2030/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on grapes in Portugal. Novartis, Switzerland. Report 2030/97. Syngenta File N° CGA329351/1034. Unpublished.

2030/99. Kühne RO. 2003. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on soil in France (south). Novartis Crop Protection, Switzerland. Report 2030/99. Syngenta File N° CGA329351/1740. Unpublished.

2032/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and copper in or on onions in Italy. Novartis Crop Protection, Switzerland. Report 2032/97. Syngenta File N° CGA329351/0921. Unpublished.

2033/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and chlorothalonil (ASF 41)in or on onions in Italy. Novartis Crop Protection, Switzerland. Report 2033/97. Syngenta File N° CGA329351/0922. Unpublished.

2035/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on grapes in Italy. Novartis, Switzerland. . Report 2035/97. Syngenta File N° CGA329351/1037. Unpublished.

2035/99. Sack S. 2000. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Italy. Novartis Crop Protection, Switzerland. Report 2035/99. Syngenta File N° CGA245704/0720. Unpublished.

2036/98. Kühne RO. 2000. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on Soil in France (South). Novartis Crop Protection AG. Basel, Switzerland. Report 2036/98 unpublished. Syngenta File N° CGA329351/1210.

2036/99. Sack S. 2000. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Italy. Novartis Crop Protection, Switzerland. Report 2036/99. Syngenta File N° CGA245704/0721. Unpublished.

2044/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Italy. Novartis Crop Protection, Switzerland. Report 2044/97. Syngenta File N° CGA329351/0948. Unpublished.

2044/99. Sack S. 2000. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on spinach in Switzerland. Novartis Crop Protection, Switzerland. Report 2044/99. Syngenta File N° CGA245704/0723. Unpublished.

2045/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Italy. Novartis Crop Protection, Switzerland. Report 2045/97. Syngenta File N° CGA329351/0949. Unpublished.

2045/99. Sack S. 2000. Residue Study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in

or on spinach in Switzerland. Syngenta, Switzerland. Report 2045/99. Syngenta File N° CGA245704/0742). Unpublished.

2046/98. Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Spain. Novartis Crop Protection, Switzerland. Report 2046/98. Syngenta File N° CGA329351/1103. Unpublished.

2046/99. Sack S. 2000. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on spinach in Switzerland. Novartis Crop Protection, Switzerland. Report 2046/99. Syngenta File N° CGA245704/0724. Unpublished.

2047/98. Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) in or on sweet peppers in Spain. Novartis Crop Protection, Switzerland. Report 2047/98. Syngenta File N° CGA329351/1104. Unpublished.

2048/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on tomatoes in Spain. Novartis Crop Protection, Switzerland. Report 2048/98. Syngenta File N° CGA329351/1147. Unpublished.

2049/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on tomatoes in Spain. Novartis Crop Protection, Switzerland. Report 2049/98. Syngenta File N° CGA329351/1148. Unpublished.

2050/99. Sack S. 2000. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Switzerland. Syngenta AG, Switzerland. Report 2050/99. Syngenta File N° CGA245704/0746. Unpublished.

2051/99. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Switzerland. Syngenta AG, Switzerland. Report 2051/99. Syngenta File N° CGA245704/0747. Unpublished.

2052/99. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in Switzerland. Syngenta AG, Switzerland. Report 2052/99. Syngenta File N° CGA245704/0748. Unpublished.

2057/99. Kühne RO. 2003. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on soil in Spain. Syngenta Crop Protection, Switzerland. Report 2057/99. Syngenta File N° CGA329351/1733. Unpublished.

2062/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and copper as copper oxychloride in or on head lettuce in Italy. Novartis Crop Protection, Switzerland. Report 2062/97. Syngenta File N° CGA329351/0964. Unpublished.

2062/99. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on spinach in France (North). Syngenta, Switzerland. Report 2062/99. Syngenta File N° CGA245704/0753. Unpublished.

2068/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on potatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2068/98.Syngenta File N° CGA329351/1150. Unpublished.

2069/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on potatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2069/98. Syngenta File N° CGA329351/1141. Unpublished.

2070/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on potatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2070/98. Syngenta File N° CGA329351/1142. Unpublished.

2071/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on tomatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2071/98. Syngenta File N° CGA329351/1143. Unpublished.

2072/98. Kühne RO. 1999. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on tomatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2072/98. Syngenta File N° CGA329351/1144. Unpublished.

208/98. Kühne RO. 2000. Crop rotation study for metalaxyl-M (CGA 329351) in follow-up crop after treatment of potatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 208/98. Syngenta File N° CGA329351/1300. Unpublished.

2081/00. Kühne RO. 2001. Residue study with metalaxyl-M (CGA 329351) in or on apples in Italy. Syngenta Crop Protection, Switzerland. Report 2081/00. Syngenta File N° CGA329351/1529. Unpublished.

2082/00. Kühne, RO. 2001. Residue study with metalaxyl-M (CGA 329351) in or on apples in Italy. Syngenta Crop Protection, Switzerland. Report 2082/00. Syngenta File N° CGA329351/1528. Unpublished.

209/98. Kühne RO. 2000. Crop rotation study for metalaxyl-M (CGA 329351) in follow-up crop after treatment of potatoes in the United Kingdom. Novartis Crop Protection, Switzerland. Report 209/98. Syngenta File N° CGA329351/1301. Unpublished.

210/98. Kühne RO. 2000. Crop rotation study for metalaxyl-M (CGA 329351) in follow-up crop after treatment of potatoes in Italy. Novartis Crop Protection, Switzerland. Report 210/98. Syngenta File N° CGA329351/1302. Unpublished.

2102/01. Kühne RO. 2002. Residue study with metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2102/01. Syngenta File N° CGA329351/1618. Unpublished.

2103/01. Kühne RO. 2002. Residue study with metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2103/01. Syngenta File N° CGA329351/1619. Unpublished.

2104/01. Kühne RO. 2002. Residue study with metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2104/01. Syngenta File N° CGA329351/1620. Unpublished.

2105/01. Kühne RO. 2002. Residue study with metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2105/01. Syngenta File N° CGA329351/1622. Unpublished.

2116/98. Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in Spain. Novartis Crop Protection, Switzerland. Report 2116/98. Syngenta File N° CGA329351/1161. Unpublished.

2118/98. Kühne RO. 1999 Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in France (north). Novartis Crop Protection, Switzerland. Report 2118/98. Syngenta File N° CGA329351/1079. Unpublished.

2119/98. Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in France (north). Novartis Crop Protection, Switzerland. Report 2119/98. Syngenta File N° CGA329351/1080. Unpublished.

212/00. Kühne RO. 2001. Validation of method REM 181.06. Validation by analysis of specimens fortified with metalaxyl-M (CGA 329351; R-enantiomer) and CGA 351920 (S-enantiomer) and determination of recoveries. Syngenta, Basel. Syngenta report No 212/00. Syngenta File N° CGA329351/1464. Unpublished.

2120/98. Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in France (south). Novartis Crop Protection, Switzerland. Report 2120/98. Syngenta File N° CGA329351/1081. Unpublished.

2121/98. Kühne RO. 1999. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in France (south). Novartis Crop Protection, Switzerland. Report 2121/98. Syngenta File N° CGA329351/1082. Unpublished.

2122/94. Kühne RO. 1996. Determination of residues of CGA 329351 and folpet in grapes (berries, must, new wine and wine) - field trial, Italy. Ciba-Geigy, Switzerland. Report 2122/94. Syngenta File N° CGA329351/0477. Unpublished.

2123/94. Kühne RO. 1996. Determination of residues of CGA 329351 and folpet in grapes (berries, must, new wine and wine) - field trial, Italy. Ciba-Geigy, Switzerland. Report 2123/94. Syngenta File N° CGA329351/0478. Unpublished.

2124/94. Kühne RO. 1996. Determination of residues of CGA 329351 and copper in grapes (berries, must, new wine and wine) -field trial, Italy. Ciba-Geigy, Switzerland. Report 2124/94. Syngenta File N° CGA329351/1544. Unpublished.

2125/94. Kühne RO. 1996. Determination of residues of CGA 329351 and copper in grapes (berries, must, new wine and wine) -field trial, Italy. Ciba-Geigy, Switzerland. Report 2125/94. Syngenta File N° CGA329351/1543. Unpublished.

2127/97, see 121/97

2128/97, see 121/97

2129/97, see 121/97

2130/97, see 121/97

2131/99. Kühne RO. 2000. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on head lettuce in the Netherlands. Novartis Crop Protection, Switzerland. Report 2131/99. Syngenta File N° CGA329351/1330. Unpublished.

2132/99. Kühne RO. 2000. Residue study with mancozeb (ASF 21) and metalaxyl-M (CGA 329351) in or on head lettuce in the Netherlands. Novartis Crop Protection, Switzerland. Report 2132/99. Syngenta File N° CGA329351/1331. Unpublished.

2139/97, see 124/97

2140/97, see 124/97

2141/97 see 125/97

2142/97 see 125/97

2143/97 see 125/97

2144/97 see 125/97

2145/97 see 125/97

2146/97 see 125/97

2147/97 see 125/97

2148/00. Kühne RO. 2002. Residue study with copper (ASF47) and metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2148/00. Syngenta File N° CGA329351/1610. Unpublished.

2148/97 see 125/97

2149/00. Kühne RO. 2002. Residue study with copper (ASF47) and metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2149/00. Syngenta File N° CGA329351/1611. Unpublished.

2149/97 see 125/97

2150/00. Kühne RO. 2002. Residue study with copper (ASF47) and metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2150/00. Syngenta File N° CGA329351/1613. Unpublished.

2150/97 see 125/97

2151/00. Kühne RO. 2002. Residue study with copper (ASF47) and metalaxyl-M (CGA 329351) in or on cocoa in Ivory Coast. Syngenta Crop Protection, Switzerland. Report 2151/00. Syngenta File N° CGA329351/1614. Unpublished.

2151/97 see 125/97

2169/99. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (north). Syngenta AG, Switzerland. Report 2169/99. Syngenta File N° CGA245704/0760. Unpublished.

2170/99. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (north). Syngenta AG, Switzerland. Report 2170/99. Syngenta File N° CGA245704/0761. Unpublished.

2171/99. Sack S. 2001. Residue study with acibenzolar-S-methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (south). Syngenta AG,

Switzerland. Report 2171/99. Syngenta File N° CGA245704/0756. Unpublished.

2172/99. Sack S. 2001. Residue study with acibenzolar-S-Methyl (CGA 245704) and metalaxyl-M (CGA 329351) in or on head lettuce in France (south). Syngenta AG, Switzerland. Report 2172/99. Syngenta File N° CGA245704/0757. Unpublished.

2186/99. Kühne RO. 2000. Residue study with metalaxyl-M (CGA 329351) in or on oranges in Spain. Syngenta AG, Switzerland. Report 2186/99. Syngenta File N° CGA329351/1429. Unpublished.

2252/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and folpet in or on head lettuce in Germany. Novartis Crop Protection, Switzerland. Report 2252/97. Syngenta File N° CGA329351/0984. Unpublished.

2253/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and folpet in or on head lettuce in Germany. Novartis Crop Protection, Switzerland. Report 2253/97. Syngenta File N° CGA329351/0985. Unpublished.

2254/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and folpet in or on head lettuce in Germany. Novartis Crop Protection, Switzerland. Report 2254/97. Syngenta File N° CGA329351/0986. Unpublished.

2255/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and folpet in or on head lettuce in Germany. Novartis Crop Protection, Switzerland. Report 2255/97. Syngenta File N° CGA329351/0987. Unpublished.

2260/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on head lettuce in France (north). Novartis Crop Protection, Switzerland. Report 2260/97. Syngenta File N° CGA329351/0940. Unpublished.

2261/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on head lettuce in France (north). Novartis Crop Protection, Switzerland. Report 2261/97. Syngenta File N° CGA329351/0941. Unpublished.

2262/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on head lettuce in France (south). Novartis Crop Protection, Switzerland. Report 2262/97. Syngenta File N° CGA329351/0942. Unpublished.

2263/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on head lettuce in France (south). Novartis Crop Protection, Switzerland. Report 2263/97. Syngenta File N° CGA329351/0943. Unpublished.

2325/97. Kühne RO. 1998. Residue Study with metalaxyl-M (CGA 329351) in or on oranges in Israel. Novartis Crop Protection, Switzerland. Report 2325/97. Syngenta File N° CGA329351/0858. Unpublished.

2327/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on oranges in Israel. Novartis Crop Protection, Switzerland. Report 2326/97. Syngenta File N° CGA329351/0859. Unpublished.

2327/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) in or on oranges in Israel. Novartis Crop Protection, Switzerland. Report 2327/97. Syngenta File N° CGA329351/0860. Unpublished.

2332/97. Kühne RO.1999. Residue study with folpet (ASF 16) and metalaxyl-M (CGA 329351) in or on grapes in Switzerland. Novartis, Switzerland. Report 2332/97. Syngenta File N° CGA329351/1062. Unpublished.

2334/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2334/97. Syngenta File N° CGA329351/0926. Unpublished.

2335/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and copper as copper oxychloride (ASF 45) in or on tomatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2335/97. Syngenta File N° CGA329351/0928. Unpublished.

2336/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2336/97. Syngenta File N° CGA329351/0927. Unpublished.

2337/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and copper as copper oxychloride (ASF 45) in or on tomatoes in Switzerland. Novartis Crop Protection, Switzerland. Report 2337/97. Syngenta File N° CGA329351/0929. Unpublished.

2338/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on onions in Switzerland. Novartis Crop Protection, Switzerland. Report 2338/97. Syngenta File N° CGA329351/0905. Unpublished.

2339/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and chlorothalonil (ASF 41) in or on onions in Switzerland. Novartis Crop Protection, Switzerland. Report 2339/97. Syngenta File N° CGA329351/0903. Unpublished.

2340/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on onions in Switzerland. Novartis Crop Protection, Switzerland. Report 2340/97. Syngenta File N° CGA329351/0906. Unpublished.

2341/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and chlorothalonil (ASF 41) in or on onions in Switzerland. Novartis Crop Protection, Switzerland. Report 2341/97. Syngenta File N° CGA329351/0904. Unpublished.

2347/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in France. Novartis Crop Protection, Switzerland. Report 2347/97. Syngenta File N° CGA329351/0907. Unpublished.

2348/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in France. Novartis Crop Protection, Switzerland. Report 2348/97. Syngenta File N° CGA329351/0908. Unpublished.

2349/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in France. Novartis Crop Protection, Switzerland. Report 2349/97. Syngenta File N° CGA329351/0909. Unpublished.

2350/97. Kühne RO. 1998. Residue study with metalaxyl-M (CGA 329351) and mancozeb (ASF 21) in or on tomatoes in France. Novartis Crop Protection, Switzerland. Report 2350/97. Syngenta File N° CGA329351/0910. Unpublished.

2383/97. Kühne RO. 1998. Study on fate and behaviour of metalaxyl-M (CGA 329351) in or on soil in Italy. Novartis Crop Protection, Switzerland. Report 2383/97. Syngenta File N° CGA329351/1028. Unpublished.

26/78. Hamböck H. 1978. Metabolism of CGA 48988 in the rat. Ciba-Geigy, Switzerland Report 26/78. Syngenta File N° CGA48988/0571. Unpublished.

26165. Das R. 1994. Report on boiling point / boiling range. Ciba-Geigy, Switzerland. Report 26165. Syngenta File N° CGA329351/0034. Unpublished.

26166. Das R. 1994. Report on density. Ciba-Geigy, Switzerland. Report 26166. Syngenta File N° File no 329351/0017. Unpublished.

26168. Stulz J. 1994. Report on octanol/water partition coefficient. Ciba-Geigy, Switzerland. Report 26168. Syngenta File N° CGA329351/0018. Unpublished.

26169. Stulz J. 1994. Report on water solubility. Ciba-Geigy, Switzerland. Report 26169. Syngenta File N° CGA329351/0016. Unpublished.

26171. Das R. 1994. Report on general physico-chemical properties (pure active ingredient). Ciba-Geigy, Switzerland. Report 26171. Syngenta File N° CGA329351/0021. Unpublished.

261B.00. Adams S. 1998. Determination of CGA 329351 in grapes, grape juice and wine by GLC. Analytical procedure 261B.00. Novartis Animal Health Australasia Pty Ltd. Unpublished.

262315. Schanné C. 1991. Degradation of ^{14}C-labelled metalaxyl (CGA 48988) in one soil incubated under various experimental conditions. RCC AG., Switzerland. Report 262315. Syngenta File N° CGA48988/3091. Unpublished.

26831. Das R. 1994. Report on general physico-chemical properties (technical grade active ingredient). Ciba-Geigy, Switzerland. Report 26831. Syngenta File N° CGA329351/0020. Unpublished.

26833. Stulz J. 1994. Report on solubility in organic solvents. Ciba-Geigy, Switzerland. Report 26833. Syngenta File N° CGA329351/0022. Unpublished.

30/77. Gross D. 1977. Metabolism of CGA 48988 in field grown potato plants. Ciba-Geigy, Switzerland. Report 30/77. Syngenta File N° CGA48988/2022. Unpublished.

35/94. Ellgehausen H. 1994. Comparison of rate of degradation of [U-^{14}C]-phenyl CGA 48988 with CGA 329351 in soil under laboratory conditions. Ciba-Geigy Ltd, Switzerland. Report 35/94. Syngenta File N° CGA48988/3745. Unpublished.

38/79. Gross D. 1979. Fate of CGA 48988 in lettuce. Ciba-Geigy, Switzerland. Report 38/79. Syngenta File N° CGA48988/2019. Unpublished.

38/80. Gross D. 1980. Identification of degradation products of CGA 48988 (Ridomil) in lettuce. Ciba-Geigy, Switzerland. Report 38/80. Syngenta File N° CGA48988/2020. Unpublished.

39/79. Gross D. 1979. Identification of metabolites of CGA 48988 (Ridomil®) in field grown potato plants. Ciba-Geigy, Switzerland. Report 39/79. Syngenta File N° CGA48988/2025. Unpublished.

4012/99. Kühne RO. 2000. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in Spain. Novartis Crop Protection, Switzerland. Report 4012/99. Syngenta File N° CGA329351/1344. Unpublished.

4013/99. Kühne RO. 2001. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in France (north). Syngenta Crop Protection, Switzerland. Report 4013/99. Syngenta File N° CGA329351/1447. Unpublished.

4014/99. Kühne RO. 2001. Residue study with metalaxyl-M (CGA 329351) in or on sunflowers in France (north). Syngenta Crop Protection, Switzerland. Report 4014/99. Syngenta File N° CGA329351/1448. Unpublished.

517/99. Kühne RO. 1999. Validation data of the method REM 181.01 (Including also the version modified for use with LC-MS-MS). Validation by analysis of fortified specimens and determination of recoveries: discussion and evaluation of previously reported results. Substrate citrus crops (lemons, mandarins, oranges). Novartis Crop Protection, Switzerland. Report 517/99. Syngenta File N° CGA329351/1229. Unpublished.

518/99. Kühne RO. 1999. Validation data of the method REM 181.01 (Including also the version modified for use with LC-MS-MS). Validation by analysis of fortified specimens and determination of recoveries: discussion and evaluation of previously reported results. Substrate cotton seeds. Novartis Crop Protection, Switzerland. Syngenta Report 518/99. Syngenta File N° CGA329351/1230. Unpublished.

519/99. Kühne RO. 1999. Validation data of the method REM 181.01 (Including also the version modified for use with LC-MS-MS). Validation by analysis of fortified specimens and determination of recoveries: discussion and evaluation of previously reported results. Substrate sunflower seeds. Novartis Crop Protection, Switzerland. Report 519/99. Syngenta File N° CGA329351/1231. Unpublished.

95EH03. Ellgehausen H. 1995. Comparison of rate of degradation of [U-^{14}C]-phenyl CGA 48988 with CGA 329351 in sandy soil Collombey under laboratory conditions. Ciba-Geigy Ltd, Switzerland. Report 95EH03. Syngenta File N° CGA48988/3827. Unpublished.

95EH04. Ellgehausen H. 1995. Aqueous photolysis of ^{14}C CGA 329351 under artificial sunlight conditions. Ciba-Geigy, Switzerland. Report 95EH04. Syngenta File N° CGA329351/0329. Unpublished.

95EH05. Ellgehausen H. 1996. Hydrolysis of ^{14}C-labelled CGA 329351 under laboratory conditions. Ciba-Geigy,

Switzerland. Report 95EH05. Syngenta File N° CGA329351/0330. Unpublished.

95EH06. Ellgehausen H. 1996. Metabolism of ^{14}C-CGA 329351 under aerobic conditions in two soils at 20°C. Project No. 95EH06. Ciba-Geigy Ltd., Switzerland. Syngenta File N° CGA329351/0326. Unpublished.

98/7/161. Dal Santo P, Adams S and Chaophrasy K. 1998. Determination of metalaxyl-M (CGA 329351) and copper residues in grapes, grape juice and wine following six applications of Ridomil Gold Plus to grapevines close to harvest.. Novartis Crop Protection, Australia. Report 98/7/1615. Syngenta File N° CGA329351/1016. Unpublished.

98JS30. Stingelin J. 2000. Comparative metabolism of [phenyl-(U)-^{14}C] CGA 329351 and [Phenyl-(U)-^{14}C] CGA 48988 in field grown lettuce. Novartis Crop Protection, Switzerland. Report 98JS30. Syngenta File N° CGA329351/1254. Unpublished.

ABR-78001. Marco JJ. 1978. Balance and metabolism of ^{14}C-CGA 48988 in potatoes. Ciba-Geigy, United States. Report ABR-78001. Syngenta File N° CGA48988/2023. Unpublished.

ABR-78013. Foster RA, Fischer WC and Cassidy JE. 1978. Uptake of ^{14}C-metalaxyl in potatoes grown in a field plot – preparation of rotational plots. Ciba Geigy Corp., United States. Report ABR-78013. Syngenta File N° CGA48988/2024. Unpublished.

ABR-78046. Fisher WC, Foster RA and Cassidy JE. 1978. Balance and metabolism of f-^{14}C-CGA-48988 in a lactating cow. M6-69-3A. Report ABR-78046. Ciba-Geigy, USA. Unpublished.

ABR-78077. Marco JJ. 1978. Uptake and characterization of Φ-^{14}C-CGA 48988 and its soil metabolites in rotation winter wheat. Ciba-Geigy Corp., United States. Report ABR-78077. Syngenta File N° CGA48988/3570. Unpublished.

ABR-78078. Marco JJ. 1978. Uptake and characterization of Φ-^{14}C-CGA 48988 and its soil metabolites in rotation lettuce. Ciba-Geigy Corp., United States. Report ABR-78078. Syngenta File N° CGA48988/3569. Unpublished.

ABR-79002. Marco JJ. 1979. Uptake and characterization of Φ-^{14}C-CGA 48988 and its soil metabolites in field rotation spring oats. Ciba-Geigy Corp., United States. Report ABR-79002. Syngenta File N° CGA48988/3568. Unpublished.

ABR-79003. Marco JJ. 1979. Uptake and characterization of Φ-^{14}C-CGA 48988 and its soil metabolites in field rotation soybeans. Ciba-Geigy Corp., United States. Report ABR-79003. Syngenta File N° CGA48988/3567. Unpublished.

ABR-79004. Marco JJ. 1979. Uptake and characterization of Φ-^{14}C-CGA 48988 and its soil metabolites in field rotation corn. Ciba-Geigy Corp., United States. Report No. ABR-79004. Syngenta File N° CGA48988/3566. Unpublished.

ABR-79005. Marco JJ. 1979. Uptake and characterization of Φ-^{14}C-CGA 48988 and its soil metabolites in field rotation sugar beets. Ciba-Geigy Corp., United States. Report ABR-79005. Syngenta File N° CGA48988/3565. Unpublished.

ABR-81014. Balasubramanian K and Ross JA. 1981. Validation of AG-349 for the determination of metalaxyl in animal tissues and milk. Ciba-Geigy, United States. Report ABR-81014. Syngenta File N° CGA48988/5085. Unpublished.

ABR-81037. Marco JJ. 1981. Uptake, balance and metabolism of f-^{14}C-CGA 48988 in field grown potatoes. Ciba-Geigy, United States. Report ABR-81037. Syngenta File N° CGA48988/2026. Unpublished.

ABR-82052. Kahrs RQ. 1982. Residues of metalaxyl and metabolites in tissues and milk of dairy cows receiving metalaxyl in their diet. Ciba-Geigy Corp., United States. Report No. ABR-82052. Syngenta File N° CGA48988/1982. Unpublished.

ABR-90077. Kennedy E, Brown K and Goins A. 1990. Metabolism of [f-^{14}C]-metalaxyl in hens. Project 409925. Report ABR-90077. Ceiba-Geigy, USA. Unpublished.

ABR-90078. Emrani J and Meadows W. 1990. Metabolism of [f-^{14}C]metalaxyl in goats. Project 409925. Report ABR-90078. Ciba_Geigy, USA. Unpublished.

ABR-90079. Itterly W and Eberle W. 1990. Characterization and identification of phenyl-[^{14}C]-metalaxyl metabolites in rats. Study M89-409-002A, M89-409-003A, M89-409-004A, M89-409-005A. Project 409925. Report ABR-90079. Ciba-Geigy, USA. Unpublished.

ABR-91008. Yokley RA. 1991. Validation of analytical method AG-576 for the determination of total residues of metalaxyl in goat tissues, milk, poultry tissues and eggs. Ciba-Geigy, United States. Report ABR-91008. Syngenta File N° CGA48988/3074. Unpublished.

ABR-91047. Eudy LW. 1991. Metalaxyl - Three level/28 day poultry study. Ciba-Geigy Corp., United States. Report No. ABR-91047. Syngenta File N° CGA48988/3167. Unpublished.

ABR-91075. Emrani J and Meadows W. 1991. Supplemental report of the metabolism of [f-^{14}C]metalaxyl in goats. Identification of the major milk metabolite "A". Project 409925. Report ABR-91075. Ciba_Geigy, USA. Unpublished.

ABR-91077. Kennedy E, Worsham C and Meadows W. 1991. Supplemental report on the metabolism of [f-^{14}C]-metalaxyl in hens. Project 409925. Report ABR-91077. Ceiba-Geigy, USA. Unpublished.

ABR-91084. McFarland J. 1992. Uptake and metabolism of metalaxyl in greenhouse rotational crops following target tobacco grown in soil treated with phenyl-^{14}C-metalaxyl. Ciba-Geigy Corp., United States. Report ABR-91084. Syngenta File N° CGA48988/3220. Unpublished.

ABR-91084 A1. Simoneaux B. 1994. Final report amendment on the uptake and metabolism of metalaxyl in greenhouse rotational crops following target tobacco grown in soil treated with [phenyl-^{14}C]-metalaxyl (Amendment 1). Ciba-Geigy Corp., United States. Report ABR-91084 A1. Syngenta File N° CGA48988/3599. Unpublished.

ABR-96108. Eudy LW and Swaim LF. 1997. Method validation trial for the determination of CGA-94689, P1/P2, and CGA-62826 in meat and eggs using analytical method AG-576. Project 409925, Study 77-95, report ABR-96108. Ciba-Geigy, USA. Unpublished.

ABR-98053. Gruenwald MC. 1998. Stability of CGA-329351, CGA 62826, and CGA-94689 in meat, milk, and eggs, under freezer storage conditions. Novartis Crop Protection, United States. Report ABR-98053. Syngenta File N° CGA329351/1006. Unpublished.

AG-349. Balasubramanian K. 1980. Analytical method for the determination of total residues of metalaxyl in animal tissues, milk and eggs as 2,6-dimethylaniline. Ciba-Geigy, United States. Method AG-349. Syngenta File N° CGA48988/0606. Unpublished.

AG-576. Eudy LW. 1991. Improved analytical method for the determination of total residues of metalaxyl in poultry tissues and eggs as 2,6-dimethylaniline. Ciba-Geigy, United States. Method AG-576. Syngenta File N° CGA48988/3073. Unpublished.

BIOL-90016. McFarland J. 1992. Biological report for greenhouse rotational crops grown after tobacco in soil treated with ^{14}Cmetalaxyl. Ciba-Geigy Corp., United States. Report BIOL-90016. Syngenta File N° CGA48988/3218. Unpublished.

BIOL-90017. McFarland J. 1992. Biological report for greenhouse grown tobacco grown in soil treated with ^{14}C-metalaxyl.Ciba-Geigy Corp., United States. Report BIOL-90017. Syngenta File N° CGA48988/3219.Unpublished.

FAO. 1992. Metalaxyl technical. FAO Specification 365/TC/S/F (1992).

FR 017/96. Machado TR. 1998. Ridomil Gold MZ, CGA 329351 + mancozeb, A-9407 B, potatoes, Brazil. Novartis Biociências S/A, Taboão da Serra SP, Brazil. Report FR 017/96 and FR 018/96. Syngenta File N° CGA329351/1821. Unpublished.

FR 018/96. See FR 017/96

FR 033/96. Machado TR. 1998. Ridomil Gold MZ, CGA 329351 + mancozeb, A-9407 B, onions, Brazil. Novartis Biociências S/A, Taboão da Serra SP, Brazil. Report FR 033/96 and FR 034/96. Syngenta File N° CGA329351/1820. Unpublished.

FR 034/96. See FR 033/96

FR0595AR. Adams S. 1998. Determination of residues of CGA 329351 and fluazinam in potatoes, after five applications of either A-9575A or Shirlan in the UK. Novartis, United Kingdom. Residue study FR0595AR. Syngenta File N° CGA329351/0836. Unpublished.

FR0595BR. Adams S. 1998. Determination of residues of CGA 329351 and fluazinam in potatoes, after five applications of either A-9575A or Shirlan in the UK. Novartis, United Kingdom. Residue study FR0595BR. Syngenta File N° CGA329351/0835. Unpublished.

gr 22998. Smith JA. 1999. Determination of residues of CGA 329351 and folpet in field grown lettuce , Germany. Novartis Agro, Germany. Report gr 22998. Syngenta File N° CGA329351/1154. Unpublished.

gr 23998. Smith JA. 1999. Determination of residues of CGA 329351 and folpet in glasshouse grown lettuce. Novartis Agro, Germany. Report gr 23998. Syngenta File N° CGA329351/1162. Unpublished.

gr 24998. Smith JA. 1999. Determination of residues of CGA 329351 and folpet in spinach, Germany. Novartis Agro, Germany. Report gr 24998. Syngenta File N° CGA329351/1152. Unpublished.

gr 25998. Smith JA. 1999. Determination of residues of CGA 329351 and folpet in spinach, Germany. Novartis Agro, Germany. Report gr 25998. Syngenta File N° CGA329351/1153. Unpublished.

gr 45396. Smith JA. 1998. Residues of CGA 329351 + CGA 143268 in potatoes (Test product: CGD 20660 F - A9575A, EC 600). Study report gr 45396. Novartis, Germany. Syngenta File N° CGA329351/0646. Unpublished.

gr 46696. Smith JA. 1998. Residues of CGA 329351 + CGA 143268 in potatoes (Test product: CGD 20660 F - A9575A, EC 600). Study report gr 46696. Novartis, Germany. Syngenta File N° CGA329351/0644. Unpublished.

gr 4795. Raum J and Smith J. 1996. Field trial for the determination of residues of CGD 20500 F in potatoes. Study gr 4795. Ciba-Geigy, Switzerland. Experiments gr 31395, gr 41395. Syngenta File N° CGA329351/0379. Unpublished.

gr 4895. Smith JA. 1997. Field trial for the determination of residues of CGA 329351 + fluazinam in potatoes (Test product: CGD 20660 F). Project report gr 4895. Novartis, Germany. Experiments gr 31495, gr 41495. Syngenta File N° CGA329351/0645. Unpublished.

gr 49500. Smith JA. 2001. Determination of residues of CGA 329351 and folpet in lettuce, Germany. Novartis Agro, Germany. Report gr 49500. Syngenta File N° CGA329351/1454. Unpublished.

gr 50597. Smith JA. 1999. Determination of residues of metalaxyl-M and folpet in comparison to metalaxyl and folpet in grapes, must and white wine. Novartis, Germany. Report gr 50597. Syngenta File N° CGA329351/1137. Unpublished.

gr 50800. Smith JA. 2001. Determination of residues of CGA 329351 and folpet in lettuce, Germany. Novartis Agro, Germany. Report gr 50800. Syngenta File N° CGA329351/1452. Unpublished.

gr 51900. Smith JA. 2001. Determination of residues of CGA 329351 and folpet in field grown lettuce, Germany. Novartis Agro, Germany. Report gr 51900. Syngenta File N° CGA329351/1453. Unpublished.

gr 5194. Leiblein M. 1995. Residues of CGA 329351 and folpet in grapes, must, and wine. Ciba-Geigy, Germany. Report GR 5194. Includes gr 52894 and gr 52994. Syngenta File N° CGA329351/0198. Unpublished.

gr 52700. Smith JA. 2001. Determination of residues of CGA 329351 and folpet in lettuce, Germany. Novartis Agro, Germany. Report gr 52700. Syngenta File N° CGA329351/1451. Unpublished.

gr 52894. See gr 5194.

gr 52994. See gr 5194.

HWI 6117-280. Fathulla R. 1995. Aerobic soil metabolism of ^{14}C-CGA 329351 in a sandy loam soil. Corning Hazleton Inc. Madison, United States. Report HWI 6117-280. Syngenta File N° CGA329351/0343. Unpublished.

NOV/MET/00111. Pointurier R. 2001. Independent laboratory validation (ILV) - validation of method REM 181.06 (validation by analysis of fortified specimens and determination of recoveries). ADME - Bioanalyses, Vergèze, France. Report NOV/MET/00111. Syngenta File N° CGA329351/1469. Unpublished.

NOV-0015. Weber H and Pelz S. 2000. Determination of the residues of CGA 329351 + folpet in lettuce - analytical part (CGA 329351 + folpet) of Novartis GLP study no gr 50800. Analytical report. Specht & Partner, Germany. Unpublished.

NOV06. Dorn R and Hein W. 2003. Degradation of CGA 62826 in soil “Birkenheide”. Staatl. Lehr-und Forschungsanstalt für Landwirtschaft, Weinbau und Gartenbau (SLFA). Nuestadt, Germany. Report NOV06. Syngenta File N° CGA62826/0023. Unpublished.

NOV07. Dorn R and Hein W. 2003. Degradation of CGA 329351 in soil "Birkenheide". Staatl. Lehr-und Forschungsanstalt für Landwirtschaft, Weinbau und Gartenbau (SLFA). Nuestadt, Germany. Report NOV07. Syngenta File N° CGA329351/1744. Unpublished.

NOV11. Dorn R. 2001. Degradation of CGA 48988 in soil "Birkenheide". Staatl. Lehr-und Forschungsanstalt für Landwirtschaft, Weinbau und Gartenbau (SLFA). Nuestadt, Germany. Report NOV11. Syngenta File N° CGA48988/5019. Unpublished.

PP-94/45P.DCW. Jäkel K.1995. Report on dissociation constant in water. Ciba-Geigy, Switzerland. Report PP-94 / 45P.DCW. Syngenta File N° CGA329351/0041. Unpublished.

PP-94/45P.VPC. Geoffroy A. 1994. Report on vapour pressure curve. Ciba-Geigy, Switzerland. Report PP-94/45P.VPC. Syngenta File N° CGA329351/0044. Unpublished.

REM 181.01. Kühne RO. 1995. CGA 329351. Determination of parent compound by gas chromatography (GC). Plant material. Ciba-Geigy, Switzerland. Method REM 181.01. Syngenta File N° CGA329351/0216. Unpublished.

REM 181.06. Kühne RO. 2001. Metalaxyl-M (CGA 329351). Plant material. Determination of parent compound by GC (enantiomer specific method). Syngenta, Switzerland. Method REM 181.06. Syngenta File N° CGA329351/1463. Unpublished.

RFFA 06/97. Garozi MJ. 1998. Fólio Gold PM, CGA 329351 + chlorothalonil, A-9857 A, potatoes, Brazil. Universidade Federal do Espirito Santo, Rio de Janeiro, Brazil. Report RFFA 06/97. Syngenta File N° CGA329351/1825. Unpublished.

RFFA 09/97. Garozi MJ. 1998. Folio Gold PM, CGA 329351 + chlorothalonil, A-9857 A, onions, Brazil. Universidade Federal do Espirito Santo, Rio de Janeiro, Brazil. Report RFFA 09/97. Syngenta File N° CGA329351/1826. Unpublished.

RFLU 06/97. Machado TR. 1998. Fólio Gold , CGA 329351 + chlorothalonil, A-9857 A, potatoes, Brazil. Novartis Biociências S/A, Taboão da Serra SP, Brazil. Report RFLU 06/97. Syngenta File N° CGA329351/1828. Unpublished.

RFZO 09/97. Machado TR. 1998. Fólio Gold, CGA 329351 + chlorothalonil, A-9857 A, onions, Brazil. Novartis Biociências S/A, Taboão da Serra SP, Brazil. Report RFZO 09/97. Syngenta File N° CGA329351/1827. Unpublished.

WHO. 2003. Metalaxyl and metalaxyl-M. Pesticide Residues in Food - 2002. Toxicological Evaluations. 165-221. WHO/PCS/03.1.

METHAMIDOPHOS (100)

The 2003 JMPR evaluated supporting information on cucumbers and concluded that the residue data were insufficient to estimate maximum residue limits for acephate or for methamidophos arising from use of acephate. The present Meeting noted that the report of the 2003 JMPR, while stating that the number of trials was inadequate for the purposes of estimating a maximum residue level for acephate on cucumber, made no recommendation with respect to the existing recommended MRL for methamidophos arising from the use of acephate.

RECOMMENDATION

The Meeting recommended that the draft MRL of 1 mg/kg for cucumber (CCN: VC0424) be withdrawn.

METHOMYL (094)

First draft prepared by Yibing He,
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EXPLANATION

A residue review of methomyl was conducted in 1975 and supervised field trial data and related data on various crops were considered in 1976-1978, 1986-1991 and most recently in 2001. At its 36th Session (2004), the CCPR noted that new data on mint hay and peppers had been reported and decided to maintain the CXLs for these for four years under the Periodic Review procedure and the evaluation was scheduled for 2004 (ALINORM 04/27/24, para. 124, p 15).

The Meeting received residue data from supervised field trials on peppers and mint hay from the manufacturer. Information on labels and current GAP was also provided.

RESIDUE ANALYSIS

Analytical methods

In the 2001 evaluation of methomyl, a limit of quantification (LOQ) of 0.02 mg/kg was reported for a number of plant commodities. Quantification was by GC with an FPD (sulphur mode) or HPLC with a fluorescence detector.

Peppers. In the method of Pease and Kirkland (1968) residues of methomyl were extracted with ethyl acetate using a homogenizer, the extract centrifuged, and the supernatant decanted. The combined organic extracts were purified by liquid-liquid partitioning and concentrated. Sodium hydroxide was added to hydrolyse methomyl to *S*-methyl *N*-hydroxythioacetimidate (MHTA), which was then extracted with ethyl acetate. Analysis of the final extract was by GC with an FPD (sulphur mode). Recoveries from samples fortified at 0.04-4.0 mg/kg were 80-110% (Ashley, 2001a,b). The LOQ was 0.02 mg/kg.

The method of Rühl and Clark (1994) with slight modifications to the HPLC gradient conditions was also used for determination of methomyl residues in peppers (Françon and Larcinese, 1999). Residues of methomyl were extracted with acetonitrile using a homogenizer. Sodium chloride was added to the extract for phase separation and direct partitioning of methomyl to the acetonitrile layer. The acetonitrile extract was purified by hexane partitioning and passed through a Florisil solid-phase extraction cartridge. Following elution with 50:50 acetone:hexane, the eluate was evaporated to dryness, reconstituted in 15:85 acetonitrile:water and filtered. The final extract was analysed by HPLC with post-column reaction to convert methomyl to methylamine which was derivatized (on-line) and detected by fluorescence. Average recoveries (n=17) from samples fortified at 0.020 and 0.20 mg/kg were 95 ± 13% and 90 ± 12% respectively. The LOQ was 0.02 mg/kg.

Mint hay. The analytical method developed for mint hay (Bishel, 2003) was based on the method of Pease and Kirkland (1968). Mint hay samples were macerated with ethyl acetate, the extracts filtered, water added and the organic solvent removed by evaporation. The aqueous phase was filtered, acidified, and washed with hexane. This was followed by extraction of methomyl into dichloromethane. Sodium hydroxide was added to hydrolyze methomyl to MHTA, and the hydrolysis mixture was acidified and extracted with dichloromethane or ethyl acetate. The extract was dried and concentrated for GC with an FPD (sulphur mode). Recoveries from samples fortified at 0.08-2.0 mg/kg were 64-100%. The LOQ

was 0.02 mg/kg.

Stability of residues in stored analytical samples

The freezer storage stability of methomyl in mint hay was studied (Bishel, 2003). Samples of mint hay fortified with methomyl at 0.1, 0.2, 0.5 and 13.9 mg/kg were stored at –10°C and analysed after 20, 49, 181 and 192 days. The results are shown in Table 1.

Table 1. Stability of residues in mint hay fortified with methomyl.

Sample	Fortification, mg/kg	Storage (°C)	Storage (days)	Methomyl remaining (%)	Reference
Fresh and spent mint hay	13.9	-10	20	101	3E 1303-2003
	0.1	-10	49	113	
	0.2	-10	49	95	
	0.5	-10	181	92	
	0.2	-10	192	91	

USE PATTERN

Methomyl is registered for use as a pesticide to control a large variety of chewing and sucking insects on a wide range of crops in many countries. Table 2 is a summary of the registered uses of methomyl on mint and peppers based on labels or label translations provided by the manufacturer.

Table 2. Registered uses of methomyl on mint and peppers.

Crop	Country	F/G[1]	Form type	Concentration	Application method	Rate kg ai/ha	Spray conc., kg ai/hl	No. of applications	PHI (days)
Mint	USA	F	SL	290 g/l	Foliar – aerial/ ground	0.75 – 1.0	6.8 – 9.1	4	14
	USA	F	SP	900 g/kg	Foliar – aerial/ ground	0.75 – 1.0	6.8 – 9.1	4	14
Pepper	Bulgaria	F	WP	250 g/kg	Foliar		0.023	3	7
	Central America	F	SP	900 g/kg	Foliar	0.45	0.11–0.23	ns	3
	Cyprus	F	SP	900 g/kg	Foliar – high volume	0.42–0.81	0.042-0.081	2 – 3	10
	Cyprus	F	SP	900 g/kg	knapsack	1.00	0.2	2 – 3	10
	Egypt	F	SP	900 g/kg	Foliar	0.64		ns	3
	Greece	F/G	SL	200 g/l	Foliar	0.45		1 – 3	15
	Greece	F/G	SL	200 g/l	soil spraying; incorporated	2.7		1	15
	Greece	F/G	SL	200 g/l	soil spraying followed by irrigation	0.72 - 0.9		1	15
	Greece	F/G	SP	900 g/kg	Foliar	0.45		1 - 3	15
	Greece	F/G	SP	900 g/kg	soil spraying; incorporated	2.7		1	15
	Greece	F/G	SP	900 g/kg	soil spraying followed by irrigation	0.72 - 0.9		1	15
	Greece	F/G	WP	250 g/kg	Foliar	0.45		1 - 3	15
	Greece	F/G	WP	250 g/kg	soil spraying; incorporated	2.7		1	15

Crop	Country	F/G[1]	Form type	Concentration	Application method	Rate kg ai/ha	Spray conc., kg ai/hl	No. of applications	PHI (days)
	Greece	F/G	WP	250 g/kg	soil spraying followed by irrigation	0.72 - 0.9		1	15
	Hungary	G	SL	200 g/l	Foliar	0.72	0.07 - 0.09	ns	5
	Italy	F	SL	200 g/l	Foliar		0.04	ns	10
	Italy	F	WP	250 g/kg	Foliar		0.04	ns	10
	Jordan	F	SP	900 g/kg	Foliar		0.05	ns	7
	Kenya	F	SP	900g/kg	Foliar	0.45 – 0.9	0.09	As needed	5
	Mexico	F	SL	290 g/l	Foliar - ground	0.65	0.16 - 0.33	ns	3
	Mexico	F	SL	290 g/l	Foliar - aerial	0.65	0.81 - 1.3	ns	3
	Morocco	F	WP	250 g/kg	Foliar		0.0375	ns	7
	New Zealand	G	SL	200 g/l	Foliar		0.024	4	2
	Tunisia	F	WP	250 g/kg	Foliar	0.038	0.038	3 - 4	7
	USA	F	SL	290 g/l	Foliar - aerial/ground	0.25 - 1.0	2.3 - 9.1	10	3
	USA	F	SP	900 g/kg	Foliar - aerial/ground	0.25 - 1.0	2.3 - 9.1	10	3
	Venezuela	F	SL	223 g/l	Foliar	0.45		ns	1
	Venezuela	F	SL	288 g/l	Foliar	0.5		ns	1
	Venezuela	F	SP	900 g/kg	Foliar	0.27		ns	1
	Yugoslavia		SP	900 g/kg	Foliar		0.045	As needed	14
Pepper, Chili	India	F	SL	112 g/l	Foliar - high volume	0.04 - 0.05	0.04 -0.10	ns	4
	Peru	F	SL	240 g/l	Foliar	0.24	0.096	ns	5
	Peru	F	SP	400 g/kg	Foliar	0.4	0.135	ns	7
	Peru	F	SP	900 g/kg	Foliar	0.4	0.135	ns	7
	Thailand	F	SP	400 g/kg	Foliar		0.04 - 0.07	ns	6 - 14
Pepper, Green	Romania	G	SP	900 g/kg	Foliar	0.45	0.045	3	3
Pepper, Green (field)	Japan	F	WP	450 g/kg	Foliar	0.3375 -1.35	0.0225 -0.045	1 - 3	14
Pepper, Red	Korea	F	SL	215 g/l	Foliar		0.0215	4	14
	Korea	F	SP	450 g/kg	Foliar		0.0675	3	7
Pepper, Red and Green	Argentina	F	SP	900 g/kg	Foliar	0.225 -0.45	0.75 - 0.15	ns	10
	Argentina	F	SP	900 g/kg	Foliar – knapsack		0.0225 -0.045		10

[1] Outdoor or field use (F) or glasshouse application (G).
ns: not stated

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue levels and application rates were reported as methomyl. Where residues were not detected, they are reported as below the LOQ, e.g. <0.02 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Although trials included control plots, no control data are given in the Tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for % recovery. Multiple results are recorded in the Tables where the trial included replicate plots and where separate samples have been identified as being from these replicate plots.

Supervised field trials were reported for peppers and mint hay. Trials are listed in the following Tables. Double-underlined residues are from trials according to GAP used to estimate maximum residue levels.

Peppers. Supervised trials on pepper were conducted in the USA in 1968 and 1970-74, and in Canada in

1971 (Ashley, 2001a,b). US GAP specifies 0.50-1.0 kg ai/ha and a PHI of 3 days. There is no GAP in Canada. Samples were collected at intervals and analysed by GC with sulphur FPD (Pease and Kirkland, 1968). Recoveries from fortified peppers were acceptable in the 0.04-4.0 mg/kg range (Ashley 2001a, b). The results are shown in Table 3.

Table 3. Residues of methomyl in or on peppers after foliar applications in the USA and Canada.

Location/Year/ Variety	Application					PHI, days	Residues, mg/kg	Ref (DuPont report no.)
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
Country label	SL, SP	1.0	NSOL[1]	-[2]	≤10	PHI: 3 day Note: Do not apply more than 5.0 kg ai/ha/crop		
GAP: Canada	None(use USA)							
Bradenton, FL USA/1968/ -	-	0.56	-	-	6	3 5	0.39 0.38	9F 0814
Bristol, MD USA/1968/-	-	0.56	-	-	5	3	0.10	9F 0814
Niles, MI USA/1968/ -	-	0.56	-	-	4	Control 2 4	0.06 0.28 0.12	9F 0814
Niles, MI USA/1968/ -	-	1.1	-	-	4	2 4	0.44 0.11	9F 0814
Weslaco, Texas USA/1968/-	-	0.56	-	-	8	Control 10	0.06 0.05	9F 0814
Weslaco, Texas USA/1968/-	-	1.1	-	-	8	10	0.03	9F 0814
Weslaco, Texas USA/1968/-	-	2.2	-	-	8	10	0.19	9F 0814
Wilmington, DE USA/1968/ -	-	0.56	-	-	9	2 5	0.16 0.05	9F 0814
Wooster, OH USA/1968/-	-	0.56	-	-	6	2 5	0.02 0.03	9F 0814
Wooster, OH USA/1968/-	-	1.1	-	-	6	2 5	0.08 0.03	9F 0814
Bradenton, FL USA/1970/Bell	SP, 900g/kg	1.1	0.12	898	1✈	1 3 5	0.11 0.10 0.04	5F 1616
Niles, MI USA/1970/Bell	SP, 900g/kg	0.50	0.09	561	2✈	1 3	0.41 0.13	5F 1616
Riverside, CA USA/1970/Chili	SP, 900g/kg	1.1	0.20	561	3	Control 0 7 14	0.02 1.3, 1.8 0.74, 0.35 0.19, 0.22	5F 1616
Westley, CA USA/1970/-	-	1.1			1	1 3 5	0.05 0.04 <0.02	5F 1616
Bakersfield, CA USA/1971/Bell	SP, 900g/kg	0.84	0.22	374	1✈	1 2	0.37 0.11	5F 1616
London, Ont Canada/1971/Bell	SP, 900g/kg	0.76	0.20	374	6	1 3 5	0.07 0.02 0.02	5F 1616
Modesto, CA USA/1971/Bell	SP, 900g/kg	1.1	0.12	935	3✈	3 7	0.03 0.03	5F 1616
Santa Maria, CA USA/1971/Bell	SP, 900g/kg	1.1	-	-	1✈	0 1 3 5	3.2 1.2 0.11 0.09	5F 1616
Immokalee, FL USA/1972/Bell	SP, 900g/kg	0.56	0.06	935	1✈	Control 1 3	0.07 0.34 0.29	5F 1616

Location/Year/ Variety	Application					PHI, days	Residues, mg/kg	Ref (DuPont report no.)
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
Immokalee, FL USA/1972/Bell	SP, 900g/kg	1.1	0.12	935	1✈	1 3	0.46 0.39	5F 1616
Immokalee, FL USA/1972/Bell	L, 216g/l	0.56	0.06	935	1✈	1 3	0.26 0.14	5F 1616
Immokalee, FL/ USA1972/Bell	L, 216g/l	1.1	0.12	935	1✈	1 3	0.29 0.26	5F 1616
Naples, FL USA/1973/Bell	L, 216g/l	1.0	1.5	65	10	1 3	0.49 0.12	5F 1616
Naples, FL USA/1973/Bell	L, 216g/l	1.0	1.5	65	11	1 3	0.23 0.24	5F 1616
Immokalee, FL USA/1974/Bell	L, 216g/l	1.0	0.11	935	5✈	1 3	0.54 0.18	5F 1616
Naples, FL USA/1974/Bell	L, 216g/l	2.0	0.21	935	5	1 3 7	2.2 0.46 0.44	5F 1616
Rio Grande City, TX USA/1974/Bell	L, 216g/l	0.75	-	-	2✈	1 3	0.13 0.10	5F 1616
Rio Grande City, TX USA/1974/Bell	L, 216g/l	1.0	-	-	2✈	1 3	0.06 0.04	5F 1616
Weslaco, TX/ USA/1974/ Bell	L, 216g/l	1.0	2.1	47	5✈	0 3	0.18 0.08	5F 1616
Weslaco, TX USA/1974/Bell	L, 216g/l	1.0 + 2.0	2.1 + 4.3	47	3 + 2	0 3	0.43 0.15	5F 1616

[1]not specified on label.
[2]not available.
✈Aerial application.

Field trials on peppers were reported from Italy in 1996 (Weidenauer *et al.*, 1998), and from Italy, France, Portugal, Spain and Greece in 1997 (Françon and Larcinese, 1999). WP and SL formulations were applied at 10-day intervals as foliar sprays in Italy at 0.40 kg ai/hl. GAP in Greece specifies 0.45 kg ai/ha and a PHI of 15 days. There is no GAP in France, Portugal or Spain. Pepper samples were collected and analysed by method AMR 3015-94 (Rühl and Clark, 1994). The analytical laboratory made slight modifications during analysis (Françon and Larcinese, 1999). Adequate recovery was demonstrated at 0.02 mg/kg fortification of control samples (Françon and Larcinese, 1999). The results are shown in Table 4.

Table 4. Residues of methomyl in or on peppers after foliar applications of WP or SL formulations in Italy, France, Spain, Portugal and Greece.

Location/Year/ Variety	Application					PHI, days	Residues, mg/kg	Ref (DuPont report no)
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
GAP: Italy	SL, WP	NSOL[1]	0.04	-[2]	NSOL	PHI: 10 day Country label		Country label
GAP: France	None(use Italy)							
GAP: Portugal	None(use Italy)							
GAP: Spain	None(use Italy)							
GAP: Greece	SL, WP	0.45	NSOL		1-3	PHI: 15 day		Country label
Montanaso, Lombardo (LO) Italy/1996/Indalo	SL, 200 g/l	0.50	0.050	990	3	7	<0.02, <0.02	AMR 3999-96
Montanaso, Lombardo (LO) Italy/1997/Indalo F1	SL, 200 g/l	0.40	0.050	798	3	+3h 1 3 5 7	0.04, 0.05 0.02, <0.02 <0.02, <0.02 <0.02, <0.02 0.02, 0.02, <0.02	AMR 4509-97

Location/Year/ Variety	Application					PHI, days	Residues, mg/kg	Ref (DuPont report no)
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.			
Montanaso, Lombardo (LO) Italy/1997/ Indalo F1	WP, 250 g/kg	0.40	0.050	800	3	+3h 1 3 5 7	0.06, 0.03 0.04, <0.02 <0.02, 0.02 <0.02, <0.02 <0.02, <0.02	AMR 4509-97
Bren, Valence Area France/1997/Le Muyo	SL, 200g/l	0.27	0.050	535	3	+3h 1 3 5 7	0.03, 0.06 <0.02, 0.02 <0.02, <0.02 <0.02, <0.02 <0.02, <0.02	AMR 4509-97
St Donat sur l'Herbasse, Valence Area France/1997/La Muyo	WP, 250g/kg	0.26	0.050	516	3	+3h 1 3 5 7	0.03, 0.03 0.02, 0.02 <0.02, <0.02 <0.02, <0.02 <0.02, <0.02	AMR 4509-97
Bela Curral, Pechão Portugal/1997/ Lamuyo	SL, 200g/l	0.50	0.051	982	3	+3h 1 3 5 7	0.07, 0.05 0.05, 0.02 0.03, 0.02 0.03, 0.02 <0.02, <0.02	AMR 4509-97
Utrera, Sevilla Spain/1997/Italico	SL, 200g/l	0.35	0.051	690	3	+3h 1 3 5 7	0.14, 0.11 0.08, 0.07 0.06, 0.04 0.02, 0.04 <0.02, 0.03	AMR 4509-97
Utrera, Sevilla Spain/1997/Italico	WP, 250g/kg	0.35	0.051	694	3	+3h 1 3 5 7	0.14, 0.12 0.08, 0.08 0.04, 0.04 0.02, 0.02 0.02, <0.02	AMR 4509-97
Palacios, Sevilla Spain/1997/Itallico	WP, 250g/kg	0.38	0.051	742	3	7	0.04, 0.03	AMR 4509-97
Metohi, Epanomis Greece/1997/Veria P-14	SL, 200 g/l	0.26	0.050	512	3	+3h 1 3 5 7	0.25, 0.11 0.06, 0.06 0.02, <0.02 <0.02, <0.02 0.02, 0.02, <0.02	AMR 4509-97
Metohi, Epanomis Greece/1997/Veria P-14	WP, 250g/kg	0.25	0.052	491	3	+3h 1 3 5 7	0.12, 0.10 0.09, 0.04 0.03, 0.03 0.02, 0.03 <0.02, <0.02	AMR 4509-97

[1]not specified on label.
[2]not available.

Mint hay. Supervised field trials on the foliar application of methomyl to spearmint or peppermint to determine residues in mint hay were conducted in the USA at 5 locations (Bishel, 2003). GAP in the USA specifies 0.75-1.0 kg ai/ha and a PHI of 14 days. Mint hay samples were collected at intervals and analysed by method 3E 1303 (Bishel, 2003), developed on the basis of the method of Pease and Kirkland (1968). Recoveries from fortified mint hay were acceptable in the 0.08-2.0 mg/kg range (Bishel, 2003). The results are shown in Table 5.

Table 5. Residues of methomyl in or on mint hay after foliar applications of SP or EC formulations to mint in the USA (3E 1303).

Location/Year/Variety	Application					PHI, days	Residues, mg/kg
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		
US GAP	SL, SP	1.0	NSOL[1]	-[2]	≤4	PHI: 14 days Note: Do not apply more then 2.0 kg ai/ha/crop	
Jasper County, IN USA/1970/-	EC, 360 g/l	0.56	0.30	187	1	22 23	1.5 0.12[3]
Jasper County, IN USA/1970/-	EC, 360 g/l	0.56	0.30	187	2	8 9	3.6, 3.6 0.28[3]
Jasper County, IN USA/1970/-	EC, 360 g/l	1.1	0.60	187	2	8 9	5.2, 5.5 0.34[3]
Jefferson, OR USA/ 1970/-	SP, 900 g/kg	0.56	0.60	94	1✈	0 7 14 19	2.1 <0.02 <0.02 <0.02, <0.02[3]
Jefferson, OR USA/ 1970/-	SP, 900 g/kg	1.1	1.2	94	1✈	0 7 14 19	4.2 0.02 <u>0.02</u> <0.02, <0.02[3]
Kings Valley, OR USA/1970/-	SP, 900 g/kg	0.56	0.60	94	1✈	0 7 14 18	7.5 0.66 0.12 <0.02, 0.05[3]
Kings Valley, OR USA/1970/-	SP, 900 g/kg	1.1	1.2	94	1✈	0 7 14 18	14 1.2 <u>0.28</u> 0.02, 0.08[3]
Pulasky County, IN USA/1970/-	EC, 360 g/l	0.56	0.30	187	1	22 23	0.12 0.08[3]
Pulasky County, IN USA/1970/-	EC, 360 g/l	1.1	0.60	187	1	22 23	0.18, 0.16 0.11[3]
Pulasky County, IN USA/1970/-	EC, 360 g/l	0.56	0.30	187	2	8 9	0.37, 0.40 0.29[3]
Pulasky County, IN USA/1970/-	EC, 360 g/l	1.1	0.60	187	2	8 9	0.55, 0.54 0.36[3]
Corvallis, OR USA/1971/ -	EC, 360 g/l	0.56	-	-	1✈	26	<0.02, <0.02[3]
Corvallis, OR USA/1971/ -	EC, 360 g/l	1.1	-	-	1✈	0 3 7 14 26	13 1.1 0.19 <u>0.02</u> <0.02, <0.02[3]
Corvallis, OR USA/1971/ -	EC, 360 g/l	2.2	-	-	1✈	0 3 7 14 26	28 1.6 0.55 0.07 0.02, 0.03[3]
Corvallis, OR USA/1971/ -	RB, 1%	0.28			1	13	0.09, <0.02[3]
Jasper County, IN USA/1971/-	SP, 900 g/kg	0.56	0.30	187	1	0 7 14 16	57 3.8 0.23 0.11[3]
Jasper County, IN USA/1971/-	SP, 900 g/kg	1.1	0.60	187	1	0 4 7 14 16	133 18 7.5 <u>0.16</u> <u>0.17</u>[3]

Location/Year/Variety	Application					PHI, days	Residues, mg/kg
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.		
Pulasky County, IN USA/1971/-	SP, 900 g/kg	0.56	0.30	187	1	0 3 7 14	10 1.5 0.18 0.02, 0.04[3]
Pulasky County, IN USA/1971/-	SP, 900 g/kg	1.1	0.60	187	1	0 3 7 14	21 2.6 0.31 0.02, 0.07[3]
Pulasky County, IN USA/1971/-	SP, 900 g/kg	0.56	0.30	187	2	14 14	0.03, 0.04 0.08[3], 0.06[3]
Pulasky County, IN USA/1971/-	SP, 900 g/kg	1.1	0.60	187	2	14 14	0.05, 0.07 0.13[3], 0.12[3]

[1]not specified on label.
[2]not available.
[3]spent mint hay.
✈Aerial application

NATIONAL MAXIMUM RESIDUE LIMITS

The manufacturer reported the following national MRLs for methomyl in peppers and mint hay.

Country	Commodity	MRL mg/kg
Argentina	Pepper; tomato; sweet corn	0.1
Australia	Mint	0.5
	Fruiting vegetables, other than Cucurbits	1
Indonesia	Peppers	1
Italy	Peppers	0.1
Japan	Bell Pepper	0.5
Korea	Peppers, Red	1
Netherlands	Paprika	0.05
Poland	Paprika	0.5
Thailand	Peppers	1
USA	Mint hay	2
	Peppers	2

APPRAISAL

Data on methomyl residues were reviewed in 1975, and data from supervised field trials with various crops and related data were considered in 1976, 1978, 1986, 1991 and 2001. At its Thirty-sixth Session, the CCPR noted that new data on mint hay and pepper had been reported and decided to maintain the CXLs for these commodities for 4 years under the periodic review procedure. The evaluation was scheduled for 2004 (ALINORM 04/27/24, para. 124, p 15).

The 2004 Meeting received data on residues from supervised field trials on pepper and mint hay from the manufacturer. Information on labels and current GAP was also provided.

Methods of analysis

The gas chromatographic method for measuring residues of methomyl in many plant commodities, evaluated by the 2001 JMPR, was validated for pepper and mint hay. This method consists of extraction with an organic solvent, liquid–liquid partition and hydrolysis with sodium hydroxide. The latter converts methomyl to methomyl oxime. The final extract is analysed by gas chromatography, usually with a flame photometric detector in the sulfur mode.

A more recent method is based on HPLC. The plant matrix is extracted with solvent, cleaned up on a Florisil column and analysed by HPLC with post-column reaction to convert methomyl to methylamine. Methylamine is derivatized (on-line) and detected by fluorescence.

The gas chromatographic method has been validated for numerous plant commodities at an LOQ of 0.02 mg/kg. The HPLC method and its modifications have been validated at an LOQ of 0.02 mg/kg for methomyl.

The Meeting concluded that adequate methods exist for the determination of methomyl in pepper and mint hay.

Stability of residues in stored analytical samples

As described by the 2001 JMPR, the stability of methomyl under frozen conditions has been demonstrated in a number crop samples, including broccoli, orange, apple and grape, for up to 24 months.

Data were presented on the stability of methomyl under frozen storage (–10°C) in mint hay. Adequate stability (> 90% remaining) was demonstrated after 6 months' storage.

The Meeting concluded that methomyl is stable under frozen conditions on mint hay and pepper.

Results of supervised trials on crops

Peppers

Supervised trials were conducted on peppers in Canada (no GAP) and the USA (GAP: 1.0 kg ai/ha, 3-day PHI). Fifteen trials (one in Canada, 14 in the USA) were conducted at US GAP, with residue concentrations of 0.02, 0.03, 0.04 (two), 0.08 (two), 0.10 (two), 0.11 (two), 0.12, 0.18, 0.24, 0.26, 0.39 and 0.44 mg/kg.

Supervised trials on peppers were conducted in France (no GAP), Greece (GAP: 0.45 kg ai/ha, 15-day PHI), Italy (GAP: 0.04 kg ai/hl, 10-day PHI), Portugal (no GAP) and Spain (no GAP). In nine trials (two in France, three in Italy, one in Portugal and three in Spain) conducted at about Italian GAP, the ranked order of concentrations was: < 0.02 (five), 0.02 (two), 0.03 and 0.04 mg/kg. The data from southern Europe and the USA were considered to represent different populations. Using only the data from the USA (higher values), the Meeting estimated an STMR value of 0.105 mg/kg, a highest residue of 0.44 mg/kg and a maximum residue level of 0.7 mg/kg, which replaces the previous estimate (1 mg/kg).

Mint hay

Supervised trials were conducted on fresh mint hay in the USA (GAP: 1.0 kg ai/ha, 14-day PHI, maximum of four applications). In the six trials conducted at GAP, the ranked order of concentrations of residues in fresh and spent mint hay was: 0.02 (three), 0.07 (two), 0.13, 0.16, 0.17, and 0.28 mg/kg. The dry matter in fresh and spent mint is 88%. The Meeting estimated an STMR value of 0.08 mg/kg, a highest residue value of 0.32 mg/kg and a maximum residue level of 0.5 mg/kg for mint hay on a dry weight basis. The Meeting agreed to withdraw the previous recommendation (2 mg/kg) and to replace it with the recommendation for mint hay (0.5 mg/kg, dry weight).

Residues in animal commodities

As mint hay is not considered to be a significant feed item, establishment of an MRL for mint hay would not significantly change the dietary exposure of animals. The Meeting therefore decided not to revise the previous MRL recommendations for animal commodities (edible offal, meat, milk) on the basis of the addition of mint hay (fresh and spent).

RECOMMENDATIONS

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

Definition of residue (for compliance with the MRL and for estimation of dietary intake): sum of thiodicarb and methomyl, expressed as methomyl.

Commodity		MRL, mg/kg		STMR	HR
CCN	Name	New	Previous	mg/kg	(mg/kg)
VO 0051	Peppers	0.7	1	0.105	0.44
AM 0738	Mint hay	0.5	2	0.08	0.32

DIETARY RISK ASSESSMENT

Long-term intake

The Meeting estimated a STMR value for pepper. This STMR value was used in combination with the STMR and STMR-P values estimated by the 2001 Meeting to calculate the long-term dietary intake of methomyl. The result is shown in Annex 3.

The dietary intakes in the five GEMS/Food regional diets, on the basis of the estimated STMRs values, represented 1-20% of the ADI (Annex 3-Report 2004). The Meeting concluded that the intake of residues of methomyl resulting from its uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI for methomyl was calculated for one food commodity for which maximum residue levels, STMR values and highest residues were estimated and for which data on consumption (of large portions and unit weight) were available. The result is shown in Annex 4.

The ARfD for methomyl is 0.02 mg/kg bw. The IESTI represented 20% of the ARfD for children and the general population. The Meeting concluded that the short-term intake of residues of methomyl, resulting from its use on peppers that has been considered by the JMPR, is unlikely to present a public health concern

REFERENCES

Ashley, R. M. 2001a. US-EPA Petition for Residue Tolerance: 5F 1616 with supporting field data. E.I. du Pont de Nemours and Company, Wilmington, Delaware, U.S.A. DuPont Report No. 5F 1616. Unpublished.

Ashley, R. M. 2001b. US-EPA Petition for Residue Tolerance: 9F 0814 with supporting field data. E.I. du Pont de Nemours and Company, Wilmington, Delaware, U.S.A. DuPont Report No. 9F 0814. Unpublished.

Bishel, D. A. 2003. US-EPA Petition for Residue Tolerance: 3E 1303 with supporting field data. E.I. du Pont de Nemours and Company, Wilmington, Delaware, U.S.A. DuPont Report No. 3E 1303. Unpublished.

Françon, B. and Larcinese, J.P. 1999. Combined Decline and Magnitude of Residues of Methomyl Insecticide in/on Solanacea Vegetables (Peppers) Grown in Southern Europe Following Treatment with Lannate 20L and/or Lannate 25WP - Season 1997. Battelle, Agricultural Product Department, Geneva, Switzerland. DuPont Report No. AMR 4509-97. Unpublished.

Pease, H.L. and Kirkland, J.J. 1968. Determination of methomyl residues using microcoulometric gas chromatography. J. Agric. Food Chem., 16: 554-557.

Rühl, J.C. and Clark S. 1994. Enforcement method for the Determination of Methomyl in dry Pea Seed and Hay, Sorghum Forage and Hay, Soybean Hay, and Sugar Beet Foliage. E.I. du Pont de Nemours and Company, Wilmington, Delaware, USA DuPont Report No. AMR 3015-94. Unpublished.

Weidenauer, M., Françon, B., and Larcinese, J.P. 1998. Combined Decline and Magnitude of Residues of Methomyl Insecticide in/on Solanacea Vegetables (Tomatoes and Peppers) Grown in Southern Europe following Treatment with LANNATE 20L and/or LANNATE 25WP - Season 1996 - (Analysis by HPLC). Battelle, Agricultural Product Department, Geneva, Switzerland. DuPont Report No. AMR 3999-96. Unpublished.

OXYDEMETON-METHYL (166)

First draft prepared by Tsuyoshi Sakamoto, Agricultural Chemicals Inspection Station, Tokyo 187-0011 Japan

EXPLANATION

Oxydemeton-methyl was evaluated for residues by the 1998 JMPR within the CCPR Periodic Review Programme, for residues by the 1999 Meeting and toxicology by the 2002 Meeting.

At the 31st (1999) Session of the CCPR, the JMPR was asked to clarify whether demeton-S-methyl and demeton-S-methylsulphon should remain in the definition of the residue of oxydemeton-methyl since it was believed that registration of these compounds would not be retained in the future.

At the 32nd Session of the CCPR, the Committee withdrew the draft MRLs for several commodities because there was no GAP for them, advanced the new proposed draft MRLs to Step 5 and returned other draft MRLs to Step 6 due to intake concerns, requesting more detailed information on support for oxydemeton-methyl. The Committee questioned the definition of the residue recommended by the 1999 JMPR and stated that as demeton-S-methyl was no longer supported and there was no GAP, it should not be included. However it was pointed out that demeton-S-methyl could not be distinguished from oxydemeton-methyl in analysis and could be generated from this compound during analytical processes. The Committee decided that the note "The residue definition and MRLs are based on the use of oxydemeton-methyl only" should be added to the residue definition.

At the 33rd Session the Committee noted the written comment from the EC expressing a general reservation (lack of an acute RfD) and specific reservations on MRLs for grapes, lemon and oranges, sweet, sour (acute risk) and decided to return the draft MRLs to Step 6.

At the 35th Session the Committee decided to return all MRLs to Step 6 pending the reporting of short-term intake calculations by the JMPR and the submission of data by the manufacturer to the 2004 JMPR for a review of the definition of the residue.

The present Meeting received new data on physical and chemical properties (partially updated information), analytical methods, fate of residues in processing, plant metabolism (apple), residue data (apples, pears, grapes, cabbage, Brussels sprouts, cauliflower, field peas, potatoes, sugar beet, fodder beet, wheat, barley, rape and sunflower), and information on GAP and on national MRLs.

IDENTITY

Physical and chemical properties (only new information is listed)

Pure active ingredient:

Physical state, colour	pale yellow liquid (Krohn, 1999)
Odour	slight mercaptan (Krohn, 1999)
Vapour pressure	3.9×10^{-5} hPa at 20°C (Krohn, 1999) 5.2×10^{-5} hPa at 25°C (Krohn, 1999)

Solubility in organic solvents, g/l, at 20°C (Krohn, 2002)

n-hexane	>0.1
toluene	>250

dichloromethane	>250
2-propanol	>250
1-octanol	>250
polyethylenoglycol + ethanol	>250
acetone	>250
dimethylformamide	>250

Relative density 1.31 (Krohn, 1999)

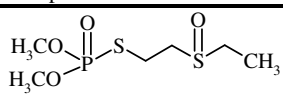
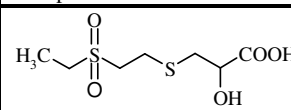
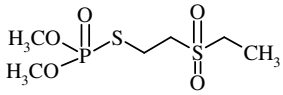
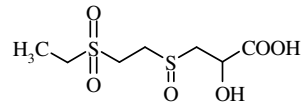
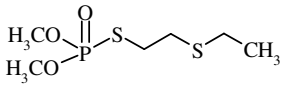
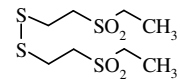
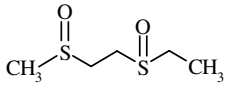
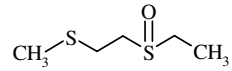
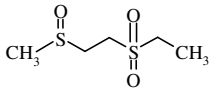
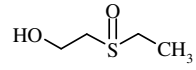
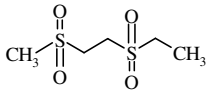
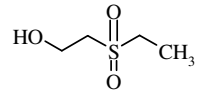
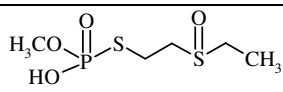
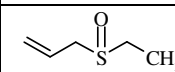
Technical formulation (purity 53.6 %)

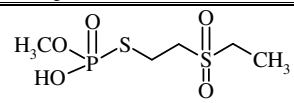
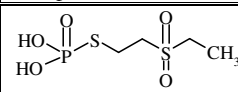
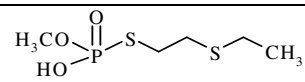
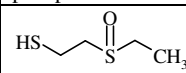
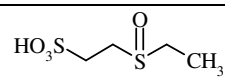
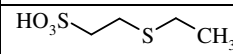
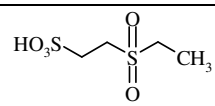
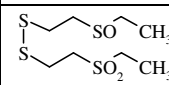
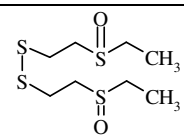
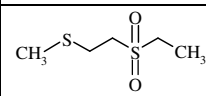
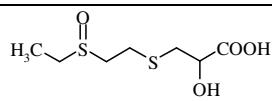
Flammability Auto ignition temperature 285°C (Eberz, 1999)

Flash point 80.5°C (Eberz, 1999)

METABOLISM AND ENVIRONMENTAL FATE

In this section the following compound codes were used.

Code	Compound	Code	Compound
ODM	 oxydemeton-methyl	M13	 2-hydroxy-3-[(2-ethylsulfonyl)ethylthio]propionic acid
M01	 demeton-S-methylsulphon	M14	 2-hydroxy-3-[(2-ethylsulfonyl-2-ethyl)-sulfinyl]-propionic acid
M02	 demeton-S-methyl	M15	 bis[2-(ethylsulfonyl)ethyl] disulfide
M03	 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane	M16	 1-(ethylsulfinyl)-2-(methylthio)ethane
M04	 1-(ethylsulfonyl)-2-(methylsulfinyl)ethane	M17	 2-ethylsulfinyl-ethanol
M05	 1-(ethylsulfonyl)-2-(methylsulfonyl)ethane	M18	 2-ethylsulfonylethanol
M06	 demethyl-oxydemeton-methyl	M19	 2-ethylsulfinylethylene

Code	Compound	Code	Compound
M07	 demethyl-demeton-S-methylsulphon	M20	 S-[2-(ethylsulfonyl)ethyl] phosphorothioate
M08	 demethyl-demeton-S-methyl	M21	 2-ethylsulfinylethanthiol
M09	 2-(ethylsulfinyl)ethanesulfonic acid	M22	 2-(ethylthio)ethanesulfonic acid
M10	 2-(ethylsulfonyl)ethanesulfonic acid	M23	 2-(ethylsulfinyl)ethyl [2-(ethylsulfonyl)ethyl disulfide
M11	 bis[2-(ethylsulfinyl)ethyl] disulfide	M24	 1-(ethylsulfonyl)-2-(methylthio)ethane
M12	 2-hydroxy-3-[(2-ethylsulfinyl)ethyl)thio]propionic acid		

Plant metabolism

Apples. The metabolism of [ethylene-1-^{14}C]oxydemeton-methyl in apples was investigated in the field (McEwen, 2002). Apple trees were sprayed twice at a nominal concentration of 1.4 g ai/l applied at a nominal rate of 350 g ai/ha approximately four (pink bud stage BBCH 57) and three months before harvest (flowers fading BBCH 67).

Samples of fruit and leaves, including controls, were taken for analysis

1. 2 h after 1st application; leaves only
2. 2 h after 2nd application; leaves only
3. 1st intermediate sample (approximately 60 days before harvest); fruit and leaves
4. 2nd intermediate sample (approximately 30 days before harvest); fruit and leaves
5. Harvest; fruit and leaves

All samples were surface-washed with acetonitrile. The peel, pulp and leaves were homogenized and extracted with acetonitrile followed by acetonitrile:water (1:1). The residues were extracted with cellulase and then with hydrochloric acid. Washes and extracts containing >10% of the radioactivity in the fruit or leaves were analysed by normal phase TLC. Components were characterized by co-chromatography with authentic reference standards.

Immediately after the first application 78.9% (255 mg/kg) of the radioactivity in the surface washes and extracts was accounted for by ODM and after the second 97.5% (128.1 mg/kg).

Analysis of leaf samples at the 1st intermediate and harvest stages detected ODM, whereas at the 2nd intermediate sampling it was not detected. However all samples contained polar material (M07 and P3) (4% of the radioactivity at 1st intermediate, 9% by 2nd) but at harvest this had decreased to 2%. The main components of the residue at the 1st intermediate were ODM 5.1%, 1.3 mg/kg, and M01 3.7%, 1.0 mg/kg, but by the 2nd intermediate ODM and M01 could not be detected, and the major components were M07 (4.7% leaf radioactivity, 0.758 mg/kg) and P4 (4.8% leaf radioactivity, 1.3 mg/kg). By harvest these had decreased to 1.5% (0.3 mg/kg) and 0.5% of leaf radioactivity (0.105 mg/kg).

Analysis of pulp and peel showed ODM in the 1st and 2nd intermediate samples but not at harvest. However the 1st and 2nd intermediate samples contained M07, P2 and P3. In the 1st intermediate sample radioactivity in the pulp accounted for 17% of the radioactivity in the fruit (0.2 mg/kg) and by the 2nd 10% (0.05 mg/kg), but at harvest no components were detected.

In the peel extracts from the 1st intermediate sample, polar metabolites accounted for 3% of the radioactivity in the fruit (0.023 mg/kg) and this increased to 4% (0.026 mg/kg) at the 2nd intermediate. No components were detected at harvest. The major components detected at the 1st intermediate sampling in the pulp and peel together were ODM (26.6% of the radioactivity in the fruit, 0.245 mg/kg) and M01 (2.8% of the radioactivity in the fruit, 0.026 mg/kg). However by the 2nd intermediate sample ODM (1.7% radioactivity in the fruit, 0.012 mg/kg) and M01 (0.2%, 0.001 mg/kg) were detected only in the peel. The main component in the pulp was P4 (14.1%, 0.072 mg/kg). No compounds were identified at harvest in either the pulp or peel owing to the low levels of radioactivity in the extracted samples (<0.001 mg/kg). The results are shown in Tables 1-5.

Table 1. Concentration of total radioactive residue (mg/kg as ODM) in apples and leaves after the application of [^{14}C]oxydemeton-methyl.

Sample	Time				
	Application 1	Application 2	1st intermediate	2nd intermediate	Harvest
Fruit	-	-	0.918	0.574	0.134
Leaves	324.8	144.2	27.19	18.89	20.41

Table 2. Radioactivity in apples after the application of [^{14}C]oxydemeton-methyl.

Sample		^{14}C, % of total in fruit & (mg/kg as ODM)		
		1st intermediate	2nd intermediate	Harvest
Surface wash		4.25 (0.042)	5.57 (0.028)	ND
Peel extracts	Acetonitrile	7.57 (0.065)	8.07 (0.055)	8.59 (0.009)
	Cellulase	1.04 (0.009)	1.33 (0.007)	2.00 (0.003)
	1M HCl	0.44 (0.004)	0.58 (0.002)	0.95 (0.001)
	6M HCl	0.26 (0.002)	0.58 (0.002)	0.51 (0.001)
	Total peel extract	9.31 (0.080)	10.56 (0.065)	12.05 (0.013)
	Unextractable residue	5.26 (0.041)	7.29 (0.046)	6.15 (0.009)
Pulp extracts	Acetonitrile	47.29 (0.441)	39.99 (0.205)	22.62 (0.024)
	Cellulase	6.45 (0.057)	12.06 (0.064)	7.27 (0.009)
	1M HCl	3.73 (0.034)	7.67 (0.052)	2.90 (0.004)
	6M HCl	1.88 (0.017)	2.37 (0.015)	0.97 (0.001)
	Total pulp extract	59.35 (0.549)	62.09 (0.335)	33.77 (0.038)
	Unextractable residue	21.84 (0.201)	11.03 (0.081)	34.94 (0.057)

Sample	^{14}C, % of total in fruit & (mg/kg as ODM)		
	1st intermediate	2nd intermediate	Harvest
Juice	NS	3.47 (0.020)	13.09 (0.018)
Total fruit	(0.918)	(0.574)	(0.134)

Table 3. Radioactivity in leaves of apple trees after the application of [^{14}C]oxydemeton-methyl.

Sample		^{14}C, % of total in leaves & (mg/kg as ODM)		
		1st intermediate	2nd intermediate	Harvest
Surface wash		13.42 (3.44)	11.03 (2.02)	1.92 (0.39)
Extracts	Acetonitrile	3.54 (0.80)	10.37 (1.60)	4.26 (0.77)
	Cellulase	11.43 (2.40)	27.05 (5.72)	ND
	1M HCl	1.89 (0.38)	8.15 (1.56)	ND
	6M HCl	0.32 (0.06)	2.10 (0.41)	ND
	Total extract	13.77 (3.65)	47.68 (9.28)	4.26 (0.77)
Unextractable residue		72.82 (20.10)	41.29 (7.60)	93.8 (19.25)
Total leaf		(27.19)	(18.89)	(20.41)

Table 4. Radioactive components in wash and acetonitrile extracts of leaves after the application of [^{14}C]oxydemeton-methyl. [1]

Sample		^{14}C, % of total in leaves & (mg/kg as ODM)						
		M07	P2	P3	P4	ODM	M01	Others
1st Intermediate	Wash	0.9 (0.241)	ND (ND)	2.3 (0.584)	ND (ND)	4.1 (1.06)	3.0 (0.78)	3.0 (0.775)
	Extract	0.3 (0.071)	ND (ND)	0.8 (0.187)	ND (ND)	1.0 (0.219)	0.7 (0.167)	0.7 (0.162)
2nd Intermediate	Wash	1.3 (0.238)	ND (ND)	2.2 (0.411)	3.8 (0.700)	ND (ND)	ND (ND)	3.7 (0.760)
	Extract	3.4 (0.520)	ND (ND)	2.0 (0.310)	1.0 (0.601)	ND (ND)	ND (ND)	4.0 (0.612)
Harvest	Wash	0.2 (0.045)	ND (ND)	0.4 (0.085)	0.5 (0.105)	0.1 (0.030)	ND (ND)	0.6 (0.127)
	Extract	1.3 (0.238)	1.8 (0.323)	0.2 (0.043)	ND (ND)	ND (ND)	ND (ND)	0.9 (0.164)

Table 5. Proportion of radioactive components in acetonitrile extracts of apples after the application of [^{14}C]oxydemeton-methyl. [1]

Sample		^{14}C, % of total in fruit & (mg/kg as ODM)						
		M07	P2	P3	P4	ODM	M01	Others
1st Intermediate	Pulp Extract	2.8 (0.026)	9.7 (0.091)	4.3 (0.040)	ND (ND)	23.0 (0.214)	2.4 (0.022)	5.2 (0.048)
	Peel Extract	0.5 (0.005)	1.4 (0.012)	0.7 (0.006)	ND (ND)	3.6 (0.031)	0.4 (0.004)	1.0 (0.008)

Sample		^{14}C, % of total in fruit & (mg/kg as ODM)						
		M07	P2	P3	P4	ODM	M01	Others
2nd Intermediate	Pulp Extracts	7.9 (0.041)	ND (ND)	2.3 (0.012)	14.1 (0.072)	ND (ND)	ND (ND)	15.7 (0.081)
	Peel Extracts	1.2 (0.008)	2.6 (0.018)	ND (ND)	ND (ND)	1.7 (0.012)	0.2 (0.001)	2.4 (0.016)
Harvest	Pulp Extracts	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)
	Peel Extracts	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)

Environmental fate in soil

Aerobic degradation

Three soils, sandy loam (Laacherhof AXXa), silt loam (Laacherhof AIII) and silt (Hoefchen am Hohenseh), were stored for a maximum of 11 days in the dark at 20°C (Babczinski, 2001a). The soil characteristics are shown in Table 6.

The soil moisture corresponded to 40% of maximum water holding capacity in the three soils. [Ethylene-1-^{14}C]oxydemeton-methyl was applied at a nominal rate of 0.67 mg/kg dry soil, equivalent to the proposed single maximum annual rate of 250 g ai/ha for 2.5 cm soil depth. The experiment was conducted in accordance with EC/SETAC/OECD guidelines. Erlenmeyer flasks, attached with traps for collection of CO_2 and volatile organics, were incubated and sampled after approximately 2 h and 1, 2, 3, 4, 7 and 11 days and in a supplementary experiment after 0.1, 1, 2, 3, 4, 5, 6 and 24 h without traps for volatiles.

The samples were extracted four times with methanol, followed by a mixture of methanol and water (1:1). Analysis was by reverse-phase TLC, and components were characterized by co-chromatography with authentic reference standards and LC/MS and LC/MS/MS. During the study the total recoveries of radioactivity in individual test vessels ranged from 90.8% to 100%. In the three soils the DT_{50} and DT_{90} ranged from 0.17 to 0.22 and 0.58 to 0.74 days respectively. Furthermore the results indicated that the main metabolites were continuously degraded, that no product accumulated toward the end of the study, and unextracted residues were participating in the natural carbon cycle of soil.

Analysis of soil extracts showed two major and one less important degradation product (about 10% of the applied radioactivity (AR)) throughout the study. M09 reached a maximum on day 1 in all soils and was highest at 26.7% of the AR in Laacherhof AIII. It decreased thereafter, in the more active soils to <LOQ by day 11. M10, an oxidation product of M09, reached its maximum on day 3 in all soils and was highest at 16.8% of the AR in Laacherhof AXXa. It decreased towards the end of the study in all three soils, in the most active to almost <LOQ by day 11.

M05 reached its peak on day 4 (9.5% of the AR) in all soils and decreased thereafter. All the other detected individual ^{14}C-zones corresponded to ≤2.1% of the AR throughout the study. The total radioactivity at the TLC origin was ≤4.9% of the AR in all cases.

Bound residues in the three soils reached a maximum level at day 11 of about 50% of the AR, and all soils showed a high mineralization capacity yielding up to >30% of $^{14}CO_2$ on day 11.

The half-life in soil is expected to be about <1 day, regardless of the type of soil. The major products were further degraded and therefore would not accumulate in the soil. The results are shown in Tables 7-11.

Table 6. Characteristics of soil used for degradation experiment.

Designation	Type	Textural analysis (USDA) (%)			Org. C (%)	Org. (%) matter	Cation ex. cap. (meq/100g soil)	pH Water
		2000-50 µm	50-2 µm	<2 µm				
Laacherhof AXXa	Sandy loam	72.4	22.6	5.0	1.02	1.75	8	7.2
Laacherhof AIII	Silt loam	36.9	51.1	12.0	0.83	1.43	8	7.4
Hoefchen am Hohenseh 4a	Silt	8.5	81.3	10.2	1.55 - 2.11	2.67 - 3.63	15	7.3 - 7.6

Table 7. Distribution of radioactivity after the application of [^{14}C]oxydemeton-methyl to three soils, % of applied radioactivity.

Soil	Distribution		Interval						
			0.2 hr	1 day	2 days	3 days	4 days	7 days	11 days
Laacherhof AXXa	Volatiles	Soda lime	-	3.2	5.7	7.8	11.0	19.2	24.6
		PU Foam	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.4
		Total	-	3.2	5.7	7.8	11.0	19.2	25.1
	Extractable	Org. extract	49.7	23.7	22.7	21.6	20.2	16.0	9.9
		Water extract	18.4	20.0	17.9	16.6	14.7	10.2	5.0
		Total	68.1	43.7	40.6	38.1	34.9	26.2	14.9
	Unextr.	Soil	30.5	46.4	46.0	47.0	46.7	45.5	50.5
		Filter	1.4	1.1	0.8	0.9	0.8	0.8	0.4
		Total	31.9	47.5	46.9	47.9	47.6	46.3	50.9
	Total		100.0	94.4	93.2	93.9	93.5	91.7	90.9
Laacherhof AIII	Volatiles	Soda lime	-	1.1	2.6	4.8	6.6	12.8	19.8
		PU Foam	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.4
		Total	-	1.1	2.6	4.8	6.6	12.8	20.2
	Extractable	Org. extract	56.2	18.9	16.6	15.0	13.6	10.6	7.8
		Water extract	24.9	26.6	29.3	26.1	24.8	18.8	11.9
		Total	81.1	48.5	45.8	41.0	38.4	29.4	19.7
	Unextr.	Soil	17.4	45.3	46.9	47.9	48.4	48.0	51.3
		Filter	1.5	1.0	0.9	0.8	0.7	0.6	0.4
		Total	18.9	46.3	47.7	48.6	49.0	48.6	51.7
	Total		100.0	95.9	96.2	94.4	94.0	90.8	91.7
Hoefchen am Hohenseh 4a	Volatiles	Soda lime	-	3.8	7.4	11.3	15.0	27.1	31.6
		PU Foam	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.5
		Total	-	3.8	7.4	11.3	15.0	27.1	32.1
	Extractable	Org. extract	59.7	25.7	22.9	20.8	17.8	11.7	7.4

Soil	Distribution		Interval						
			0.2 hr	1 day	2 days	3 days	4 days	7 days	11 days
		Water extract	16.7	16.3	14.3	12.2	10.2	6.3	3.1
		Total	76.5	42.0	37.2	33.0	28.1	18.0	10.5
	Unextr.	Soil	22.3	50.3	50.0	52.2	51.0	50.6	51.2
		Filter	1.2	1.2	0.8	0.8	0.6	0.4	0.2
		Total	23.5	51.6	50.8	53.0	51.5	51.0	51.5
	Total		100.0	97.4	95.4	97.3	94.6	96.0	94.0

Table 8. Distribution of radioactivity (% of the AR) after the application of [^{14}C]oxydemeton-methyl to Laacherhof AIII soil in the supplementary study.

Distribution		Interval (h)							
		0	1	2	3	4	5	6	24
Extractable	Org. extract	67.6	76.2	72.2	73.7	73.8	69.1	64.5	45.5
	Water extract	26.0	19.1	18.2	18.7	19.4	18.0	19.7	22.0
	Total	93.6	95.3	90.4	92.4	93.2	87.2	84.3	67.5

Table 9. Distribution of oxydemeton-methyl and degradation products after the application of [^{14}C]oxydemeton-methyl to three soils.

Time (days)	% applied radioactivity										
	Total extd.	Origin	ODM	M05	ROI 2	M10	M09	ROI 5	ROI 6	ROI 7	Diffuse
Laacherhof AXXa											
0	68.1	2.5	39.1	3.1	1.7	4.9	12.4	0.2	0.1	<0.1	4.1
1	43.7	1.7	1.7	8.4	<0.1	13.9	15.0	<0.1	<0.1	1.0	1.9
2	40.6	2.5	0.8	8.6	0.2	15.9	9.6	<0.1	<0.1	1.4	1.7
3	38.1	1.5	0.7	9.3	<0.1	16.8	6.3	<0.1	<0.1	1.8	1.7
4	34.9	1.6	0.5	9.5	<0.1	16.7	4.0	<0.1	<0.1	2.0	0.7
7	26.2	2.1	0.5	8.4	<0.1	11.1	<0.1	<0.1	<0.1	2.1	2.1
11	14.9	1.6	0.4	6.7	<0.1	3.7	<0.1	<0.1	<0.1	1.5	1.1
Laacherhof AIII											
0	81.1	1.6	70.9	<0.1	0.9	0.8	4.6	<0.1	<0.1	<0.1	2.3
1	48.5	3.7	2.3	2.8	0.2	7.9	26.7	<0.1	<0.1	<0.1	4.9
2	45.8	4.4	1.3	3.0	1.0	8.4	23.7	<0.1	<0.1	0.3	3.7
3	41.0	1.9	0.7	3.4	<0.1	8.9	22.8	<0.1	<0.1	0.4	2.8
4	38.4	1.9	0.4	3.5	<0.1	8.3	22.4	<0.1	<0.1	0.5	1.4
7	29.4	3.2	0.3	2.5	<0.1	2.2	17.2	<0.1	<0.1	0.6	3.3
11	19.7	2.7	0.3	1.5	<0.1	3.0	9.4	<0.1	<0.1	0.4	2.4
Hoefchen am Hohenseh 4a											
0	76.5	1.6	60.4	0.5	1.3	1.9	7.9	<0.1	<0.1	<0.1	2.7
1	42.0	1.7	1.1	7.0	<0.1	13.0	15.7	<0.1	<0.1	0.9	2.6
2	37.2	1.8	0.6	7.3	0.3	15.2	8.6	<0.1	<0.1	1.4	1.9

Time (days)	% applied radioactivity										
	Total extd.	Origin	ODM	M05	ROI 2	M10	M09	ROI 5	ROI 6	ROI 7	Diffuse
3	33.0	1.2	0.4	7.7	<0.1	15.2	5.2	<0.1	<0.1	1.4	1.8
4	28.1	1.0	0.3	8.2	<0.1	13.6	2.7	<0.1	<0.1	1.4	1.0
7	18.0	1.8	0.3	7.2	<0.1	5.4	<0.1	<0.1	<0.1	1.3	2.0
11	10.5	0.6	0.2	7.4	<0.1	0.8	<0.1	<0.1	<0.1	1.0	0.4

Table 10. Distribution of oxydemeton-methyl and degradation products after the application of [^{14}C]oxydemeton-methyl to Laacherhof AIII soil in supplementary study.

Time (h)	% applied radioactivity									
	Total extd.	Origin	ODM	M05	ROI 2	M10	M09	ROI 6	ROI 7	Diffuse
0	93.6	0.6	92.7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.4
1	95.3	0.8	92.1	<0.1	<0.1	<0.1	0.4	<0.1	<0.1	2.0
2	90.4	0.5	88.8	<0.1	<0.1	<0.1	0.2	<0.1	<0.1	0.9
3	92.4	0.5	87.0	<0.1	0.2	<0.1	0.4	<0.1	<0.1	4.2
4	93.2	1.0	87.7	<0.1	0.3	<0.1	1.2	<0.1	0.4	2.6
5	87.2	1.1	78.5	<0.1	0.6	1.8	0.9	<0.1	0.4	3.8
6	84.3	1.5	74.3	<0.1	0.8	<0.1	2.9	<0.1	0.7	4.0
24	67.5	2.2	40.0	0.6	1.0	<0.1	16.6	0.2	1.1	6.0

Table 11. Degradation parameters of oxydemeton-methyl in three soils.

Parameter	Laacherhof AXXa	Laacherhof AIII	Hoefchen am Hohenseh 4a
Main study			
K (1/day)	3.10	3.39	3.99
DT50 (days)	0.22	0.20	0.17
DT_{75} (days)	0.45	0.41	0.35
DT_{90} (days)	0.74	0.68	0.58
R^2	0.999	0.999	1.000
Supplementary study			
K (1/h)	-	0.036	-
DT_{50} (h)	-	19.4	-
DT_{75} (h)	-	38.8	-
DT_{90} (h)	-	64.4	-
R^2	-	0.980	-

Environmental fate in water/sediment systems

Hydrolysis

The hydrolysis of oxydemeton-methyl was studied in sterile 0.01M buffer solutions adjusted to pH 4, 7 and 9, stored for a maximum of 31 days in the dark at two temperatures (Babczinski, 2001b). The

experiment was carried out in compliance with GLP standards and EPA/SETAC/OECD/EC Guidelines.

The test solution was prepared with [ethylene-1-^{14}C]oxydemeton-methyl at a concentration of about 5 mg/l. A pre-test solution was incubated for 7 days under sterile conditions in the dark at 50°C with sampling at 0, 2.5 and 6 h and 1, 2 and 7 days, and in the main test the solutions at pH 4 and pH 7 were incubated for 31 days under sterile conditions in the dark at 25°C with sampling intervals of 0, 5, 11, 14, 29, 26 and 31 days deduced from the results of the pre-test. At pH 9, samples were taken after 0, 0.25, 1, 1.25, 2, 2.25, 3 and 6 days.

In the pre-test at 50°C and in the main-test at 25°C, ODM was unstable at pH 4, 7 and 9 and considerable degradation occurred. The compound was hydrolysed to M06 (maximum 20.4% of the AR) and, at pH 7 and 9, M21 (maximum 9.0% of the AR) by cleaving the P-S bond. The sulfonic acid M10 was tentatively identified as an oxidized P-S cleavage product at low percentages (maximum 2.2% of the AR).

M21 underwent dimerization to M11 (maximum 74.2% AR). This reaction is unlikely to happen in the environment since exposure would be expected to be at a significantly lower level. Realistically, this degradation product should by calculation be added to M21 making this compound the main hydrolytic degradation product at pH ≥7. Finally, desdimethyl-ODM was identified at low concentrations at 50°C and pH 9 but not quantified.

At 50°C, first-order half-lives for ODM were estimated to be 4.9, 3.5 and 0.2 days at pH 4, 7 and 9 at 25°C, 91, 42 and 2.5 days. Half-lives at 20°C were calculated from Arrhenius plots (1/T versus In k) as 174, 73 and 4.5 days. These results indicate that hydrolytic processes contribute to the degradation of ODM in the environment. The results are shown in Tables 12-15.

Table 12. Half-lives for the hydrolysis of oxydemeton-methyl in sterile aqueous solutions at 50°C (pre-test).

Solution	DT_{50} (days)
pH 4 (0.01M acetate buffer)	4.9
pH 7 (0.01M TRIS buffer)	3.5
pH 9 (0.01M borate buffer)	0.2

Table 13. Half-lives, DT_{75}s and DT_{90}s for the hydrolysis of oxydemeton-methyl in sterile aqueous solutions at 25°C.

Solution	DT_{50} (days)	DT_{75} (days)	DT_{90} (days)
pH 4 (0.01M acetate buffer)	91	182	303
pH 7 (0.01M TRIS buffer)	42	85	141
pH 9 (0.01M borate buffer)	2.5	4.9	8.2

Table 14. Half-lives and DT_{90}s for the hydrolysis of oxydemeton-methyl in aqueous solution at 20°C (calculated from experimental data at 25°C).

Solution	DT_{50} (days)	DT_{90} (days)
pH 4 (0.01M acetate buffer)	174	577
pH 7 (0.01M TRIS buffer)	73	243
pH 9 (0.01M borate buffer)	4.5	15

Table 15. Distribution of oxydemeton-methyl and degradation products (% of the AR) during the hydrolysis of oxydemeton-methyl in sterile aqueous buffer solutions at 25°C.

Solution	Interval (days)	Total radioactivity (% of applied)	ODM	M06	M11	M10	M21	Others
0.5 m g/l buffer pH 4	0	100.0	100.0	-	-	-	-	-
	5	100.3	96.3	3.4	-	-	-	0.6
	11	95.2	87.7	6.7	-	-	-	0.7
	14	96.7	87.3	8.4	-	-	-	0.9
	20	98.9	85.7	11.9	-	-	-	1.2
	26	98.3	81.9	15.1	-	-	-	1.1
	31	97.5	78.5	16.7	-	-	-	2.2
0.5 m g/l buffer pH 7	0	100.0	99.7	0.3	-	-	-	-
	5	104.0	96.7	4.6	2.4	0.3	-	0.1
	11	100.8	84.7	6.0	7.5	1.0	0.1	1.5
	14	99.9	80.4	6.2	9.2	1.3	0.3	2.5
	20	100.7	70.8	9.1	10.7	1.4	0.2	8.6
	26	102.5	68.8	20.4	11.2	1.2	-	0.9
	31	101.1	60.3	11.9	16.5	2.2	0.4	9.6
0.5 m g/l buffer pH 9	0	100.0	98.7	-	0.7	-	0.5	-
	0.25	101.5	95.6	0.3	2.1	0.2	2.8	0.6
	1	101.9	79.2	0.6	13.5	0.4	7.4	0.8
	1.25	101.6	72.8	1.1	16.9	0.5	8.9	1.5
	2	101.7	57.7	1.4	31.0	0.4	9.0	2.2
	2.25	104.2	54.1	1.6	37.3	0.4	6.8	3.9
	3	102.3	42.6	1.7	47.8	0.3	4.8	5.1
	6	101.8	16.8	2.4	74.2	0.2	0.5	7.6

Photolysis

The quantum yield of direct photodegradation of oxydemeton-methyl in water was determined according to the ECETOC method in polychromatic light (Hellpointner *et al.*, 1992). From the UV absorption data and the kinetic result of photodegradation experiments in a merry-go round irradiation apparatus the quantum yield was calculated to be 0.00078. The quantum yield and UV absorption data in aqueous solution were used to estimate the environmental half-life of ODM during photodegradation in water by two different simulation models. The results are shown in Tables 16 and 17.

Table 16. Calculated half-life of oxydemeton-methyl in water (GC-Solar program).

Season	Environmental half-life (days) at degrees latitude			
	30	40	50	60
Spring	112	117	126	143
Summer	112	117	126	143
Autumn	188	260	428	909
Winter	274	476	1110	4010

Table 17. Calculated half-life of oxydemeton-methyl in water (Frank-Klöpffer program).

Month	Photolysis constant	Environmental half-life (days)		
		Minimum	Mean	Maximum
March	0.157 10E-7	270	510	2100
April	0.291 10E-7	150	280	1100
May	0.393 10E-7	130	200	820
June	0.451 10E-7	120	180	710
July	0.401 10E-7	130	200	670
August	0.378 10E-7	140	210	710
September	0.208 10E-7	230	390	1400
October	0.101 10E-7	420	790	3600

RESIDUE ANALYSIS

Analytical methods

Many of the methods developed for the determination of residues of oxydemeton-methyl in various samples were reviewed at the 1998 Meeting under the Periodic Review Programme. The present Meeting received supplementary information on more recent methods. These still depended on oxidising ODM to demeton-S-methylsulphon as analyte.

Method 00585 (Blass *et al.*, 2001a)
This method is used to determine residues of oxydemeton-methyl, demeton-S-methyl and demeton-S-methylsulphon after oxidation to demeton-S-methylsulphon in plant materials. Oxydemeton-methyl and the other two compounds are extracted and oxidized in one step with aqueous acidic potassium permanganate solution using a microwave. Clean-up on an Extrelut column (preceded by hexane partitioning for brassica) is followed by partition against dichloromethane. The dichloromethane extract is evaporated to dryness and re-dissolved in acetone before quantification by GC-FPD. Recoveries from plant samples fortified at 0.005 to 1.0 mg/kg ranged from 70 to 110% with an RSD of ≤20%. The limit of quantification was 0.005 mg/kg for apples, potatoes and grapes and 0.01 mg/kg for Brussels sprouts.

Method 00585/M001 (Blass *et al.,* 2001)
This modification permits analysis of low-water content crops such as wheat grain, forage and straw by the addition of water and Celite before oxidation. Recoveries were within the 70 to 110% range with an RSD of ≤20% from samples of grain and straw fortified at 0.02-2.0 and 0.05-5.0 mg/kg respectively and the limits of quantification were 0.02 and 0.05 mg/kg.

Methods 00255/E001 and 00255/E002 (Seym, 1994, 1995)
Method 00255 (Ohs, 1992) was described in the 1998 Residue Evaluation. These supplements consist of validation data for the determination of residues in cauliflower, corn and sunflower (E001) and cauliflower (E002). They evaluate the sensitivity of the detector used in the quantification and confirm the limit of quantification. In E001 mean recoveries ranged from 70 to 108% after fortification with 0.01 and 0.04 mg/kg of oxydemeton-methyl and the overall mean was 89% with an RSD of 9%. In E002 recoveriesranged from 89 to 97% from cauliflower after fortification at 0.01 and 0.1 mg/kg with individual values of 85-97%. The mean recovery was 93% with an RSD of 6%. The limit of quantification for all samples was 0.01 mg/kg.

Method 00255/E004 and 00255/E005 (Seym, 1996)

These consist of a minor variation to the basic method for the determination of residues in head cabbage (E004), and Savoy and red cabbage (E005) in that the sample size is reduced to 50 g and the extraction solvent volume to 150 ml. The rest of method is unchanged. Mean recoveries from head cabbage after fortification at 0.01-0.1 mg/kg were 80-90% with an RSD of 7.0%, and from Savoy and red cabbage 73-98% and 89-92% respectively at the same fortification levels with individual recoveries ranging from 69 to 109%. The limit of quantification was 0.01 mg/kg.

Method 00255/E008 and 00255/E009 (Schoning, 1998)
In this variation to the basic method for the determination of residues in apples, pears, grapes and Brussels sprouts (E008) and wheat and barley grain (E009) the sample size is again reduced to 50 g and the extraction solvent to 150 ml. For dry samples (E009) water is added during extraction, and the extract is filtered. The rest of method is unchanged. In E008 mean recoveries at each fortification level from the four crops were 96-101% with an RSD of 0.8 to 8.1% at fortification levels of 0.01-0.1 mg/kg, and in E009 83-106% from cereal grain at levels of 0.01-0.1 mg/kg with an RSD of 9.3 to 12.9%. The limit of quantification for both modifications was 0.01 mg/kg.

Method 00255/E012 (Schoning, 2001)
In this minor variation for the determination of residues in rape foliage and seed 50 g samples are extracted with 150 ml of acetone and filtered. After filtration the extract is filtered through Celite which is washed with acetone/water (2:1) and the filtered extracts partitioned three times with dichloromethane. The remainder of the method is unchanged. Mean recoveries from foliage and seed were 80-83% and 74-79% respectively at fortification levels of 0.01-0.1 mg/kg with individual recoveries ranging from 70 to 88%. The limit of quantification was 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

The storage stability of oxydemeton-methyl in cabbage, maize, lettuce and papaya was briefly reported to the 1998 JMPR, which concluded that data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton-methyl were highly desirable as the information available was unrepresentative of the various crop groups, did not cover extended storage intervals, and suggested variable storage stability. The manufacturer reported a new study to the present Meeting.

The study was conducted to determine the storage stability of oxydemeton-methyl in spiked commercial samples of apple, dried peas, potato and oilseed rape (meal and oil) treated with formulated oxydemeton-methyl and stored at -20°C (Smith, 2002). Apple and potato samples were finely chopped in an industrial food processor (the preparation method for dried peas was not reported). Twenty g of samples of each crop were spiked with oxydemeton-methyl at nominal concentrations of 0.1, 1.0 and 10 mg/kg and stored at -20°C. Two samples from each treatment were analysed after 0, 3, 6, 12 and 24 months' storage. The analytical method (Thornton *et al.,* 1977) was validated for each crop at day 0, but after three months a modified procedure (Hill *et al.,* 1994) was used to improve the oxidation/extraction phase. The results are shown in Table 18.

The results do not show any substantial loss of residue over 24 months' storage in apple, potato or oil, but about half the residue was lost from dried peas and meal in 6 and 3 months respectively. Control samples contained ODM (apple <0.005-0.022 mg/kg, dried peas <0.005-0.016 mg/kg, potatoes <0.01-0.016 mg/kg, rape meal 0.013-0.03 and oil <0.005-0.011 mg/kg). The Meeting concluded that the storage stability data were inadequate and maintained the former requirement.

Table 18. Analyses of stored spiked samples.

Crop	Spiking level (mg/kg)	Mean oxydemeton-methyl content, mg/kg[1], and (% of initial value)			
		3 months	6 months	12 months	24 months
Apple	0.101 1.01 10.1	0.094 (92.5) 1.045 (94.0) 9.325 (92.5)	0.095 (94.5) 0.836 (82.5) 8.81 (87.5)	0.113 (111.5) 0.868 (86.0) 7.96 (79.0)	0.097 (96.0) 0.971 (95.5) 8.365 (82.5)
Dries peas	0.101 1.01 10.1	0.114 (113.5) 0.811 (80.0) 7.405 (73.5)	0.0648 (64.0) 0.620 (61.5) 5.535 (54.5)	0.051 (51.0) 0.438 (43.0) 5.80 (57.0)	0.067 (51.5) 0.473 (45.5) 4.63 (46.0)
Potato	0.101 1.01 10.1	0.029 (29.0) 0.625 (61.5) 9.81 (95.5)	0.083 (82.0) 0.786 (77.5) 8.455 (84.0)	0.071 (70.0) 0.744 (74.0) 7.000 (69.5)	0.0775 (76.5) 0.728 (72.5) 7.865 (77.5)
Oilseed rape (meal)	0.101 1.01 10.1	0.047 (47.0) 0.51 (50.5) 6.715 (66.5)	0.047 (46.5) 0.655 (64.5) 5.565 (55.0)	0.037 (36.5) 0.608 (60.0) 5.96 (59.0)	0.0774 (47.0) 0.529 (49.5) 5.325 (52.5)
Oilseed rape (oil)	0.101 1.01 10.1	0.010 (99.0) 0.913 (90.5) 8.815 (87.5)	0.081 (80.0) 0.804 (79.5) 7.815 (77.5)	0.080 (79.5) 0.918 (91.0) 8.355 (83.0)	0.089 (88.5) 0.861 (81.0) 8.525 (84.5)

[1] Corrected for apparent residue in control samples

USE PATTERN

Product labels from Europe were submitted to the Meeting together with translations into English.

Table 19. Registered uses of oxydemeton-methyl.

Crop	Country	Formulation	Dose (formulation)	Dose (ai) (kg/hl or kg/ha)	PHI (days)	No. of applns.
almond	Greece	500 g/l SL	0.10%	0.05	90	2
almond	Spain	250 g/l EC	0.10%	0.025	until petals fall	
apple	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
apple	Greece	500 g/l SL	0.10%	0.05	60	2
apple	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	90	
barley	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1
beans (horse)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	28	
beet	Spain	250 g/l EC	0.10%	0.025	30	
beet (fodder)	Austria	48.5 g/l EC	2 l/ha	0.097	35	4
beet (fodder)	Germany	265.29 g/l EC	0.6-0.8 l/ha	0.16-0.21	28	3
beet (sugar)	Germany	265.29 g/l EC	0.6-0.8 l/ha	0.16-0.21	28	3
beet (sugar)	Austria	48.5 g/l EC	2 l/ha	0.097	35	4
beet (sugar)	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	30	
broccoli	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cabbage	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cabbage (Chinese)	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cabbage (fodder)	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1

Crop	Country	Formulation	Dose (formulation)	Dose (ai) (kg/hl or kg/ha)	PHI (days)	No. of applns.
cabbage (green)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cabbage (red)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cabbage (Savoy)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cabbage (white)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cauliflower	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cauliflower	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
citrus fruit	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
citrus fruit	Greece	500 g/l SL	0.10%	0.05	90	2
citrus fruit	Spain	250 g/l EC	0.10%	0.025	90	
fruit trees	Finland	250 g/l EC	0.01-0.02%	0.0025-0.005	60 fruit size <20 mm	
grape vine	Germany	265.29 g/l EC	0.10%	0.027	up to inflorescences fully developed	1
kale	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
kohlrabi	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
kohlrabi	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
melon	Portugal	250 g/l EC	0.10%	0.025	before fruiting begins	
peach	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
pear	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
pear	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	90	
plum	Spain	250 g/l EC	0.10%	0.025	until petals fall	
plum	Germany	265.29 g/l EC	0.5 l/ha	0.13	immediately after flowering	1
plum	Austria	48.5 g/l EC	0.50%	0.024	up to 2 weeks after flowering	1
pome fruit	Germany	265.29 g/l EC	0.5 l/ha	0.13	immediately after flowering	1
pome fruit	Austria	48.5 g/l EC	0.50%	0.024	up to 2 weeks after flowering	1
potato	Greece	500 g/l SL	0.10%	0.05	28	3
potato	Spain	250 g/l EC	0.10%	0.025	30	
potato	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	28	
rye	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1
salad crops (except chicory and lambs lettuce)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
stone fruit	Spain	250 g/l EC	0.10%	0.025	until petals fall	

Crop	Country	Formulation	Dose (formulation)	Dose (ai) (kg/hl or kg/ha)	PHI (days)	No. of applns.
strawberries	Portugal	250 g/l EC	0.10%	0.025	before fruiting begins	
strawberries	Germany	265.29 g/l EC	2 l/ha 2000 l/ha (water)	0.53	after harvest	1
strawberries	Austria	48.5 g/l EC	0.50%	0.024	90	1
tomato	Portugal	250 g/l EC	0.10%	0.025	before fruiting begins	
triticale	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1
wheat	Portugal	250 g/l EC	0.1% 1 l/ha	0.025 0.25	until flowering	
wheat	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	30	
wheat	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised trials data on apples, pears, grapes, cabbages, Brussels sprouts, cauliflowers, field peas, potatoes, sugar beets, fodder beets, wheat, barley, rape and sunflower seeds were reported to the Meeting. Results are shown in Tables 20-33, where all residues are expressed as oxydemeton-methyl.

Table 20. Residues of oxydemeton-methyl and demeton-S-methylsulphon in apples after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)			
France (North) 1997	258EC	0.25-0.38	0.025	2 2nd fruit fall	29 44 59 74 89	0.08 0.04 0.02 0.02 0.01	RA-2157/97 0406-97
France (North) 1997	258EC	0.25-0.38	0.025	2 2nd fruit fall	30 45 60 75 90	0.08 0.05 0.04 0.02 0.02	RA-2157/97 0745-97
France (North) 1998	258EC	0.30-0.45	0.030	2 End of flowering	36 126	0.02 <u>≤0.01</u>	RA-2029/98 1361-98
France (South) 1997	258EC	0.25-0.38	0.025	2 20 mm fruit	45 60 74 91 109	0.01 <0.01 <0.01 <u>≤0.01</u> <0.01	RA-2156/97 0407-97
Italy 1997	250EC	0.25-0.44	0.025 0.029	2 20 mm fruit	30 45 60 75 90	0.10 0.06 0.03 0.02 <u>0.01</u>	RA-2109/97 0424-97

Country (Area) Year	Application				PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)			
Italy 1997	250EC	0.25-0.38	0.025	2 20 mm fruit	30 45 60 75 87	0.02 <0.01 <0.01 <0.01 <u>≤0.01</u>	RA-2109/97 0425-97
Spain[2] 1997	250EC	0.25-0.38	0.025	2 10 mm fruit	29 42 60 76 89	0.08 0.04 <0.01 <0.01 <u>≤0.01</u>	RA-2109/97 0571-97
Spain[2] 1997	250EC	0.25-0.38	0.025	2 10 mm fruit	29 42 60 76 89	0.11 0.03 0.02 <0.01 <0.01	RA-2109/97 0572-97
France (South) 1997	258EC	0.25-0.38	0.025	2 10 mm fruit	121	<0.01	RA-2156/97 0742-97
France (South) 1998	258EC	0.30-0.45	0.030	2 10 mm fruit	63 76 93 107 114	<0.01 <0.01 <u>≤0.01</u> <0.01 <0.01	RA-2030/98 1037-98
Spain 1998	250EC	0.25-0.38	0.025	3 10 mm fruit	69 90 129	<0.01 <u>≤0.01</u> <0.01	RA-2031/98 1042-98
Portugal 1998	250EC	0.25-0.38	0.025	2 20 mm fruit	49 126	0.01 <0.01	RA-2031/98 1364-98
Italy 1998	250EC	0.25-0.38	0.025	2 Flowers fading	58 150	<u>≤0.01</u> <0.01	RA-2031/98 1365-98
France (South) 1998	250EC	0.25-0.38	0.025	2 10 mm fruit	35 90	0.04 <u>≤0.01</u>	RA-2031/98 1366-98
Spain 1998	250EC	0.25-0.38	0.025	2 20 mm fruit	61 138	<u>≤0.01</u> <0.01	RA-2031/98 1367-98

[1] Expressed as oxydemeton-methyl.
[2] Trials conducted in same location.

Table 21. Residues of oxydemeton-methyl and demeton-S-methylsulphon in pears after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1998	258EC	0.30-0.45	0.030	2 2nd fruit fall	Fruit	50 91	<0.01 <u>≤0.01</u>	RA-2029/98 1036-98
France (South) 1998	258EC	0.30-0.45	0.030	2 10 mm fruit	Fruit	48 104	<0.01 <u>≤0.01</u>	RA-2030/98 1362-98

[1] Expressed as oxydemeton-methyl.

Table 22. Residues of oxydemeton-methyl and demeton-S-methylsulphon in grapes after foliar applications of oxydemeton-methyl.

Country Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1997 (Bacchus)	250 EC	0.2	0.025	1 9 or more leaves unfolded	Berry	113	<0.01	RA-2090/97 0516-97
					Bunch	113	≤0.01	
Germany 1997 (Mueller-Thurgau)	250 EC	0.2	0.025	1 9 or more leaves unfolded	Berry	115	<0.01	RA-2090/97 0578-97
					Bunch	115	≤0.01	
Germany 1997 (Portugieser)	250 EC	0.2	0.025	1 9 or more leaves unfolded	Berry	115	<0.01	RA-2090/97 0579-97
					Bunch	115	≤0.01	

[1] Expressed as oxydemeton-methyl.

Table 23. Residues of oxydemeton-methyl and demeton-S-methylsulphon in cabbages after foliar applications of oxydemeton-methyl.

Country (Area) Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (North) 1996 (Red cabbage)	258EC	0.15	0.054	1 (30% of the head size reached)	Head	0 7 10 14 21	0.03 <0.01 <0.01 <0.01 ≤0.01	RA-2150/96 0679-96
Germany 1996 (Savoy cabbage)	258EC	0.15	0.025	1 (30% of the head size reached)	Whole plant without roots	0	2.1	RA-2150/96 0676-96
					Head	21	≤0.01	
Germany 1996 (Savoy cabbage)	258EC	0.15	0.025	1 (30% of the head size reached)	Whole plant without roots	0	1.8	RA-2150/97 0677-97
					Head	21	≤0.01	
France (North) 1996 (White cabbage)	258EC	0.15	0.054	1 (70% of the head size reached)	Head	0 7 10 14 21	0.02 <0.01 <0.01 <0.01 ≤0.01	RA-2150/96 0678-96

[1] Expressed as oxydemeton-methyl.

Table 24. Residues of oxydemeton-methyl and demeton-S-methylsulphon in Brussels sprouts after foliar applications of oxydemeton-methyl.

Country (Area)	Application	Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.

	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1997	250EC	0.15	0.025	1 (First sprouts tightly closed)	Button	0 4 7 14 21 29	0.08 0.07 0.06 0.02 0.02 0.03	RA-2088/97 0419-97
Germany 1997	250EC	0.15	0.025	1 (First sprouts tightly closed)	Button	0 4 7 14 21 29	0.09 0.09 0.07 0.04 0.03 0.02	RA-2088/97 0420-97
Great Britain 1997	250EC	0.15	0.025	1 (50% of sprouts tightly closed)	Button	0 4 7 14 21 28	0.11 0.08 0.12 0.15 0.02 0.02	RA-2088/97 0574-97
Belgium 1997	250EC	0.15	0.025	1 (70% of sprouts tightly closed)	Button	0 4 7 14 21 28	0.01 <0.01 0.01 <0.01 <0.01 <0.01	RA-2088/97 0575-97
Belgium 1998	250EC	0.15	0.025	1 (Sprouts below terminal bud tightly closed)	Button	0 21	0.09 <0.01	RA-2118/98 1044-98
Germany 1998	250EC	0.15	0.025	1 (First sprouts tightly closed)	Button	0 21	0.18 <0.01	RA-2118/98 1513-98
France (North) 1998	250EC	0.15	0.025	1 (Sprouts below terminal bud tightly closed)	Button	0 21	0.02 <0.01	RA-2118/98 1514-98
Great Britain 1998	250EC	0.16	0.025	1 (First sprouts tightly closed)	Button	0 21	0.24 <0.01	RA-2118/98 1515-98
Germany 1998	250EC	0.15	0.025	1 (50% of sprouts tightly closed)	Button	0 21	0.07 <0.01	RA-2118/98 1516-98

[1] Expressed as oxydemeton-methyl.

Table 25. Residues of oxydemeton-methyl and demeton-S-methylsulphon in cauliflower after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1996	258EC	0.15	0.025	1 (Head beginning	Whole plant without roots	0	3.2	RA-2152/96 0669-96

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
				to form)	Head	7 14 21 28	<0.01 <0.01 ≤0.01 <0.01	
Germany 1996	258EC	0.15	0.025	1 (Head beginning to form)	Whole plant without roots	0	3	RA-2152/96 0670-96
					Head	7 14 21 28	<0.01 <0.01 ≤0.01 <0.01	
Great Britain 1996	258EC	0.15	0.025	1 (Bud development)	Whole plant without roots	0	1.1	RA-2152/96 0671-96
					Head	21	≤0.01	
France (North) 1996	258EC	0.38	0.063	1 (Head beginning to form)	Whole plant without roots	0	4.8	RA-2152/96 0672-96
					Head	21	≤0.01	
France (South) 1997	258EC	0.15	0.054	1 (30% of head diameter reached)	Whole plant without roots	0	0.84	RA-2153/96 0673-96
					Head	7 10 14 21	<0.01 <0.01 <0.01 ≤0.01	
France (South) 1996	258EC	0.15	0.054	1 (Head beginning to form)	Whole plant without roots	0	1.2	RA-2153/96 0675-96
					Head	7 10 14 21	<0.01 <0.01 <0.01 ≤0.01	
France (South) 1997	258EC	0.15	0.025	1 (Head beginning to form)	Whole plant without roots	0	1.4	RA-2160/97 0409-97
					Head	21	≤0.01	
France (South) 1997	258EC	0.16	0.025	1 (30% of the head diameter reached)	Whole plant without roots	0	1.4	RA-2160/97 0750-97
					Head	21	≤0.01	

[1] Expressed as oxydemeton-methyl.

Table 26. Residues of oxydemeton-methyl and demeton-S-methylsulphon in field peas after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South)	258EC	0.10-0.11	0.033-0.034	2 (Pods have	Whole plant without roots	0 14	2.4 0.06	RA-2161/97 0408-97

1997				reached typical size)	Seed	28	<0.01	
					Plant, dried	28	<0.01	
France (South) 1997	258EC	0.10	0.033	2 (10% of pods ripe)	Whole plant without roots	0 14	1.1 0.13	RA-2161/97 0751-97
					Seed	28	<0.01	
					Plant, dried	28	0.05	

[1] Expressed as oxydemeton-methyl.

Table 27. Residues of oxydemeton-methyl and demeton-S-methylsulphon in potatoes after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (North) 1996	258EC	0.15	0.054	2 (End of flowering)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2148/96 0665-96
France (North) 1996	258EC	0.15	0.054	2 (End of flowering)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2148/96 0773-96
France (North) 1996	258EC	0.15	0.054	2 (End of flowering)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2148/96 0774-96
France (North) 1996	258EC	0.15	0.054	2 (30% of total final tuber mass reached)	Tuber	0 7 14 21 28	<0.01 <0.01 <0.01 <0.01 ≤0.01	RA-2149/96 0666-96
France (North) 1996	258EC	0.15	0.050	2 (50% of total final tuber mass reached)	Tuber	0 7 14 21 28	<0.01 <0.01 <0.01 <0.01 ≤0.01	RA-2149/96 0667-96
France (North) 1996	258EC	0.15	0.054	2 (50% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2149/96 0668-96
France (North) 1996	258EC	0.15	0.054	2 (Tuber initiation)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2149/96 0781-96
France (North) 1996	258EC	0.20-0.25	0.050-0.063	3 (30% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2147/96 0664-96
Germany 1996	258EC	0.20-0.25	0.067-0.083	3 (70% of total final tuber mass reached)	Tuber	0 0 7 14 21 28	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RA-2147/96 0661-96

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1996	258EC	0.20-0.25	0.067-0.083	3 (Maximum of total final tuber mass reached)	Tuber	0 0 6 13 20 27	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RA-2147/96 0662-96
UK 1996	258EC	0.20-0.25	0.050-0.063	3 (60% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 <0.01	RA-2147/96 0663-96
Spain[2] 1997	250EC	0.30-0.32	0.050	3 (Berry ripening)	Tuber	-1 20 27	<0.01 <0.01 <u>≤0.01</u>	RA-2087/97 0426-97
Spain[2] 1997	250EC	0.30-0.32	0.050	3 (Berry ripening)	Tuber	-1 20 27	<0.01 <0.01 <0.01	RA-2087/97 0428-97
Italy 1997	250EC	0.30	0.050	3 (30% of total final tuber mass reached)	Tuber	0 21 28	0.01 <0.01 <u>≤0.01</u>	RA-2087/97 0695-97
Italy 1997	250EC	0.30	0.050	3 (30% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 <u>≤0.01</u>	RA-2087/97 0696-97
Italy 1998	250EC	0.30	0.050	3 (60% of total final tuber mass reached)	Tuber	21 28	<0.005 <u>≤0.005</u>	RA-2116/98 1046-98
Greece 1998	250EC	0.30	0.050	3 (Development of fruit)	Tuber	22 28	0.007 <u>≤0.005</u>	RA-2116/98 1507-98
Spain 1998	250EC	0.30-0.32	0.050	3 (10% of berries in first fructification have reached full size)	Tuber	21 28	<0.005 <u>≤0.005</u>	RA-2116/98 1509-98
France (South) 1998	250EC	0.30	0.050	3 (Development of fruit)	Tuber	21 28	<0.005 <u>≤0.005</u>	RA-2116/98 1510-98
Spain 1998	250EC	0.30	0.050	3 (40% of total final tuber mass reached)	Tuber	21 28	<0.005 <u>≤0.005</u>	RA-2116/98 1512-98

[1] Expressed as oxydemeton-methyl.
[2] Trials conducted in same location.

Table 28. Residues of oxydemeton-methyl and demeton-S-methylsulphon in sugar beet after foliar applications of oxydemeton-methyl.

Country Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Spain[2] 1997	250EC	0.38	0.13	3 (Beetroot has reached harvestable size)	Leaf	0 6 14 28 35	4.0 0.42 <0.04 <u>≤0.04</u> <0.04	RA-2091/97 0580-97

Country Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
					Beet	0 6 14 28 35	<0.01 <0.01 <0.01 ≤0.01 <0.01	
Spain[2] 1997	250EC	0.38	0.13	3 (Beetroot has reached harvestable size)	Leaf	0 6 14 28 35	3.9 0.27 <0.04 <0.04 <0.04	RA-2091/97 0581-97
					Beet	0 6 14 28 35	<0.01 <0.01 <0.01 <0.01 <0.01	
Italy 1997	250EC	0.13	0.042	3 (Fruit developing)	Leaf	0 7 14 28 35	2.4 0.11 <0.04 ≤0.04 <0.04	RA-2091/97 0421-97
					Beet	0 7 14 28 35	<0.01 <0.01 <0.01 ≤0.01 <0.01	
Italy 1997	250EC	0.13	0.042	3 (Fruit developing)	Leaf	0 7 14 28 35	1.7 0.04 <0.04 ≤0.04 <0.04	RA-2091/97 0423-97
					Beet	0 7 14 28 35	<0.01 <0.01 <0.01 ≤0.01 <0.01	
Italy 1998	250EC	0.38	0.13	3 (Root developing)	Leaf	0 28	4.1 ≤0.01	RA-2117/98 1045-98
					Beet	0 28	0.02 ≤0.01	
Italy 1998	250EC	0.38	0.13	3 (Root devloping)	Leaf	0 28	9.4 ≤0.01	RA-2117/98 1521-98
					Beet	0 28	0.01 ≤0.01	
Spain 1998	250EC	0.38	0.13	3 (Beetroot has reached harvestable size)	Leaf	0 28	4.0 ≤0.01	RA-2117/98 1522-98
					Beet	0 28	<0.01 ≤0.01	

[1] Expressed as oxydemeton-methyl.
[2] Trials conducted same location.

Table 29. Residues of oxydemeton-methyl and demeton-S-methylsulphon in fodder beet after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1998	250EC	0.36-0.40	0.13-0.14	3 (Beetroot has reached harvestable size)	Leaf	0 28	8.4 <0.01	RA-2117/98 1523-98
					Beet	0 28	0.11 <0.01	
France (South) 1998	250EC	0.38-0.42	0.13-0.14	3 (Beetroot has reached harvestable size)	Leaf	0 28	6.1 <0.01	RA-2117/98 1524-98
					Beet	0 28	0.05 <0.01	

[1] Expressed as oxydemeton-methyl.

Table 30. Residues of oxydemeton-methyl and demeton-S-methylsulphon in wheat after foliar applications of oxydemeton-methyl.

Country Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Italy 1997 (spring)	250EC	0.13	0.042	1 (Early milk)	Foliage	0	3.2	RA-2089/97 0417-97
					Grain	21 28	<0.01 <0.01	
					Straw	21 28	<0.04 <0.04	
Italy 1997 (spring)	250EC	0.13	0.042	1 (Early milk)	Foliage	0	3.8	RA-2089/97 0418-97
					Grain	21 28	<0.01 <0.01	
					Straw	21 28	0.09 <0.04	
France (South) 1997 (winter)	250EC	0.14	0.043	1 (Soft dough)	Whole plant without roots	0	2.6	RA-2089/97 0576-97
					Grain	21 28	<0.01 <0.01	
					Straw	21 28	<0.04 <0.04	
France (South) 1997 (winter)	250EC	0.13	0.042	1 (Soft dough)	Foliage	0	2.1	RA-2089/97 0577-97
					Grain	21 29	<0.01 <0.01	
					Straw	21 29	<0.04 <0.04	
France (South) 1997 (winter)	258EC	0.10	0.033	2 (Soft dough)	Foliage	0	1.6	RA-2162/97 0754-97
					Grain	21 29	<0.01 <0.01	
					Straw	21 29	<0.04 <0.04	

Country Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1998 (winter)	250EC	0.13	0.040	1 (Early dough)	Foliage	0	2.9	RA-2115/98 1043-98
					Grain	22 29	<0.02 <u>≤0.02</u>	
					Straw	22 29	<0.05 <u>≤0.05</u>	
France (South) 1998 (winter)	250EC	0.13	0.039	1 (Early dough)	Foliage	0	3.4	RA-2115/98 1517-98
					Grain	22 29	<0.02 <u>≤0.02</u>	
					Straw	22 29	0.10 <u>0.06</u>	
Spain 1998 (winter)	250EC	0.13	0.042	1 (Early dough)	Foliage	0	4.3	RA-2115/98 1518-98
					Grain	21 28	<0.02 <u>≤0.02</u>	
					Straw	21 28	0.64[2] 0.55[2]	
Italy 1998 (winter)	250EC	0.13	0.042	1 (Medium milk)	Foliage	0	1.4	RA-2115/98 1519-98
					Grain	21 28	<0.02 <u>≤0.02</u>	
					Straw	21 28	<0.05 <u>≤0.05</u>	
Italy 1998 (winter)	250EC	0.13	0.042	1 (Medium milk)	Foliage	0	1.8	RA-2115/98 1520-98
					Grain	21 28	<0.02 <u>≤0.02</u>	
					Straw	21 28	<0.05 <u>≤0.05</u>	
France (South) 1998 (winter)	258EC	0.12	0.040	2 (Early dough)	Foliage	0	1.1	RA-2086/98 1450-98
					Grain	21 28	<0.01 <u>≤0.01</u>	
					Straw	21 28	<0.04 <u>≤0.04</u>	

[1] Expressed as oxydemeton-methyl.
[2] Contamination suspected, since control sample had residues of 0.35 mg/kg.

Table 31. Residues of oxydemeton-methyl and demeton-S-methylsulphon in barley after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1997 (winter)	258EC	0.10	0.033	2 (Hard dough)	Whole plant without root	0	1.1	RA-2162/97 0753-97
					Grain	21 28	<0.01 <u>≤0.01</u>	
					Straw	21 28	<0.04 <u>≤0.04</u>	
France (South) 1998 (winter)	258EC	0.12	0.040	2 (Early dough)	Foliage	0	3	RA-2086/98 1039-98
					Grain	20 27	0.01 <u>≤0.01</u>	

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
					Straw	20 27	0.04 <0.04	

[1] Expressed as oxydemeton-methyl.

Table 32. Residues of oxydemeton-methyl and demeton-S-methylsulphon in rape after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1998	258EC	0.15	0.053	1 (15-16 leaf stage)	Foliage	0	6.8	RA-2113/98 1307-98
					Seed	248	<0.01	

[1] Expressed as oxydemeton-methyl.

Table 33. Residues of oxydemeton-methyl and demeton-S-methylsulphon in sunflower after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (North) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	1.6	RA-2154/97 0410-97
					Head	30	<0.04	
					Seed	93	<0.01	
France (North) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	2.5	RA-2154/97 0733-97
					Head	36	<0.04	
					Seed	97	<0.01	
France (North) 1998	258EC	0.12	0.040	1 (Inflorescence visible between youngest leaves)	Foliage	0	0.63	RA-2114/98 1040-98
					Head	35	<0.04	
					Seed	92	<0.01	
France (South) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	3.6	RA-2155/97 0411-97
					Head	29	<0.04	
					Seed	76	<0.01	
France (South) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	3.5	RA-2155/97 0737-97
					Head	24	<0.04	
					Seed	76	<0.01	
France (South) 1997	258EC	0.11	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	5	RA-2155/97 0738-97
					Head	20	<0.04	
					Seed	72	<0.01	

[1] Expressed as oxydemeton-methyl.

FATE OF RESIDUES IN STORAGE AND PROCESSING

A number of processing studies were reported to the 1998 JMPR for evaluation, but the residues in the raw agricultural commodities and the processed products were too low to estimate the processing factors except for apple and cotton seed. All data were from supervised trials where the plants were treated in accordance with the recommended GAP.

In view of the low levels of residues in crops treated according to GAP, the manufacturer conducted a new study in which two types of peas were fortified with oxydemeton-methyl and processed. The first was a marrowfat variety (dry harvested) used exclusively for canning and the second a vining variety (green pea) used for both canning and quick-freezing (Stanley, 2002). The raw peas were spiked by soaking in solutions of an oxydemeton-methyl formulation containing 1.0 mg/l for approximately 24 h to achieve quantifiable residues. The peas were then processed as follows.

Marrowfat peas
On removal from the spiking solution the peas were drained, washed with water, blanched for three minutes at 92 to 94.5°C, drained, cooled and foreign matter (stones, split peas etc.) was removed. The peas were then canned (approximately 220 g/can), covered with a brine preparation (9.5 g salt/37.5g sugar), sealed and sterilized at 121.1°C for six minutes or more, then cooled in water, dried overnight and deep frozen (-18°C) before analysis.

Vining peas (canning)
The process was identical to that used for marrowfat peas except the sterilization at 121.1°C was extended to 13 minutes and the can weight was 285g.

Vining peas (quick frozen)
The process was identical to the canning process up to the inspection phase. Thereafter the peas were placed in a fluidised bed freezer at -34°C for three minutes. The frozen peas were then stored at -16°C before sampling, thawing and boiling in water for one minute.

Samples of peas and processing water were analysed at critical stages by an analytical method based on that of Thornton, 1977, with modifications by Hill, 1984, which was validated for peas at fortification levels of 0.051, 0.20 and 3.13 mg/kg. Recoveries ranged from 84 to 110%.

The results showed that residues in marrowfat peas were 81.57 mg/kg and in vining peas 92.52 mg/kg, appreciably higher than the intended level of 10-50 mg/kg.

The results are shown in Table 34.

Table 34. Residues of oxydemeton-methyl peas and processed fractions.

Sample	Residues (mg/kg)	Residue, % of spiked RAC
Marrowfat peas, canning process		
Treated peas	81.75	100
Treated peas after blanching	46.56	57.0
Blanching water	5.03	6.2
Treated peas after canning	2.85	3.5
Vining peas, canning process		
Treated peas	92.52	100.0
Treated peas after blanching	53.28	57.6
Blanching water	6.33	6.8

Sample	Residues (mg/kg)	Residue, % of spiked RAC
Treated peas after canning	2.23	2.4
Vining peas, freezing and cooking process		
Treated peas	92.52	100.0
Treated peas after blanching	52.97	57.3
Treated peas after freezing	51.85	56.0
Treated peas after cooking	39.69	42.9

The results show appreciable losses of oxydemeton-methyl during blanching and canning. Losses in the blanching process (treatment for three minutes at 94.9-97°C) were more than 40% of which no more than 7% was recovered in the water. Similarly, during canning (8-13 minutes at 121.1°C) losses increased to approximately 97%. In contrast blanching and freezing resulted in a loss of approximately 44% of the original residue and the final domestic cooking of frozen vining pea samples resulted in a further reduction of 13% of the original to give a total reduction of 57% of the residues in the unprocessed peas.

The domestic cooking process (1 minute at 100°C) resulted in a reduction in the residue of 13.1%.

The processing factors derived from the study are as follows:

for processed marrowfat peas (canning)	0.035
for vining peas (canning)	0.024
for vining peas (freezing)	0.56
for vining peas (freezing + domestic cooking)	0.43

NATIONAL MAXIMUM RESIDUE LIMITS

Since the evaluation of oxydemeton-methyl by the 1998 JMPR the European Community has evaluated oxydemeton-methyl and established the MRLs shown below.

Table 34. Recommended EU MRLs for oxydemeton-methyl.

Commodity		MRL, mg/kg	Commodity		MRL, mg/kg
Citrus fruit			Legume vegetables		
	Grapefruit	0.02*		Beans (with pods)	0.02*
	Lemons	0.02*		Beans (without pods)	0.02*
	Limes	0.02*		Peas (with pods)	0.02*
	Mandarins (inc. clementines and similar hybrids)	0.02*		Peas (without pods)	0.02*
	Oranges	0.02*		Others	0.02*
	Pomelos	0.02*	Brassicas		
	Others	0.02*		Flowering Brassicas	
Tree nuts				Broccoli	0.02*
	Almonds	0.02*		Cauliflower	0.02*
	Brazil nuts	0.02*		Others	0.02*
	Cashew nuts	0.02*		Head Brassicas	
	Chestnuts	0.02*		Brussels sprouts	0.05
	Coconuts	0.02*		Head cabbage	0.05

Commodity		MRL, mg/kg
	Hazelnuts	0.02*
	Macadamia nuts	0.02*
	Pecans	0.02*
	Pine nuts	0.02*
	Pistachios	0.02*
	Walnuts	0.02*
	Others	0.02*
Pome fruit		
	Apples	0.02*
	Pears	0.02*
	Quinces	0.02*
	Others	0.02*
Stone fruit		
	Apricots	0.02*
	Cherries	0.02*
	Peaches (inc. nectarines and similar hybrids)	0.02*
	Plums	0.02*
	Others	0.02*
Berries and small fruit		
	Table and wine grapes	0.02*
	Table grapes	0.02*
	Wine grapes	0.02*
	Strawberries (other than wild)	0.02*
	Cane Fruit (other than wild)	0.02*
	Blackberries	0.02*
	Dewberries	0.02*
	Loganberries	0.02*
	Raspberries	0.02*
	Other small fruits and berries (other than wild)	0.02*
	Bilberries	0.02*
	Cranberries	0.02*
	Currants (red, black and white)	0.02*
	Gooseberries	0.02*
	Others	0.02*
	Wild berries and wild fruit	0.02*
Miscellaneous fruit		
	Avocados	0.02*
	Bananas	0.02*
	Dates	0.02*
	Figs	0.02*
	Kiwifruit	0.02*
	Kumquats	0.02*
	Litchis	0.02*
	Mangoes	0.02*
	Olives (table consumption)	0.02*

Commodity		MRL, mg/kg
	Others	0.02*
Leafy Brassicas		
	Chinese cabbage	0.02*
	Kale	0.02*
	Others	0.02*
	Kohlrabi	0.05
Leafy veg. and fresh herbs		
Lettuce and similar		
	Cress	0.05
	Lambs lettuce	0.05
	Lettuce	0.05
	Escarole	0.05
	Others	0.05
Spinach and similar		
	Spinach	0.02*
	Beet leaves (Chard)	0.02*
	Others	0.02*
	Watercress	0.02*
	Witloof	0.02*
Herbs		
	Chervil	0.02*
	Chives	0.02*
	Parsley	0.02*
	Celery leaves	0.02*
	Others	0.02*
Stem vegetables		
	Asparagus	0.02*
	Cardoons	0.02*
	Celery	0.02*
	Fennel	0.02*
	Globe artichokes	0.02*
	Leeks	0.02*
	Rhubarb	0.02*
	Others	0.02*
Funghi		
	Cultivated mushrooms	0.02*
	Wild mushrooms	0.02*
Pulses		
	Beans	0.02*
	Lentils	0.02*
	Peas	0.02*
	Others	0.02*
Oilseeds		
	Linseed	0.05*
	Peanuts	0.05*

Commodity		MRL, mg/kg	Commodity		MRL, mg/kg
	Olives (oil extract)	0.02*		Poppy seed	0.05*
	Papaya	0.02*		Sesame seed	0.05*
	Passion fruit	0.02*		Sunflower seed	0.05*
	Pineapples	0.02*		Rape seed	0.05*
	Pomegranates	0.02*		Soya bean	0.05*
	Others	0.02*		Mustard seed	0.05*
Root and tuber vegetables				Cotton seed	0.05*
	Beetroot	0.02*		Others	0.05*
	Carrots	0.02*	Potato		
	Celeriac	0.02*		Early potatoes	0.02*
	Horseradish	0.02*		Ware potatoes	0.02*
	Jerusalem artichokes	0.02*	Tea		
	Parsnips	0.02*		(Dried leaves and stalks, fermented or otherwise, Camellia sinensis)	0.05*
	Parsley root	0.02*	Hops		
	Radishes	0.02*		Including hop pellets and unconcentrated powder	0.05*
	Salsify	0.02*	Cereals		
	Sweet potatoes	0.02*		Wheat	0.02*
	Swedes	0.02*		Rye	0.02*
	Turnips	0.02*		Barley	0.1
	Yams	0.02*		Sorghum	0.02*
	Others	0.02*		Oats	0.1
Bulb vegetables				Triticale	0.02*
	Garlic	0.02*		Maize	0.02*
	Onions	0.02*		Buckwheat	0.02*
	Shallots	0.02*		Millet	0.02*
	Spring onions	0.02*		Rice	0.02*
	Others	0.02*		Other cereals	0.02*
Fruiting vegetables			Products of animal origin		
			Meat, fat and preparations of meat		0.02*
	Tomatoes	0.02*	Milk and Dairy Produce		0.02*
	Peppers	0.02*	Eggs		0.02*
	Chili peppers	0.02*			
	Aubergines	0.02*			
	Others	0.02*			
Cucurbits - edible peel					
	Cucumbers	0.02*			
	Gherkins	0.02*			
	Courgettes	0.02*			
	Others	0.02*			
Cucurbits - inedible peel					
	Melons	0.02*			
	Squashes	0.02*			

Commodity		MRL, mg/kg	Commodity			MRL, mg/kg
	Watermelons	0.02*				
	Others	0.02*				
	Sweet corn	0.02*				

APPRAISAL

Oxydemeton methyl was evaluated for residues by the 1998 JMPR within the CCPR periodic review programme and then for residues and toxicology by the JMPR in 1999 and 2002, respectively.

At its 31st Session, the CCPR asked the JMPR to clarify whether demeton-*S*-methyl and demeton-*S*-methylsulfon should remain in the residue definition of oxydemeton methyl, as it was believed that registration of these compounds would not be retained. At its Thirty-second Session, the CCPR withdrew the draft MRLs for several commodities, as there was no existing GAP for them. The Committee advanced the proposed draft MRLs to Step 5 and returned the draft MRLs to Step 6 because of intake concerns, which would be considered at its next session. The Committee requested detailed information on oxydemeton methyl. The Committee discussed the definition of the residue that had been confirmed by the 1999 JMPR. It stated that, as demeton-*S*-methyl was no longer supported and there was no GAP, its use should be prevented by removing this compound from the definition of the residue. It was pointed out, however, that demeton-*S*-methyl could not be distinguished from oxydemeton methyl in analysis and that it could be generated from oxydemeton methyl during analysis. As no agreement was reached, the Committee agreed as a compromise to maintain the present residue definition but to specify that the residue definition and MRLs apply only to residues resulting from use of oxydemeton methyl. Those conditions would be met by adding a note to the residue definition, reading: "The residue definition and MRLs are based on the use of oxydemeton methyl only."

At its 33rd Session, the CCPR noted a written comment from the European Commission stating a general reservation (lack of an acute RfD) and a specific reservation on the MRLs for grape, lemon and oranges, sweet, sour (acute risk) and decided to return the draft MRLs to Step 6. The CCPR decided to return all the MRLs to Step 6 until calculations of short-term intake had been obtained from the JMPR. The Committee was informed by the manufacturer that data would be submitted to the 2004 JMPR for a review of the definition of the residue.

The Meeting received new data on physical and chemical properties (partially updated), analytical methods, fate of residues in processing, plant metabolism (apple), residue data (apples, pears, grapes, cabbage, Brussels sprouts, cauliflower, field peas, potatoes, sugar-beet, fodder beet, wheat, barley, rape and sunflower), GAP and national MRLs.

Metabolism

Plants

The metabolism of [ethylene-1-^{14}C]oxydemeton methyl in *apple* was investigated in the field. Apple trees were sprayed on two separate occasions about 4 months before harvest (pink bud stage, BBCH 57) and then about 3 months before harvest (flowers fading, BBCH 67). The formulated material was prepared at a nominal concentration of 1.4 g ai/l and applied at a nominal rate of 350 g ai/ha. Samples of fruit and leaves were taken for analysis 2 h after the first application (leaves only), 2 h after the second application (leaves only), about 60 days before harvest (first intermediate sample; fruit and leaves), about 30 days before harvest (second intermediate sample; fruit and leaves) and at harvest (fruit and leaves).

Analysis of fruit samples (pulp and peel) from the two intermediate samples showed the presence of oxydemeton methyl, but none was found at harvest. The two intermediate samples did, however, contain desmethyl-oxydemeton methyl sulfone (metabolite 7) and two polar materials (P2

and P3). In the first intermediate sample, polar radioactivity in the pulp accounted for 17% of the radioactivity in the fruit (0.2 mg/kg). In the second intermediate sample, polar radioactivity in the pulp had decreased to 10% of that in fruit (0.05 mg/kg), and by harvest no components were detected.

In the peel extracts, polar metabolites accounted for 3% of the radioactivity in the fruit (0.023 mg/kg) at the first intermediate sampling, and this had increased to 4% (0.026 mg/kg) by the second intermediate sampling. No components were detected at harvest.

The main metabolites detected at the first intermediate sampling time in both pulp and peel were oxydemeton methyl (26.6% radioactivity in the fruit, 0.245 mg/kg) and demeton-*S*-methylsulfone (2.9% radioactivity in the fruit, 0.026 mg/kg). In the second intermediate sample, oxydemeton methyl (1.7% radioactivity in the fruit, 0.012 mg/kg) and demeton-*S*-methylsulfone (0.2% radioactivity in the fruit, 0.001 mg/kg) were detected only in peel extracts. The main component detected in pulp extracts was an unidentified polar compound (14.1% radioactivity, 0.072 mg/kg). No components were detected at harvest in either pulp or peel extracts owing to the low levels of radioactivity in the extract samples (<0.001 mg/kg). The results of this study do not change the conclusions reached in the 1998 evaluation.

Environmental fate

Soil

The aerobic degradation of oxydemeton methyl was studied in three soils for a maximum of 11 days under aerobic conditions in the dark at 20°C. [ethylene-1-^{14}C]Oxydemeton methyl was applied at a nominal rate of 0.67 mg/kg dry soil, equivalent to the proposed single maximum annual use rate of 250 g ai/ha calculated for 2.5 cm depth of soil.

During the study, the total recovery of radioactivity in individual test vessels ranged from 90.8% to 100%, and the times to 50% and 90% decomposition (DT50 and DT_{90}) in the three soils ranged from 0.17 to 0.22 and from 0.58 to 0.74 days, respectively. The results also indicate that the main metabolites were continuously degraded, that no metabolite accumulated towards the end of the study and that the bound residues participated in the natural carbon cycle of soil.

Analysis of soil extracts showed two major and one semi-major degradation product, representing 10% or more of the applied radioactivity at any time during the study. The concentration of the 2-ethylsulfinyl ethane sulfonic acid metabolite reached a maximum on day 1 and then declined gradually until day 11. Its concentration was below the LOQ towards the end of the study in soils with higher microbial activity. The 2-ethylsulfonyl ethane sulfonic acid metabolite is an oxidation product of 2-ethylsulfinyl ethane sulfonic acid, and its concentration reached a maximum on day 3 in all soils; it ranked highest, at 16.8% of the applied radioactivity. The level declined towards the end of the study in all soils, and in the most active soil to below the LOQ by day 11. Significant formation of bound residues occurred during overall metabolism of parent compound. The concentration of bound residues reached a maximum on day 11 at about 50% of the applied radioactivity. Soils showed high mineralization capacity, yielding values for $^{14}CO_2$ of > 30% by day 11. The results of study demonstrate that oxydemeton methyl is quickly degraded in aerobic soils.

Water–sediment systems

The hydrolysis of oxydemeton methyl was studied in sterile 0.01 mol/l buffer solutions, which were adjusted to pH4, 7 or 9, for a maximum of 31 days in the dark at two temperatures. The experiment was carried out in compliance with good laboratory practice (GLP) and in accordance with guidelines of the US Environmental Protection Agency, the Society of Environmental Toxicology and Chemistry, the OECD and the European Commission. The test solutions were prepared with [ethylene-1-^{14}C]oxydemeton methyl at a concentration of about 5 m g/l. The pre-test solutions were incubated for 7 days under sterile conditions in the dark at 50°C. The solutions at pH 4 and pH 7 in the main test were incubated for a maximum of 31 days under sterile conditions in the dark at 25°C.

In the pre-test at 50°C and in the main test at 25°C, oxydemeton methyl was not stable at pH 4, 7 or 9, and considerable degradation occurred. Especially at higher pH values, the compound was

thoroughly hydrolysed to desmethyl-oxydemeton methyl and 2-ethylsulfinyl-ethyl mercaptan by cleavage of the P–S bound. Furthermore, 2-ethylsulfonyl ethane sulfonic acid was observed as an oxidized P–S cleavage product at low percentages (maximum of 2.2% of the applied radioactivity), although it was identified only tentatively.

By calculation from the data obtained at 50°C, orienting DT50 values (first order) for the hydrolysis of oxydemeton methyl were estimated to be 4.9, 3.5 and 0.2 days at pH 4, 7 and 9, respectively. Using the data obtained at 25°C, the DT_{50} values (first order) were estimated to be 91, 42 and 2.5 days at pH 4, 7 and 9, respectively. At 20°C, the DT_{50} values calculated from Arrhenius plots (1/T versus ln(k)) were 174, 73 and 4.5 days at pH 4, 7 and 9, respectively. The results indicate that hydrolytic processes contribute to the degradation of oxydemeton methyl in the environment.

The quantum yield from direct photodegradation of oxydemeton methyl in water was determined according to the European Centre for Ecotoxicilogy and Toxicology of Chemicals method in polychromatic light. The quantum yield calculated from the ultraviolet absorption data and the kinetics of photodegradation was 0.00078. The resulting quantum yield and data on ultraviolet absorption in aqueous solution were used to estimate the environmental half-life of oxydemeton methyl after direct photodegradation in water in two simulation models. The calculated half-lives were 112 days in summer and 274 days in winter at 30° latitude and 200 days in May and 790 days in October at 50° latitude.

Methods of analysis

A number of methods have been developed for the analysis of residues of oxydemeton methyl in various matrices, many of which were reviewed by the 1998 Meeting as part of the periodic review of this compound. The Meeting was provided with additional methods based on the same principle as those evaluated earlier, i.e. use of the oxidation process to produce demeton-*S*-methylsulfone as the analyte. The LOQs were 0.005 mg/kg for potato; 0.005–0.01 mg/kg for apples and grapes; 0.01 mg/kg for pear, Brussels sprouts, cauliflower, cabbage, corn, sunflower, rape-seed and rape (green plant material); 0.02 mg/kg for wheat grain and 0.05 mg/kg for wheat straw.

Stability of residues in stored analytical samples

The stability of oxydemeton methyl in stored cabbage, maize, lettuce and papaya was evaluated by the 1998 JMPR, which concluded that data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton methyl were highly desirable. The available information was not representative of the various crop groups, did not cover extended storage intervals and suggested variable storage stability. The manufacture submitted new study data on the storage stability of oxydemeton methyl in several crops.

A study was conducted to determine the stability of oxydemeton methyl in spiked samples of stored apple, dried peas, potato and oil-seed rape (meal and oil at-20°C). Samples for the study were obtained from a commercial source. The samples of apple and potato were prepared for use by fine chopping in an industrial food processor; the preparation of samples of dried peas was not recorded in the report. For each crop, 20 g of prepared sample were spiked with formulated oxydemeton methyl at a nominal concentration of 0.1, 1.0 or 10 mg/kg and placed in storage at-20°C. Two samples of each were removed for analysis after 0, 3, 6, 12 and 24 months of storage. The analytical method was validated for each crop at each sampling time.

There was no substantial loss of residue during 24 months' storage from apple, potato or oil; however, the residues in dried peas and rape meal apparently decreased to half the initial values after 3 or 6 months of storage. The control samples (un-spiked samples) also showed residues of oxydemeton methyl, with <0.005–0.022 mg/kg in apple, <0.005–0.016 mg/kg in dried peas, <0.01–0.016 mg/kg in potatoes, 0.013–0.03 mg/kg in rape meal and <0.005–0.011 mg/kg in rape oil. The Meeting concluded that the data submitted on storage stability were insufficient or inadequate, and the former requirement was maintained.

Definition of the residue

The Meeting received the results of new studies of plant metabolism and supplementary information on the analytical method. The new data did not, however, provide a basis for changing the current definition of the residue. The Meeting confirmed its previous recommendation.

Results of supervised trials on crops

The results of supervised field trials on apples, pears, grapes, cabbage, Brussels sprouts, cauliflower, field peas, potatoes, sugar-beet, fodder beet, wheat, barley, rape and sunflower were submitted to the Meeting. The new data were evaluated against current GAPs, and highest residue levels were estimated for commodities evaluated by the 1998 JMPR, as the 1998 JMPR did not do so. When no residues were found in any sample in older trials in which the analytical methods used had higher LOQs than current methods, the Meeting decided to use only data from the newly submitted trials in order to avoid unnecessarily high maximum residue levels.

Citrus fruit

No data from new supervised trials were submitted. From 11 trials on *orange* and *lemon*, the 1998 JMPR estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.01 mg/kg.

In the 1998 evaluations, the reported residues in pulp, in ranked order, were <0.01 (seven), 0.01, 0.02 (two) and 0.04 mg/kg. The Meeting estimated a highest residue level of 0.04 mg/kg for orange and lemon.

Pome fruit

The results of 15 new supervised trials on apples and two on pears were submitted to the Meeting

One supervised trial on *apples* in northern France in 1998 involved higher doses than used in German GAP (0.13 kg ai/ha once, immediately after flowering). Nevertheless, the residue data could be used, since the residue levels were below the LOQ. Three supervised trials on apples in southern France in 1997 and 1998, three on apples in Italy in 1997 and 1998 and four on apples in Spain in 1997 and 1998 were conducted according to Italian GAP (0.023–0.028 kg ai/hl, 90-day PHI). As two of the four Spanish trials (conducted in 1997) were carried out in the same location under almost identical trial conditions, one residue level was taken from each. The residue levels of oxydemeton methyl in apples were <0.01 (nine) and 0.01 mg/kg.

One supervised trial on *pears* in southern France in 1998 was conducted according to Italian GAP (0.023–0.028kg ai/hl, 90-day PHI), and one trial on pears in Germany in 1998 was conducted according to Austrian GAP (0.024 kg ai/hl once, up to 2 weeks after flowering). The residue level of oxydemeton methyl in pears was <0.01 (two) mg/kg.

The 1998 JMPR evaluated 10 supervised trials on apples and pears and estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg for apple and pear, based on residue levels, in ranked order, of <0.01 (seven), 0.03 and <0.04 (two) mg/kg.

The Meeting confirmed that the residue levels found in the newly submitted trials did not exceed the formerly estimated maximum and highest residue values of 0.04 mg/kg for apple and pear in the 1998 JMPR evaluations.

Grape

Three new supervised trials from Germany were conducted within German GAP (0.027 kg ai/hl, up to fully developed inflorescence). The residue levels were <0.01 (three).

The 1998 JMPR evaluated the results of five supervised trails and estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.04 mg/kg.

The Meeting confirmed that the residue levels found in the newly submitted trials did not exceed the formerly estimated maximum and highest residue values of 0.06 mg/kg for grapes in the 1998 JMPR evaluations. The residue levels, in ranked order, were <0.04 (four) and 0.06 mg/kg.

Brassica vegetables

Cabbage (head)

The results of four new supervised trials were submitted to the Meeting.

Two supervised trials on red and white cabbage in northern France in 1996 and two supervised trials on Savoy cabbage in Germany were conducted according to German GAP (0.16 kg ai/ha (<50 cm) once, 21-day PHI). The residue levels were <0.01 (four) mg/kg.

The 1998 JMPR evaluated 16 supervised trials and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.03 mg/kg. The highest value of <0.06 mg/kg was disregarded because of the high LOQ in the older trials (1976).

The Meeting confirmed that the residue levels in the submitted trials did not exceed the formerly estimated maximum and highest residue values of 0.05 mg/kg in the 1998 JMPR evaluations. The residue levels, in ranked order, were <0.01 (six), 0.02, <0.03 (three), <0.04 and <0.05 (four) mg/kg.

Kale

No data from new supervised trials were submitted to the Meeting. Four supervised trials were evaluated by the 1998 JMPR, which estimated a maximum residue level of 0.01* mg/kg and an STMR of 0.01 mg/kg.

On the basis of the reported residue level, <0.01 (four) mg/kg, the Meeting estimated a highest residue level of 0.01mg/kg.

Kohlrabi

No data from new supervised trials were submitted to the Meeting. Four supervised trials were evaluated by the 1998 JMPR, which recommended a maximum residue level of 0.05 mg/kg and an STMR of 0.02 mg/kg. The reported residue levels, in ranked order, were <0.01 (two), 0.03 and <0.06 mg/kg. The highest value was disregarded because of the high LOQ associated with the older trials (1979).

The Meeting estimated a highest residue level of 0.05 mg/kg, at the same level as the maximum residue level recommended by the 1998 JMPR.

Brussels sprouts

Five supervised trials conducted in Germany in 1997 and 1998, two conducted in Belgium in 1997 and 1998, one conducted in the United Kingdom in 1998 and one conducted in northern France in 1998 were submitted to the Meeting; however, no comparable GAP was submitted. The Meeting could not therefore estimate a maximum residue level, an STMR or a highest residue level.

Cauliflower

The results of eight new supervised trials were submitted to the Meeting. Two trials in Germany in 1996 were conducted according to German GAP (0.16 kg ai/ha (<50 cm) once, 21-day PHI) and also according to Belgian GAP (0.15 kg ai/ ha once, 28-day PHI).

Four supervised trials in southern France in 1996 and 1997 and one in the United Kingdom in 1996 were conducted according to German GAP. One of the trials in France in 1996 involved higher doses than in German GAP, but the residue data could be used for evaluation as the level was below the LOQ.

In all the trials the residue level was <u>≤0.01</u> (eight) mg/kg. The Meeting estimated a maximum residue level of 0.01* mg/kg and STMR and highest residue values of 0.01 mg/kg for cauliflower.

Field peas (dry)

Data from two supervised trials on field peas were submitted to the Meeting, but no information on GAP was provided. The Meeting could therefore not estimate a maximum residue level, an STMR or a highest residue level.

Potatoes

The results of 20 supervised trials were submitted to the Meeting.

Two supervised trials in Germany in 1996 and one in the United Kingdom in 1996 were not matched by comparable GAP. Nine supervised trials in France in 1996 and 1998, one in Greece in 1998, three in Italy in 1997 and 1998 and four in Spain in 1997 and 1998 were conducted according to Greek GAP (0.05kg ai/hl three times, 28-day PHI). As two of four Spanish trials conducted in 1997 were carried out in the same location under similar trial conditions, one residue level was taken from each. The residue levels, in ranked order, were <0.005 (five) and ≤0.01 (11) mg/kg.

The 1998 JMPR evaluated the results of 16 supervised trials and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.02 mg/kg. The reported residue levels, in ranked order, were <0.01 (seven), <0.02 (nine) and <0.05 (two) mg/kg.

The Meeting decided to use the data from the newly submitted trials and estimated a maximum residue level of 0.01*mg/kg and STMR and highest residue values of 0.01 mg/kg to replace the former recommendations.

Sugar-beet (root)

The results of seven supervised trials on sugar-beet and two on fodder beet were submitted to the Meeting.

Three supervised trials on sugar-beet in Spain in 1997 and 1998 and four in Italy in 1997 and 1998 involved higher doses than in Spanish GAP (0.025 kg ai/hl, 30-day PHI) or Italian GAP (0.023–0.028 kg ai/hl, 30-day PHI); however, the residue data could be used for evaluation as all the levels in leaf and root were below the LOQ. As two of the three Spanish trials were conducted at the same location under the same trial conditions, one residue level was taken from each.

Two supervised trials on fodder beet in southern France in 1998 involved higher doses than in Spanish GAP (0.025 kg ai/hl, 30-day PHI), but the residue data could be used for evaluation as all the levels in leaf and root were below the LOQ. Six trials on sugar-beet and two on fodder beet could be evaluated together, as all the residue levels were ≤0.01 (eight) mg/kg.

The 1998 JMPR evaluated seven supervised trials on sugar-beet and two on fodder beet in Germany, and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.04 mg/kg. The reported residue levels, in ranked order, were <0.01 (four) and <0.04 (five) mg/kg.

The Meeting decided to use the data from the newly submitted trials and estimated a maximum residue level of 0.01*mg/kg and an STMR of 0.01 mg/kg, to replace the former recommendations.

Wheat, barley and rye

Eleven supervised trials on wheat and two on barley were submitted to the Meeting.

Four supervised trials on wheat in Italy in 1997 and 1998, six in southern France in 1997 and 1998 and one in Spain in 1998 involved higher doses than in Italian GAP (0.023–0.028 kg ai/hl, 30-day PHI), but the data on residues on wheat grain could be used for evaluation as all the levels were below the LOQ. The residue levels, in ranked order, were <0.01 (six) and <0.02 (five) mg/kg.

Two supervised trials on barley in southern France in 1997 and 1998 involved slightly higher doses than in the Italian GAP for wheat. The Meeting concluded that this GAP could be applied to trials on barley, as the two crops are cultivated similarly. The residue levels were <0.01 (two) mg/kg.

The 1998 JMPR evaluated seven supervised trials on wheat and three on barley and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.04 mg/kg. The reported residue levels, in ranked order, were <0.04 (seven) and <0.05 (three) mg/kg.

The Meeting decided to use the data from the newly submitted trials to estimate the maximum residue level. The combined residue levels, in ranked order, were ≤0.01 (eight) and <0.02 (five)

mg/kg. The Meeting estimated a maximum residue level of 0.02*mg/kg and an STMR of 0.01 mg/kg, to replace the former recommendations.

Rape-seed

One supervised trial on rape was submitted to the Meeting; however, no information on GAP was provided. The Meeting could therefore not estimate a maximum residue level, an STMR or a highest residue level.

Sunflower seed

Six supervised trial data on sunflower were submitted to the Meeting; however, no information on GAP was provided. The Meeting could therefore not estimate a maximum residue level, an STMR or a highest residue level.

Sugar-beet (tops)

The residue levels in the leaves of sugar-beets and fodder beets treated according to GAP, in ranked order, were ≤0.01 (five) and <0.04 (three) mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR of 0.01 mg/kg and a highest residue level of 0.04 mg/kg, to replace the former recommendations.

Wheat, barley and rye straw and fodder

The residue levels in straw and fodder from wheat and barley, in ranked order, were ≤0.04 (eight), <0.05 (three) and 0.06 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR of 0.04 mg/kg and a highest residue level of 0.06 mg/kg, to replace the former recommendations.

Fate of residues during processing

The results of studies on residues in processed peas were provided to the Meeting. The samples were fortified with diluted oxydemeton methyl formulation by soaking because the levels of residue in samples produced under GAP conditions were expected to be too low. The reported processing factors were 0.034 for processed marrowfat peas (canning), 0.024 for vining peas (canning), 0.43 for vining peas (freezing) and 0.43 for vining peas (freezing and domestic cooking).

Residues in animal commodities

The 1998 JMPR, estimated the dietary burden of farm animals and concluded that quantifiable residues of demeton-*S*-methyl, oxydemeton methyl or demeton-*S*-methylsulfone are unlikely to occur in commodities of animal origin (meat, milk, poultry and egg). Therefore, MRLs could be set at the practical LOQ of 0.05* mg/kg for all commodities except milk and at 0.01* mg/kg for milk. The current Meeting did not recommend the addition of further feed items or an increase in the recommended residue levels. It therefore confirmed the previous maximum residue levels and STMRs for commodities of animal origin and estimated a highest residue level of 0 mg/kg for cattle fat, eggs, meat of cattle, pigs and sheep, pig fat, poultry fats, poultry meat and sheep fat.

Further work or information

Desirable

Data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton methyl are highly desirable, as the information provided was not representative of the various crop groups, did not cover extended storage and suggested variable storage stability.

RECOMMENDATIONS

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

Definition of the residue for compliance with MRLs and dietary intake: Sum of oxydemeton-methyl, demeton-S-methyl and demeton-S-methylsuphon expressed as oxydemeton-methyl.

The definition of the residue and MRLs are based on the use of oxydemeton-methyl only.

Commodity		Recommendations, mg/kg			
CCN	Name	New	Previous	STMR or STMR-P	HR or HR-P
FP 0226	Apple		0.05[1]	0.01	0.04
JF 0226	Apple juice			0.01	
	Apple sauce			0.005	
GC 0640	Barley	0.02(*)	0.05(*)	0.01	
AS 0640	Barley straw and fodder, dry	0.1	2		
VB 0041	Cabbage, Head		0.05(*)[1]	0.03	0.05
MF 0812	Cattle fat		0.05(*)	0	0
VB 0404	Cauliflower	0.01(*)	W	0.01	0.01
VD 0526	Common bean (dry)		0.1	0.01	
SO 0691	Cotton seed		0.05	0.01	
OR 0691	Cotton seed oil, edible			0.002	
PE 0112	Eggs		0.05(*)	0	0
FB 0269	Grape		0.1[1]	0.04	0.06
VL 0480	Kale		0.01(*)	0.01	0.01
VB 0405	Kohlrabi		0.05	0.02	0.05
FC 0204	Lemon		0.2	0.01	0.04
MM 0097	Meat of cattle, pigs and sheep		0.05(*)	0	0
ML 0106	Milks		0.01(*)	0	
FC 0004	Oranges, Sweet, Sour		0.2[1]	0.01	0.04
FP 0230	Pear		0.05	0.01	0.04
MF 0818	Pig fat		0.05(*)	0	0
VR 0589	Potato	0.01(*)	0.05(*)	0.01	0.01
PF 0111	Poultry fat		0.05(*)	0	0
PM 0110	Poultry meat		0.05(*)	0	0
GC 0650	Rye	0.02(*)	0.05(*)	0.01	
AS 0650	Rye straw and fodder, dry	0.1	2		
MF 0822	Sheep fat		0.05(*)	0	0
VR 0596	Sugar beet	0.01(*)	0.05(*)	0.01	
AV 0596	Sugar beet leaves or tops	0.05	0.05(*)		
GC 0654	Wheat	0.02(*)	0.05(*)	0.01	
AS 0654	Wheat straw and fodder, dry	0.1	2		

FURTHER WORK OR INFORMATION

Desirable

Data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton-methyl are highly desirable. The information was not

representative of the various crop groups, did not cover extended storage, and suggested variable storage stability.

DIETARY RISK ASSESSMENT

Long-term intake

STMR or STMR-P values were estimated by the 1998 JMPR and by the present Meeting for 27 commodities. When data on consumption were available, these values were used in the estimates of dietary intake.

The dietary intake from the five GEMS/Food regional diets, on the basis of the STMR values, represented 3–30% of ADI (Annex 3 of the Report). The Meeting concluded that the intake of residues of oxydemeton methyl resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI for oxydemeton methyl was calculated for the commodities for which maximum residue levels, STMR values and highest residue levels were established and for which data on consumption (of large portions and unit weight) were available. The results are shown in Annex 4 of the Report.

The ARfD for oxydemeton methyl is 0.002 mg/kg bw. The IESTI represented 0–220% of the ARfD for children and 0–90% of that for the general population. For children, 100% of the ARfD was exceeded in apple (130%), cabbage (120%), grape (220%) and orange (120%).

The Meeting concluded that the short-term intake of residues of oxydemeton methyl from uses on commodities other than apples, cabbages, grapes and oranges that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Babczinski, P. 2001a. Oxvdemeton-methyl [(R)Metasystox R]:Aerobic degradation and metabolism of Oxydemeton-methyl in three soils. Bayer AG, Report No.: MR-457/01, Date: 29.11.2001, GLP, unpublished, Owner: ODM Task Force.

Babczinski, P. 2001b. Oxydemeton-methyl [(R) Metasystox R]: Hydrolysis of oxydemeton-methyl in sterile aqueous buffer solutions. Bayer AG, Report No.:MR-500/01, Date:29.11.2001, unpublished, Owner: ODM Task Force.

Blass, W. 1999. Determination of residues of Oxydemeton-methyl and Beta-Cyfluthrin in/on cauliflower after treatment with Enduro 258 EC in the field in France. (Study numbers: 0409-97 and 0750-97), Bayer AG, Report No.: RA-2160/97, Date 28.10.1998, GLP, unpublished.

Blass, W. and Block, H. 1997. Determination of residues of Enduro 258EC in/on sunflower in the field in France. (Study numbers: 0411-97, 0737-97 and 0738-97), Bayer AG, Report No.: RA-2155/97, Date 08.02.1999, GLP, unpublished.

Blass, W. and Block, H. 1999a. Determination of Enduro 258 EC in/on apple in the field in France. (Study numbers:0407-97 and 0742-97), Bayer AG, Report No.: RA-2156-97, Date 20.01.1999, GLP, unpublished.

Blass, W. and Block, H. 1999b. Determination of Enduro 258 EC in/on apple in the field in France. (Study numbers: 0406-97 and 0745-97), Bayer AG, Report No.: RA-2157/97, Date 11.01.1999, GLP, unpublished.

Blass, W. and Block, H. 1999c. Determination of Enduro 258 EC in/on winter barley and winter wheat in the field in France. (Study numbers: 0754-97 and 0753-97), Bayer AG, Report No.: RA-2162/97. Date 28.01.1999, GLP, unpublished.

Blass, W. and Block, H. 1999d. Determination of residues of Enduro 258EC in/on field pea in the field in France. (Study numbers: 0408-97 and 0751-97), Bayer AG, Report No.:RA-2161/97, Date 15.01.1999, GLP, unpublished.

Blass, W. and Block, H. 1999e. Determination of residues of Enduro 258EC in/on sunflower in the field in France. (Study numbers: 0410-97 and 0733-97), Bayer AG, Report No.: RA-2154/97, Date 07.01.1999, GLP, unpublished.

Blass, W. and Block, H. 1999f. Determination of residues of Metasystox R 250EC on Brussels sprouts in the field in Belgium, Germany, France and Great Britain. Study numbers: 1044-98, 1531-98, 1514-98, 1515-98 and 1516-98), Bayer AG, Report No.:RA-2118/98, Date 26.07.1999, GLP, unpublished.

Blass, W. and Philipowski, C. and Preu, M. 2001a. Analytical method 00585 for the determination of residues of oxydemeton-methyl in/on plant materials by GC-FPD. Bayer AG, Report No.:00585, Date

08.08.2001, GLP, unpublished, Owner: ODM Task Force.

Blass, W., Philipowski C. and Preu, M. 2001b. Analytical method 00585/M001 for the determination of residues of oxydemeton-methyl in/on wheat grain, foliage and straw by GC-FPD. Bayer AG, Report No.: 00585/N001, Date 08.08.2001, GLP, unpublished, Owner: ODM Task Force.

Eberz, A. 1999. Determination of safety-relevant data Metasystox R VL50. Bayer AG, Report No.: 99/00454, Date 27.04.2000, GLP, unpublished, Owner: ODM Task Force.

Heinemann, O and Seym, M. 1997. Determination of residues of Enduro 250 EC in/on potato following spray application in the field in France. (Study numbers: 0666-96, 0667-96, 0668-96 and 0781-96), Bayer AG, Report No.: RA-2149/96, Date 03.12.1997, GLP, unpublished.

Heinemann, O. and Seym, M. 1998a. Determination of residues of Enduro 258 EC in/on potato following spray application in the field in Germany, Great Britain and France. (Study numbers: 0661-96, 0662-96, 0663-96 and 0664-96), Bayer AG, Report No.: RA-2147/96, Date 20.02.1998, GLP, unpublished.

Heinemann, O. and Seym, M. 1998b. Determination of residues of Enduro 258 EC in/on potato following spray application in the field in France. (Study numbers: 0665-96, 0773-96 and 0774-96), Bayer AG, Report No.:RA2148/98, Date 20.02.1998, GLP, unpublished.

Heinemann, O. and Seym, M. 1998c. Determination of residues of Enduro 250 EC in/on Savoy cabbage, white cabbage and red cabbage following spray application in the field in Germany and France. (Study numbers:0676-96, 0677-96, 0678-96 and 0679-96), Bayer AG, Report No.:RA-2150/96, Date 27.04.1998, GLP, unpublished.

Hellpointner, E. 1992. Determination of the quantum yield and assessment of the environmental half-life of the direct photodegredation of oxydemeton-methyl in water. Bayer AG, Report No.:PF3663,Date:20.02.1992, GLP, unpublished, Owner: ODM Task Force.

Hoffman, M. 2001. Determination of residues of Oxydemeton-methyl and Beta-Cyfluthrin in/on winter barley and winter wheat after spray application of Enduro 258 EC in the field in France. (Study numbers: 1450-98 and 1039-98), Bayer AG, Report No.: RA-2086/98. Date 26.09.2001 GLP, unpublished.

Hoffman, M. 2001a. Determination of residues of Oxydemeton-methyl in/on winter wheat after spray application of Metasystox R 250EC in the field in Spain, Italy and France. (Study numbers: 1043-98, 1517-98, 1518-98, 1519-98 and 1520-98), Bayer AG, Report No.: RA-2115/98, Date 24.09.2001, GLP, unpublished.

Hoffman, M. 2001b. Determination of residues of Oxydemeton-methyl in/on potato after spray application of Metasystox R 250EC in the field in Italy, France, Spain and Greece. (Study numbers: 1046-98, 1507-98, 1509-98, 1510-98, and 1512-98), Bayer AG, Repor No.: RA-2116/98, Date 18.10.2001, GLP, unpublished.

Hoffman, M. 2001c. Determination of residues of Oxydemeton-methyl in/on sugar beet and fodder beet after spray application of Metasystox R 250EC in the field in Spain, Italy and France. (Study numbers: 1045-98, 1521-98, 1522-98, 1523-98 and 1524-98), Bayer AG, Report No.:RA-2117/98, Date 28.09.2001, GLP, unpublished.

Hoffman, M. 2001d. Determination of residues of Oxydemeton-methyl and Beta-Cyfluthrin in/on winter rape after spray application of Enduro 258EC in the field in southern France. (Study number: 1307-98), Bayer AG, Report No.: RA-2113/98, Date 26.09.2001, GLP, unpublished.

Hoffman, M. 2001e. Determination of residues of Oxydemeton-methyl and Beta-Cyfluthrin in/on sunflower after spray application of Enduro 258EC in the field in northern France. (Study number: 1040-98), Bayer AG, Report No.: RA-2114/98, Date 00.00.0000, GLP, unpublished.

Hoffman, M. 2001f. Determination of residues of Oxydemeton-methyl and Beta-Cyfluthrin in/on pear and apple after spray application of Enduro 258 EC in the field in Germany and France. (Study numbers: 1036-98 and 1361-98), Bayer AG, Report No.:RA-2029/98, Date 26.09.2001, GLP, unpublished.

Hoffman, M. 2001g. Determination of residues of Oxydemeton-methyl and Beta-Cyfluthrin in/on pear and apple after spray application of Enduro 258 EC in the field in France. (Study numbers: 1037-98 and 1362-98), Bayer AG, Report No.:RA-2030/98, Date 26.09.2001, GLP, unpublished.

Hoffman, M. 2001h. Determination of residues of Oxydemeton-methyl in/on apple after spray application of Metasystox R 250 EC in the field Spain, Portugal, Italy and France. (Study numbers: 1024-98, 1364-98, 1365-98, 1366-98, and 1367-98), Bayer AG, Report No.:RA-2031/98, Date 24.09.2001, GLP, unpublished.

Krohn,J 1993. Vapour Pressure Curve of Oxydemeton-methyl. Bayer AG, Report No.: MO-00-007155, Date 08.06.1993, GLP, unpublished, Owner: ODM Task Force.

Krohn, J. 1999. Density and surface tension of Oxydemeton-methyl. Bayer AG, Report No.: 14032980, Date 18.05.1999, GLP, unpublished, Owner: ODM Task Force.

Krohn, J. 2002. Solubilities of Oxydemeton-methyl in water and in organic solvents. Bayer AG, Report No.: REG02-0011, Date 19.02.2002, GLP, unpublished, Owner: ODM Task Force.

McEwen, A. 2001. [^{14}C]-Oxydemeton-methyl: Metabolism in apples, BioDynamics UK. Bayer AG, Report No.: JRF/01, Date 20.05.2001, GLP, unpublished, Owner: ODM Task Force.

McEwen, A.2002. [14C]-oxydemeton-methyl: metabolism in Apples. BioDynamics UK. Bayer AG, Report No.: JRF/01.Date: 20.05.2002,GLP, unpublished, Owner: ODM Task Force.

Nusslein, F. 1999. Determination of Metasystox R 250EC on sugar beet in the field in Italy and Spain. (Study numbers: 0580-97 and 0581-97), Bayer AG, Report No.: RA-2091/97, Date 09.02.1999, GLP, unpublished.

Nusslein, F., Block, H. 1997. Determination of residues of Metasystox R 250EC in/on Brussels sprouts in the

field in the Federal Republic of Germany, Great Britain and Benelux. (Study numbers: 0419-97, 0420-97, 0574-97 and 0575-97), Bayer AG, Report No.: RA-2088/97, Date 28.01.1999, GLP, unpublished.

Nusslein, F. and Block, H. 1999. Determination of residues of Metasystox R 250EC in/on potato in the field in Spain and Italy. (Study numbers: 0426-97, 0428-97, 0695-97 and 0696-97), Bayer AG, Report No.: RA-2087/99, Date 08.02.1999, GLP, unpublished.

Nusslein, F., Block, H. 1999a. Determination of Metasystox R 250EC in/on apple in the field in Italy and Spain. (Study numbers:0424-97, 0425-97, 0571-97 and 0572-97), Bayer AG, Report No.:RA-2109-27. Date 09.03.1999, GLP, unpublished.

Nusslein, F., Block, H. 1999b. Determination of Metasystox R 250EC in/on grape in the field in the Federal Republic of Germany. (Study numbers: 0516-97, 0578-97, 0578-97, and 0579-97.), Bayer AG, Report No.: RA-2090-97, Date 28.01.1999, GLP, unpublished.

Nusslen, F., Block, H. 1999c. Determination of Metasystox R 250EC in/on spring wheat and winter wheat in the field Italy and France. (Study numbers: 0417-97, 0418-97, 0576-97 and 0577-97), Bayer AG, Report No.: RA-2089/99, Date 03.05.1999, GLP, unpublished.

Schoning, R 1998. Supplement E008 of method 00255 for the determination for residues of Bata-Cyfluthrin in/on following sample materials: Apple and Pear, Oxydemeton-methyl in/on following sample materials: winter wheat/grain and spring barley/grain. Bayer AG, Report No.: MR-605/98, Date 01.09.1998, GLP, unpublished.

Schoning, R 1998a. Supplement E009 of method 00255 for the determination for residues of Oxydemeton-methyl in/on following sample materials: Apple, Pear, Grape and Brussels sprouts. Bayer AG, Report No.: MR-282/98, Date 18.05.1998, GLP, unpublished.

Schoning, R. 1998b. Determination of residues of Enduro 250 EC on cauliflower following spray application in the field in Germany, Great Britain and France. (Study numbers: 0669-96, 0670-96, 0671-96 and 0672-96), Bayer AG, Report No.: RA-2152/96, Date 04.08.1998, GLP, unpublished.

Schoning, R. 1998c. Determination of residues of Enduro 250 EC on cauliflower following spray application in the field in France. (Study numbers: 0673-96 and 0675-96), Bayer AG, Report No.: RA-2153/96, Date 31.07.1998, GLP, unpublished.

Schoning, R 2001. Supplement E012 on the residue analytical method 00255 for the determination for residues of Beta-Cyfluthrin/oxydemeton-methyl in plant materials. Bayer AG, Report No.:MR-396/00, Date 10.09.2001, GLP, unpublished.

Seym, M. 1994. Supplement E001 to method 00255 for the determination of residues of Cyfluthrin in/on Apple, Beta-Cyfluthrin and Oxydemeton-methyl in/on the following plant materials: Cauliflower, Corn and Sunflower. Bayer AG, Report No.: RA-181/94, Date 12.10.1994, GLP, unpublished.

Seym, M. 1995. Amendment E002 to method 00255 for determination of residues of Bata-Cyfluthrin and Oxydemeton-methyl in/on cauliflower. Bayer AG, Report No.: MR-182/94, Date 10.04.1995, GLP, unpublished.

Seym, M. 1996. Supplement E004 of method 00255 for the determination of residues of Bata-Cyfluthrin in/on following sample materials: Alfalfa, Round Cabbage and Tomato. Bayer AG, Report No.:MR-452/96. Date 28.06.1996, GLP, unpublished.

Seym, M. 1996a. Supplement E005 of method 00255 for the determination of residues of Cyfluthrin in/on Apple, Beta-Cyfluthrin in/on Savoy Cabbage, Red Cabbage and the Green Cabbage and Oxydemeton-methyl in/on Savoy Cabbage and Red Cabbage. Bayer AG, Report No.: MR-504/96, Date 18.11.1996, GLP, unpublished.

Smith, J.S.C 2002. Oxydemeton-methyl Residues in Apples, Dried Peas, Potatoes and Oilseed Rape (Meal and Oil). Freezer Storage Stability Trials. GC Laboratories Study No.:J13374, Date 21.03.2002, unpublished, Owner: ODM Task Force.

Stanley, R. 2002. Sample fortification, processing and residues analysis of peas treated with oxydemeton-methyl C.C.F.R.A., Report No.: AG/REP/61081/01, Date 11.03.2002, GLP, unpublished. Owner: ODM Task Force

Cross reference for report numbers

00585	Blass, W., Philipowski, C., Preu,M. 2001a,
00585/N001	Blass, W., Philipowski, C., Preu, M. 2001,
14032980	Krohn, J. 1999,
99/00454	Eberz, A. 1999,
AG/REP/61081/01	Stanley, R. 2002,.
J13374	Smith, J.S.C 2002,
JRF/01	McEwen, A. 2001,
JRF/01	McEwen, A.2002,
MO-00-007155	Krohn,J 1993,
MR-182/94	Seym, M. 1995,
MR-282/98	Schoning, R 1998a,
MR-396/00	Schoning, R 2001,
MR-452/96	Seym, M. 1996,
MR-457/01	Babczinski, P. 2001a
MR-500/01	Babczinski, P. 2001a,
MR-504/96	Seym, M. 1996a,
MR-605/98	Schoning, R 1998,
PF3663	Hellpointner, E. 1992,
RA-181/94	Seym, M. 1994,
RA-2029/98	Hoffman, M. 2001f,
RA-2030/98	Hoffman, M. 2001g,
RA-2031/98	Hoffman, M. 2001h,
RA-2086/98	Hoffman, M. 2001,
RA-2086/98	Hoffman, M. 2001a,
RA-2087/99	Nusslein, F. and Block, H. 1999,
RA-2088/97	Nusslein, F., Block, H. 1997,
RA-2089/99	Nusslen, F., Block, H. 1999c,
RA-2090-97	Nusslein, F., Block, H. 1999b,
RA-2091/97	Nusslein, F. 1999,

RA-2109-27 Nusslein, F., Block, H. 1999a,
RA-2117/98 Hoffman, M. 2001c,
RA-2113/98 Hoffman, M. 2001d,
RA-2114/98 Hoffman, m. 2001e,
RA-2116/98 Hoffman, M. 2001b,
RA-2118/98 Blass, W., Block, H. 1999f,.
RA-2147/96 Heinemann, O. and Seym, M. 1998a,
RA2148/98 Heinemann, O. and Seym, M. 1998b,
RA-2149/96 Heinemann, O and Seym, M. 1997,
RA-2150/96 Heinemann, O., Seym, M. 1998,
RA-2152/96 Schoning, R. 1998b,
RA-2153/96 Schoning, R. 1998c,
RA-2154/97 Blass, W., Block, H. 1999e,
RA-2155/97 Blass, W., Block, H. 1997,
RA-2156-97 Blass, W., Block, H. 1999a,
RA-2157/97 Blass, W. and Block, H. 1999c,
RA-2160/97 Blass, W. 1999,
RA-2161/97 Blass, W., Block, H. 1999d,
RA-2162/97 Blass, W., Block, H. 1999,
RA-2162-97 Blass, W., Block, H. 1999b,
REG02-0011 Krohn, J. 2002,