METHAMIDOPHOS (100)

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EXPLANATION

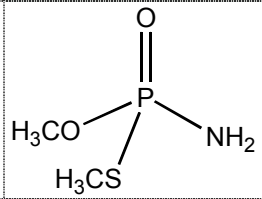
Methamidophos was evaluated initially in 1976 for residues and toxicology and the latest residues evaluation was in 1997. It was identified as a priority compound under the Periodic Re-evaluation Programme of the 29th Session of the CCPR, for review by 2002 JMPR (ALINORM 97/24A). At the 31st Session of the CCPR, the Committee noted that an acute RfD would be established by the 2000 JMPR but it was finally established in 2002 JMPR. The Meeting received information on methamidophos metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials, farm animal feeding studies, fate of residues in processing and national MRLs.

The 2002 JMPR established an ADI and acute RfD for methamidophos of 0-0.004 mg/kg bw and 0.01 mg/kg bw, respectively.

Some information on GAP, national MRLs and residue data was submitted by Australia, Germany and the Netherlands.

Methamidophos is a broad-spectrum organophosphate insecticide with uses on many crops. It is also formed as a metabolite of the insecticide acephate.

IDENTITY

ISO common name:	methamidophos
Company code numbers:	RE-9006, KRJ 230184, Ortho 9006, SRA 5172
Chemical names	
IUPAC:	thiophosphoramidic acid, *O,S*-dimethyl ester
CA:	phosphoramidothioic acid, *O,S*-dimethyl ester
CAS number:	10265-92-6
CIPAC number:	355
Molecular formula:	$C_2H_8NO_2PS$
Relative molecular mass:	141.1
Structural formula:	

Physical and chemical properties

Pure active ingredient

Appearance:	colourless crystals
Melting point:	45 °C (Krohn, 1994)
Boiling point:	not applicable
Relative density	1.27 g/cm^3 at 20 °C (Krohn, 1995)
Vapour pressure:	2.3×10^{-3} Pa at °C (Weber, 1988) 4.7×10^{-3} Pa at °C
Henry's law constant:	1.6×10^{-6} Pa m^3 mol^{-1} (calculated, water solubility 200 g l^{-1}) (Krohn, 1988)
Solubility in water:	>200 g/l at 20 °C (Krohn, 1987a)
Octanol/water partition coefficient:	log P_{ow} = -0.80 at 20 °C (Krohn, 1987b)

Hydrolysis:	pH 4: t½ approx 124 hours at 70 °C (Krohn 1984) pH 7: t½ 137 hours at 20 °C pH 9: t½ 85 hours at 20 °C
Half-life of ^{14}C-methamidophos in sterile aqueous buffers	pH 5: <8% degradation in 30 days at 25 °C, t½ 309 days pH 7: ca. 50% degradation in 30 days at 25 °C, t½ 27 days pH 9: 78% degradation in 7 days at 25 °C, t½ 3 days (extrapolated) (Chopade, 1985a)
Dissociation constant	no dissociation
Photo-stability in water	sunlight: 10% degradation in 30 days, t½ 90 days (extrapolated). continuous artificial light: ca. 4% degradation in 5 days, t½ 37 days (extrapolated) (Chopade, 1985b)

Technical material

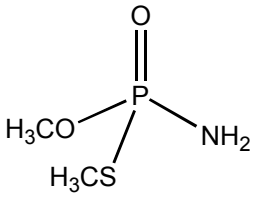
Appearance:	Colourless to yellowish liquid of crystal slurry
Solubility in organic solvents	*n*-hexane : <1 g/l at 20 °C toluene 2-5 g/l at 20 °C dichloromethane >200 g/l at 20 °C 2-propanol >200 g/l at 20 °C acetone >200 g/l at 20 °C dimethylformamide >200 g/l at 20 °C (Krohn, 1987c)

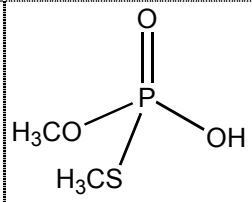
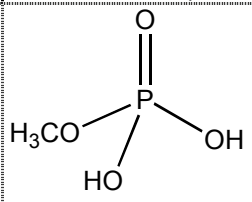
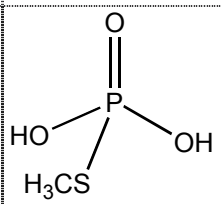
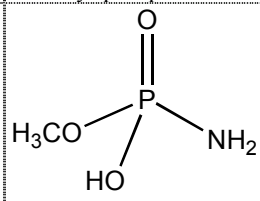
Formulations

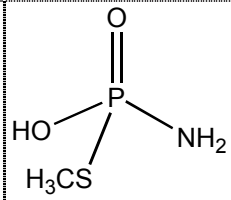
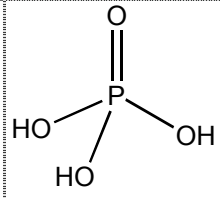
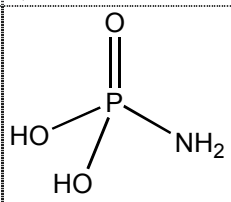
Methamidophos is available as emulsifiable concentrates (EC) and soluble concentrates (SL).

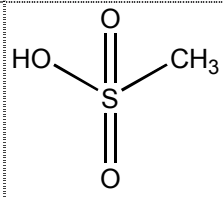
METABOLISM

Methamidophos and its metabolites were given various trivial names, systematic names and code numbers in study reports. These are summarised below.

Metabolite No.	Term used in evaluation	Formulae, CAS number/name, other names/codes used in study reports	Study reports
ai	methamidophos	 stoichiometric formula: $C_2H_8NO_2PS$ CAS No: 10265-92-6 CAS: phosphoramidothioic acid, *O,S*-dimethyl ester RE-9006 KRJ 230184 Ortho 9006 SRA 5172	Animals rat dairy goat laying hen Plants bean cabbage lettuce potato tobacco tomato Soil aerobic metabolism anaerobic metabolism photolysis microbial degradation Water hydrolysis photolysis aerobic metabolism

Metabolite No.	Term used in evaluation	Formulae, CAS number/name, other names/codes used in study reports	Study reports
M01	desamino-methamidophos, DMPT	 stoichiometric formula: $C_2H_7O_3PS$ CAS No: 42576-53-4 CAS: phosphorothioic acid, *O,S*-dimethyl ester dimethyl phosphorothioate *O,S*-dimethyl O-phosphorothioate *O,S*-dimethyl phosphorothioate *O,S*-dimethyl phosphorothioate, sodium salt *O,S*-dimethyl phosphorothioic acid *O,S*-dimethyl phosphorothiolate *O,S*-dimethyl phosphorothiolate, NH_4^+ salt *O,S*-dimethyl phosphorthioate *O,S*-dimethyl thiophosphoric acid DMPT Note: Ortho 18421, RE-18421 = anion of M01	Animals rat dairy goat laying hen Plants cabbage tomato cell cultures Soil aerobic metabolism photolysis Water hydrolysis photolysis
M02	monomethyl phosphate	 stoichiometric formula: CH_5O_4P CAS No: 812-00-0 CAS: monomethyl phosphate methyl dihydrogen phosphate methyl phosphate MDP	Animals rat Plants [cabbage] [tomato] [cell culture]
M03	*S*-methyl thiophosphoric acid	 stoichiometric formula: CH_5O_3PS *S*-methyl-phosphorothiolate *S*-methyl-phosphorothiolic acid	Animals rat Plants lettuce
M04	methyl phosphoramidate	 stoichiometric formula: CH_6O_3PN *O*-methyl phosphoramidate *O*-methyl phosphoric acid amide desthiomethyl-methamidophos	Animals rat

Metabolite No.	Term used in evaluation	Formulae, CAS number/name, other names/codes used in study reports	Study reports
M05	S-methyl phosphoramidothioate	 stoichiometric formula: CH_6O_2PNS CAS No: 17808-29-6 CAS: phosphoramidothioic acid-S-methyl ester desmethamidophos desmethyl-methamidophos *O*-desmethyl methamidophos *S*-methyl phosphoramidothioate *S*-methyl phosphoramidothiolate *S*-methyl phosphoramidothiolate, as NH_4^+ salt *S*-methyl phosphoramidothioic acid *S*-methyl phosphoroamidothioate SMPAA RE-54543 RIB 13823	Animals rat dairy goat laying hen Soil aerobic metabolism anaerobic metabolism photolysis Water hydrolysis photolysis
M06	phosphoric acid	 stoichiometric formula: H_3PO_4 CAS No: 7664-38-2 CAS: phosphoric acid inorganic phosphate phosphate PA IV	Animals rat Plants [cabbage] [tomato] [cell cultures]
M07	phosphoramidic acid	 stoichiometric formula: H_4O_3PN CAS No: CAS: phosphoramidate amidophosphoric acid phosphoramidic acid dihydroxyphosphoramidate	Animals [rat]
M08	methyl mercaptan	CH_3-SH stoichiometric formula: CH_4S CAS No: 74-93-1 CAS: methyl mercaptan mercaptan methanethiol methylmercaptane	Animals [rat] Plants [cabbage] [tomato] [cell cultures] Soil aerobic metabolism anaerobic metabolism
M09	dimethyl sulfide	CH_3-S-CH_3 stoichiometric formula: C_2H_6S CAS: dimethyl sulfide	

Metabolite No.	Term used in evaluation	Formulae, CAS number/name, other names/codes used in study reports	Study reports
M10	dimethyl disulfide	CH_3-S_2-CH_3 stoichiometric formula: $C_2H_6S_2$ CAS No: 624-92-0 CAS: dimethyl disulfide DMDS	Soil aerobic metabolism anaerobic metabolism Water hydrolysis
M11	methane	CH_4 stoichiometric formula: CH_4	
M12	methanol	CH_3OH stoichiometric formula: CH_3OH	Plants [cabbage] [tomato] [cell cultures]
M21	methanesulfonic acid	 stoichiometric formula: CH_4O_3S CAS No: 75-75-2 CAS: methanesulfonic acid MSA	Plants lettuce potato
M22	sulfate	SO_4^{2-}	Animal rat
M23	formic acid	HCOOH	Animal rat
CO_2	carbon dioxide	CO_2	Animals rat Plants tobacco Soil aerobic metabolism anaerobic metabolism Water aerobic metabolism

Animal metabolism

The Meeting received animal metabolism studies for methamidophos in rats, lactating goats and laying hens. The data for rats were reported in the evaluation of toxicology by the 2002 JMPR and are not repeated here.

Goats

The metabolism of ^{14}C-methamidophos in lactating goats was followed in three studies.

In the first, Crossley and Lee (1972) studied the fate of [*S*-methyl-^{14}C]methamidophos in lactating goats (*ca.* 19 kg bw). Goats were orally dosed by capsule for seven consecutive days, at a nominal level equivalent to 2 ppm methamidophos in the diet. Animals were slaughtered 11 days after the final dose. Radioactivity in milk was extracted with ethyl acetate and from tissues with ethyl acetate and acetonitrile. Analysis of residues was by GLC and TLC. This study, with results for additional goats dosed with acephate and acephate/methamidophos residues, is also reported in the evaluation for acephate.

Approximately 26% administered dose was excreted during the course of the study (urine 18%, faeces 4.7%, milk 3.2%). Total accountability of the administered dose, assuming that fat and muscle comprised 5 and 40% of body weight, was estimated to be 69%.

^{14}C residues observed in tissues of goats slaughtered 11 days after the final dose were similar for all the tissues analysed (Table 1). Only small amounts of radioactivity were extracted with ethyl acetate and the proportion of the ^{14}C extracted (2-5% of TRR) was similar regardless of the tissue (Table 2). From this it was inferred that the ^{14}C was mostly associated with metabolic pool

extractables, as both ethyl acetate and acetonitrile were expected to extract residues of methamidophos almost quantitatively.

Table 1. Distribution of ^{14}C residues in tissues and milk from a [*S*-methyl-^{14}C]methamidophos-treated goat, slaughtered 11 days after last dosing at 2 ppm (Crossley and Lee, 1972).

Tissue	TRR (mg/kg expressed as methamidophos)
Liver	0.22
Kidney	0.16
Fat	0.16
Muscle	0.16
Milk	
evening 4th dose	0.26
24 h post dose	0.30
48 h post dose	0.14
168 h post dose	0.03

Table 2. Radioactivity extracted from goat tissues with ethyl acetate and subsequently transferred to acetonitrile [1/] (Crossley and Lee, 1972).

Tissue	%TRR [3/]	
	EtAc	CH_3CN
Liver	2.9 (0.007)	- (0)
Kidney	4.1 (0.007)	- (0)
Fat [2/]	-	2.0 (0.003)
Muscle	5.0 (0.008)	- (0)

[1/] Selected ethyl acetate extracts were evaporated to dryness and the residue extracted with acetonitrile.
[2/] Ethyl acetate was not used to extract fat samples. The acetonitrile extract was produced by partition with a hexane extract/solution of the fat.
[3/] Figures in brackets are ^{14}C concentrations expressed as methamidophos in mg/kg.

Samples of milk were analysed for methamidophos by gas chromatography. Traces of methamidophos at *ca.* 0.002 mg/kg were detected in milk during the dosing period but none in the recovery period.

Tucker (1974) administered [*S*-methyl-^{14}C]methamidophos to a lactating goat (50 kg bw) at 4 mg/day, after first pre-dosing for 7 days with unlabelled methamidophos (this study is also reported in the acephate evaluation). The dose was equivalent to feeding at approximately 2.1 ppm in the diet and was administered in three separate daily doses for two consecutive days, in the morning, at noon and in the evening. Milk was collected morning and evening. The goat was slaughtered 3 hours after the last dose. Radioactive residues in tissues were extracted with acetonitrile to remove acephate and methamidophos, water to remove conjugates and HCl to release "bound" radioactivity. In addition, the water extracts were incubated with glucoronidase-aryl sulfatase or dilute HCl, to hydrolyze conjugates (Table 3). Methamidophos residues in milk were determined by HPLC.

None of the tissues contained bound or conjugated residues of acephate or methamidophos and TLC did not detect DMPT (*O,S*-dimethyl-*O*-phosphorothioate, ORTHO 18421). During the course of the study, methamidophos residues in milk from the morning milking were all <LOD, while residues in the evening milk reached a maximum of 0.01 mg/kg on day 5 from the start of dosing and were 0.008 mg/kg by day 9. Milk was extracted with hexane, to remove fat, followed by treatment with ethanol to precipitate proteins. The ethanol-soluble fraction was extracted with dichloromethane, followed by methanol. The methanol-insoluble fraction contained lactose. The protein fraction was analysed for individual amino acids. The majority of the ^{14}C in milk was distributed in the protein and lactose fractions (evening milk, day 9: 1% fat, 29% proteins, 47% lactose). Analysis of protein fractions isolated from milk showed that the ^{14}C was distributed amongst the amino acids, indicating that *S*-methyl-^{14}C is released to the metabolic pool and incorporated into natural products. Flushing acidified milk samples with air did not release any ^{14}C carbonate.

Table 3. Distribution of ^{14}C residues in tissues from goats dosed with [*S*-methyl-^{14}C]methamidophos at 2.1 ppm (Tucker 1974).

Tissue	TRR (mg/kg)	Radioactive residue (mg/kg) [1/]			
		Extracted with acetonitrile	Extracted with water	Extracted with 1M HCl	PES [2/]
Liver	0.23	0.014 (6.1%)	0.056 (24%)	0.003 (1.3%)	0.13 (57%)
Kidney	0.097	0.006 (6.2%)	0.023 (24%)	0.02 (21%)	0.048 (49%)
Subcutaneous fat	0.008	0.001 (13%)	0.001 (13%)	<0.001	<0.003
Peritoneal fat	0.014	<0.001	0.001 (7.1%)	<0.001	<0.002
Muscle	0.036	0.007 (19%)	0.008 (22%)	0.001 (2.8%)	0.015 (42%)

[1/] Expressed as methamidophos equivalents.
[2/] PES = post extraction solids.

Methamidophos residues in tissues and milk were below the LOD of the GC and TLC methods used (Table 4) and, together with the extraction results, this suggests incorporation of a significant proportion of the ^{14}C into natural products.

Table 4. Characterization of ^{14}C residues in tissues and milk from goats dosed with [*S*-methyl-^{14}C]methamidophos at 2.1 ppm(Tucker 1974).

Tissue	TRR (mg/kg)	Methamidophos (mg/kg)	
		GC [1/] (RM-12A)	TLC [2/] (acetonitrile extract)
Liver	0.23	<0.005	0.0
Kidney	0.097	<0.005	NA
Subcutaneous fat	0.008	<0.005	NA
Peritoneal fat	0.014	<0.005	NA
Muscle	0.036	<0.005	NA
Milk (morning. day 10)	0.14	<0.005	NA

[1/] Method RM-12A involved extraction with ethyl acetate and analysis by GC/FPD.
[2/] Recovery of methamidophos was *ca*. 50% by the TLC system used.
NA = not analyzed.

In the third and most comprehensive study, Baker and Bautista (1997) orally dosed a lactating goat (38 kg bw, mean 2.7 kg feed/day) with [*S*-methyl-^{14}C]methamidophos by capsule for three consecutive days at 0.7 mg/kg bw/day, equivalent to feeding in the diet at a nominal rate of 11.5 ppm. The daily dose was divided into two, half being administered each morning and evening. Milk was collected twice per day and composited into three samples (0-24 h, 24-48 h and 48 h to sacrifice). The goat was sacrificed 18 hours after the last dose and tissue samples collected for analysis.

Sequential solvent extractions removed approximately 50% of the ^{14}C residue from tissues and 90% from milk. Digestion of the post-extraction solids with α-amylase and/or 6 M HCl solubilized the majority of the remaining ^{14}C residue.

Radioactive residues were highest in liver followed by milk, kidney, muscle and fat. Methamidophos was not detected in tissues and only low levels were detected in milk (Table 5). The majority of the ^{14}C was associated with proteins and amino acids, especially methionine (liver, kidney, muscle), lactose (milk) and triglycerides (fat) (Table 6).

Table 5. Extraction of ^{14}C components and material balance in edible tissues and milk following oral dosing of goats with [*S*-methyl-^{14}C]methamidophos at 11.5 ppm (Baker and Bautista 1997).

Extract	Tissue				
	Liver	Kidney	Muscle	Fat	Milk (48 h to sacrifice)
TRR (mg/kg)	1.7	0.65	0.21	0.033	0.77
CH_3CN/H_2O	NA	0.18 (27%)	0.079 (38%)	NA	NA
Methanol	NA	0.045 (6.9%)	0.008 3.8%)	NA	0.44 (57%)
CH_2Cl_2	NA	0.035 (5.5%)	0.003 (1.6%)	NA	0.079 (10%)
Water	0.009 (0.5%)	0.005 (0.7%)	0.003 (1.7%)	NA	NA
Methanol/water	0.44 (25%)	NA	NA	0.006 (20%)	NA
$CHCl_3$	0.40 (23%)	NA	NA	0.015 (46%)	NA

Extract	Tissue				
	Liver	Kidney	Muscle	Fat	Milk (48 h to sacrifice)
0.1 M HCl	NA	0.009 (1.4%)	0.003 (1.8%)	NA	0.021 (2.7%)
0.1 M NaOH	NA	0.035 (5.3%)	0.008 (4.3%)	NA	0.14 (19%)
α-amylase	0.066 (3.8%)	NA	NA	NA	NA
6 M HCl	0.89 (51%)	0.25 (39%)	0.25 (44%)	NA	0.002 (0.2%)
PES	0.024 (1.4%)	0.001 (0.2%)	0.001 (0.6%)	0.011 (34%)	0.00 (0%)
Accountability	105%	86%	96%	101%	89%

NA = not applicable, solvent system not used to extract the tissue.

Table 6. Characterization of radioactivity in tissues of lactating goats following oral dosing of goats with [*S*-methyl-^{14}C]methamidophos at 11.5 ppm for three consecutive days (Baker and Bautista 1997).

Component	Residue (mg/kg)				
	Liver	Kidney	Muscle	Fat	Milk (48 h to sacrifice)
TRR	1.7	0.65	0.21	0.033	0.77
Methamidophos	<0.002 (<0.11%)	<0.003 (<0.46%)	-	<0.006 (<18%)	0.02 (2.6%)
SMPAA	<0.01 (<0.6)	0.002 (0.3)	-	-	-
DMPT	<0.01 (<0.6%)	0.027 (4.2%)	-	-	-
Glucose/ galactose [1/]	0.25 (15%)	-	-	-	-
Lactose [2/]	-	-	-	-	0.37 (49%)
Phosphatidylcholine [3/]	0.32 (19%)	0.06 (9.2%)	-	-	-
Other phospholipids [4/]	0.058 (3.4%)	0.038 (5.8%)	-	-	-
Choline [5/]	-	0.075 (12%)	-	-	-
Triglycerides [6/]	-	-	-	0.015 (46%)	0.083 (11%)
Proteins/amino acids [7/]	0.89 (51%)	0.25 (39%)	0.091 (44%)	NA	0.12 (16%)

NA = PES not investigated.

[1/] TLC radio-chromatograph, co-chromatography with authentic standards.

[2/] TLC radio-chromatograph, co-chromatography with authentic standards, visualisation with napthoresorcinol and also by derivatisation with phenylhydrazine.

[3/] TLC radio-chromatograph, co-chromatography with an authentic standard and based on saponification and chromatography.

[4/] Tentative assignment based on staining with Dragendorff's reagent in TLC radio-chromatograph, as per phosphatidylcholine.

[5/] Tentative assignment based on staining with Dragendorff's reagent in TLC radio-chromatograph.

[6/] TLC radio-chromatograph, co-chromatography, staining with rhodamine B, HPLC.

[7/] TLC radio-chromatograph of the hydrolysate, visualised by chromogenic reaction with ninhydrin.

Hens

The metabolism of ^{14}C-methamidophos was reported in two studies on laying hens.

In the first study, laying hens (16 birds) were dosed by intubation with a single dose of ^{14}C-methamidophos at 1 mg/kg bw (Ackerman and Wilkes, 1974). The birds were slaughtered in groups of four at 6, 24, 48 and 96 hours after dosing and samples of tissue were collected for analysis. Eggs were collected each day. Body weights of the hens were 1.4-1.8 kg. Levels of radiolabel appearing in the tissues and eggs are shown in Table 7.

Low levels of ^{14}C, <0.05 mg/kg ^{14}C, were observed in eggs collected 0-12 hours after dosing. The average TRR levels in eggs reached a maximum at 72 hours after dosing, at 0.32 mg/kg. Radioactive residues were highest in liver and kidney at all time intervals.

Table 7. Average TRR in tissues and eggs of laying hens at various times after administration of a single dose at 1 mg/kg bw with ^{14}C-methamidophos (Ackerman and Wilkes, 1974).

Tissue/matrix	TRR (mg/kg)			
	6 hours	24 hours	48 hours	96 hours
Visceral fat	0.024	0.027	0.035	0.018
Skin	0.15	0.047	0.045	0.031
Breast muscle	0.19	0.068	0.068	0.058
Thigh muscle	0.18	0.063	0.048	0.045
Gizzard	0.21	0.079	0.079	0.067

Tissue/matrix	TRR (mg/kg)			
	6 hours	24 hours	48 hours	96 hours
Heart	0.26	0.12	0.10	0.083
Kidney	0.70	0.39	0.30	0.20
Liver	1.1	0.60	0.33	0.18

The ^{14}C in the tissues and eggs was further characterized by extraction with methanol (twice), to determine the extractable and non-extractable residues (Ackerman *et al.* 1975a). The percentage of methanol-extractable ^{14}C residue in tissues ranged from 60-85% at 6 hours after dosing, declining to 11-66% in hens slaughtered 96 hours after dosing. From eggs collected 72 hours after dosing, approximately 50% of the ^{14}C was extracted with methanol.

Attempts to characterize the ^{14}C further in liver samples were unsuccessful. However, sequential extraction and hydrolysis of a lyophilised sample of breast muscle, collected 6 hours post-dosing, indicated that the majority of the ^{14}C was associated with polar lipids (79%) and smaller amounts with phosphoproteins (12%), acid-soluble phosphorus compounds (2.7%) and phospholipids (1.8%).

Hatton *et al.* (1997) dosed White Leghorn hens orally with [*S*-methyl-^{14}C]methamidophos by capsule twice daily for three consecutive days at 0.8 mg methamidophos/kg bw/day, equivalent to feeding at 10 ppm in the diet. Eggs were collected daily and separated into composite daily samples of egg white and yolk (0-24 h, 24-48 h, 48 h-sacrifice). Hens were slaughtered at 17-18 hours after the final dose. Liver and egg yolk had the highest ^{14}C residues. In liver, muscle, fat and egg white, solvent-extractable residues that were subsequently liberated by hydrolysis accounted for the majority of the ^{14}C while, in egg yolk, chloroform-soluble residues represented the largest fraction (Table 8).

Table 8. Extraction of radio-labelled components and material balance in edible tissues and milk following oral dosing of laying hens with [S-methyl-^{14}C]methamidophos at 10 ppm (Hatton *et al.* 1997).

Extract	Tissue (mg/kg)				
	Liver	Muscle	Fat	Egg white (48 h-sacrifice)	Egg yolk (48 h-sacrifice)
TRR (mg/kg)	1.0	0.13	0.056	0.4	0.75
Hexane	NA	NA	0.039 (71%)	NA	NA
CH_3CN/H_2O	0.23 (23%)	0.053 (41%)	0.012 (22%)	NA	NA
Methanol	0.21 (21%)	0.005 (3.8%)	0.002 (3.3%)	0.007 (1.6%)	NA
CH_2Cl_2	0.11 (11%)	0.003 (2.4%)	NA	NA	NA
Water	0.005 (0.5%)	0.002 (1.8%)	NA	NA	NA
Methanol/water	NA	NA	NA	0.065 (16%)	0.058 (7.8%)
$CHCl_3$	NA	NA	0.001 (1.4%)	0.027 (6.6%)`	0.44 (58%)
0.1 M HCl	0.009 (0.9%)	0.012 (9.7%)	NA	0.005 (1.1%)	0.006 (0.8%)
0.1 M NaOH	0.19 (19%)	0.006 (5%)	NA	0.001 0.3%)	0.017 (2.3%)
0.1 M KH_2PO_4	0.021 (2.1%)	NA	NA	NA	NA
α-amylase	0.089 (8.8%)	NA	NA	NA	NA
6 M HCl	0.22 (22%)	0.061 (48%)	NA	0.29 (71%)	0.22 (29%)
PES	0.026 (2.6%)	0.003 (2.0%)	0.009 (16%)	0.047 (11%)	0.004 (0.5%)
Accountability	110%	113%	113%	108%	99%

NA = not applicable, solvent system not used to extract tissue.

Methamidophos was a relatively minor component of the TRR, with the majority of the ^{14}C associated with natural products: lipids and proteins and amino acids (Table 9).

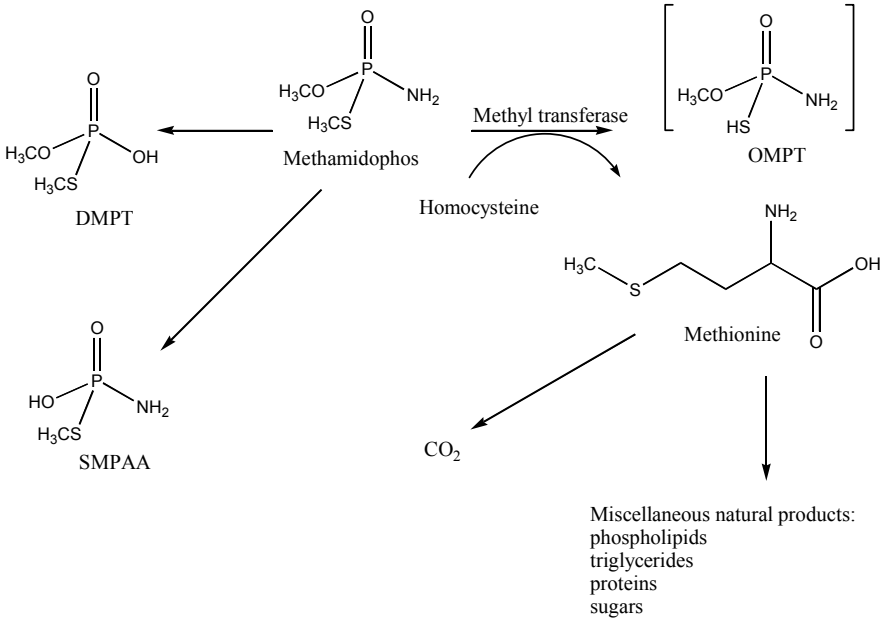
Table 9. Characterization of radioactivity in tissues of laying hens following oral dosing of hens with [*S*-methyl-^{14}C]methamidophos at 10 ppm for three consecutive days (Hatton *et al.* 1997).

Component	Residue (mg/kg)				
	Liver	Muscle	Fat	Egg white (48 h-sacrifice)	Egg yolk (48 h-sacrifice)
TRR	1.0	0.13	0.056	0.4	0.75
Methamidophos	0.007 (0.7%)	-	-	0.025 (6.3%)	0.045 (6.0%)
SMPAA	0.010 (1.0%)	-	<0.001	0.007 (1.8%)	-
DMPT	0.015 (1.5%)	<0.005	<0.001	<0.001	-
Methionine [1/]	0.013 (1.3%)	-	-	-	-
Phosphatidylcholine [2/]	0.19 (19%)	-	-	0.009 (2.3%)	0.24 (32%)
Other lipids [3/]	0.13 (13%)	-	0.034 (61%)	0.018 (4.5%)	0.19 (25%)
Choline [4/]	0.041 (4.1%)	-	-	-	-
Proteins/amino acids [5/]	0.22 (22%)	0.061 (47%)	-	0.29 (73%)	0.22 (29%)
Polar unknowns (origin)	0.099 (9.9%)	0.055 (42%)	0.003 (5.4%)	0.034 (8.5%)	-
Unknowns	0.21 (21%)	-	0.005 (8.9%)	-	-

[1/] TLC radio-chromatograph, co-chromatography with authentic standard, visualized with ninhydrin.
[2/] TLC radio-chromatograph, co-chromatography, visualization using Dragendorff's reagent.
[3/] Tentative assignment based on staining with Dragendorff's reagent in TLC radio-chromatograph, as per phosphatidylcholine.
[4/] Tentative assignment, TLC co-chromatography, visualization using Dragendorff's reagent.
[5/] TLC radio-chromatograph, co-chromatography, visualized with ninhydrin, hydrolysates positive to Bradford's reagent.

In lactating goats and laying hens, methamidophos undergoes hydrolysis to form DMPT and SMPAA.. Additionally, methamidophos may be involved in reactions such as the methyl transfer from the S-methyl moiety, to form methionine, and subsequent transformation to form choline and phospholipids including phosphatidylcholine. Oxidation of small carbon fragments formed by the ester/thioester hydrolysis of methamidophos and/or methionine may lead to the production of CO_2 and incorporation of ^{14}C into natural products such as proteins/amino acids, triglycerides and lactose.

Figure 1. Proposed pathways of methamidophos metabolism by livestock (lactating goats and laying hens).



Plant metabolism

The Meeting received plant metabolism studies of methamidophos in bean, cabbage, tomato, tobacco, sweet potato, potato and lettuce plants or tissue cultures.

White cabbage and tomato plants with 5-7 leaves (glasshouse grown, in soil) were treated with ^{14}C-methamidophos by stem injection and the plants were harvested 7, 14 and 21 days after

application, in the case of cabbages, and 1, 2, 7, 19, 36 and 40 days after application, in the case of tomatoes (Anonymous 1968a). Tissue cultures of sweet potatoes and tobacco were grown for 6 weeks in a medium containing ^{14}C-methamidophos. To obtain enough material for characterization of metabolites, extracts were combined from tomato plants harvested at 19, 36 and 40 days after application. Samples of the four types of plant were analyzed by sequential extraction with ethanol and aqueous ethanol, followed by separation of the ^{14}C in the combined ethanol solutions, firstly by extraction with petroleum ether (lipid fraction) and then by cation-exchange (organic base fraction) and anion-exchange (organic acid fraction) chromatography. Approximately 65-75% of the ^{14}C in cabbage plants was in the lipid fraction, based on associations with TLC spots, and it was postulated to have been incorporated into plant pigments. Although much lower amounts of radioactivity were present in the plant material from tissue cultures of sweet potato and tobacco, it was also associated with plant pigments. DMPT was identified in the organic acid fractions of cabbage and tomato plants. The low levels of radioactivity in the organic base fractions on separation by TLC had retention times coincident with those of amino acids.

Tomato (variety Tropic) and lettuce seedlings (variety White Boston) were treated with 12 applications of a 790 mg/l solution of [*O*-methyl-^{14}C]methamidophos at 14- to 33-day intervals, in the case of tomatoes, and 4 and 7 applications at 16- to 33-day intervals in the case of lettuce (Carazo *et al.* 1984). Most of the radioactivity was extracted from leaves (lettuce, tomato) and tomato fruit with water. TLC of the extract indicated that the majority of the radioactivity was associated with methamidophos. The proportion of ^{14}C that was not extracted was highest for tomato and lettuce stems and roots and may reflect formation of conjugates and/or incorporation of the radioactivity into natural products (Table 10).

Table 10. Distribution of ^{14}C residues in tomato and lettuce plants following application of [*O*-methyl-^{14}C]methamidophos (Carazo *et al*. 1984).

Sample	Number of applications	Days after last application	Aqueous extract (mg/kg)	Methanol soxhlet extract (mg/kg)	PES (mg/kg)	Total (mg/kg)
Tomato						
Fruit	7	8	86	5.7	8.2	4.9
	8	32	71	6.1	21	3.1
	9	29	86	4.6	10	4.3
	10	35	89	5.1	5.1	4.5
	12	40	[1/]	<LOQ	<LOQ	0.07
Fresh leaves	12	40	75	1.0	24	20
Dead leaves	12	40	73	0.01	27	149
Stems	12	40	39	1.5	59	5.4
Roots	12	40	14	8.5	79	1.4
Lettuce						
Leaves	5	1	92	2.3	5.0	13
Stem & roots	5	1	44	5.8	49	1.9
Leaves	3	3	93	2.6	5.1	9.7
Stem & roots	3	3	-	29	71	0.41

[1/] A meaningful calculation of %TRR was not possible due to the low level of radioactivity in fruit, 0.07 mg/kg.

Horler *et al.* (1974) treated individual fruit and leaves of tomato plants (variety V F Napoli) with ^{14}C/^{32}P-labelled methamidophos. Applications were made at fruit set, corresponding to approximately 100 µg or 2-5 ppm on the fruit. Ten fruits or leaves were harvested at weekly intervals, together with adjacent non-treated fruits or leaves. The decline in the total ^{14}C activity measured in fruit and leaves was initially rapid, with a half-life of approximately 7-10 days, but slowed dramatically thereafter, especially in the case of fruit (half-life of *ca.* 6 weeks). Translocation of ^{14}C from treated branches was also studied. At one week after three applications to the leaves of a branch, 57% of the activity was on/in the treated leaves, 2% in untreated fruit and 0% was associated with the rest of the plant. By four weeks after the last application, 23% was associated with the treated leaves, 5% with untreated fruit on the same branch and 2% with the rest of the plant. In 2 week old tomato seedlings grown in the laboratory and treated with ^{32}P-methamidophos, the decline of ^{32}P-labelled material was slow, half-life 14-16 days, with approximately 40% of the radioactivity not recovered in

water washes, in leaf extracts or in leaf residue. It was postulated that, as translocation was apparently relatively unimportant, some loss of radioactivity occurred by volatilization.

Lettuce plots were sprayed with 4 applications of [*S*-methyl-^{14}C]methamidophos at 2.2 kg ai/ha at intervals of 5-7 days and harvested 21 days after the last application (Jalal and Maurer, 1997a). The lettuce material was pulverised and extracted with hexane, acetonitrile and aqueous methanol. The post-extraction solids were subject to a combination of enzymatic and chemical hydrolysis (β-amylase, 0.6 M H_2SO_4, 2 M NaOH) and the released residues were characterized by TLC, HPLC, GC-MS and ^{31}P NMR (Table 11).

The majority of the ^{14}C was extracted by acetonitrile (73%), with smaller amounts extracted by methanol/water (11%) and hexane (0.9%). Post-extraction solids accounted for 16% of the ^{14}C. Methamidophos was the major component of the radioactive residue, accounting for ca. 65% of the TRR.

Table 11. Characterization of radio-labelled residues in lettuce treated with four applications of [*S*-methyl-^{14}C]methamidophos at 2.2 kg ai/ha and harvested 21 days after the last spray (Jalal and Maurer, 1997a).

	Extract/hydrolysate	TRR (%)	Residue (mg/kg)
Extracted residues		85	12
Methamidophos		66	9.1
	Acetonitrile	65	9.0
	Water-MeOH	0.8	0.11
	PES	0.2	0.023
Fructose, glucose & sucrose [1/]		6.1	0.83
	Acetonitrile	2.5	0.34
	Water-MeOH	3.6	0.49
Methanesulfonic acid [2/]		4.9	0.68
	Acetonitrile	4.0	0.56
	Water-MeOH	0.9	0.12
Miscellaneous natural products	Acetonitrile, water-MeOH	4.5	0.61
S-methyl phosphorothiolate conjugate [3/]	Water-MeOH	1.5	0.20
Total lipids [4/] (including di- and tri-glycerides)		1.3	0.19
	Hexane	0.9	0.13
	Water-MeOH	0.4	0.057
Amino acids [5/]	Water-MeOH	0.7	0.10
Unextracted residues (PES)		15	2.1
Starch & other carbohydrates [6/]	β-amylase & acid hydrolysates	7.9	1.1
Base hydrolyzed semi-volatiles		0.2	0.034
Ethyl acetate-soluble base hydrosylate (polyphenols) [7/]		1.3	0.18
Water-soluble base hydrosylate (proteins, peptides etc)		2.1	0.28
Acid-precipitated base hydrosylate (lignin) [8/]		1.8	0.26
Post-hydrolysis solid residue (cellulose)		1.9	0.26
Total		100	14

[1/] Co-chromatography and chromogenic reactions (napthoresorcinol reagent), acetate derivatives.

[2/] ^{31}P and ^{1}H NMR, TLC and HPLC co-chromatography, desorption chemical ionisation mass spectrometry.

[3/] Tentative assignment based on assumptions about retention times and ^{31}P NMR, no standard available, low concentration did not enable further characterization.

[4/] Co-chromatography (TLC, HPLC) and chromogenic reactions (iodine), saponification and TLC co-chromatography, mostly linolenic acid and smaller amounts of linoleic and palmitic acids. It was reported that linolenic acid makes up 55-63% of the total fatty acids in lettuce (Nakatsu *et al*.,. 1984; Nasirullah *et al*., 1984). Linoleic (17-23%) and palmitic (12-13%) acids were also found in considerable amounts.

[5/] Co-chromatography and chromogenic reactions (ninhydrin reagent).

[6/] β-amylase successively removes glucosyl-α-1,4-glucose (maltose) units from soluble oligosaccharides. Acid hydrolysis conditions were strong enough to hydrolyze starch, glycosides and some cell wall polysaccharides but not proteins. TLC co-chromatography of the hydrosylate identified glucose (left over from enzymatic hydrolysis), galactose and xylose. Base hydrolysis conditions were sufficient to hydrolyze, or partially hydrolyze, proteins and cell wall lignin.

[7/] Polyphenols solubilized by base hydrolysis, tentative assignment as extracted with ethyl acetate.

[8/] Tentative assignment, base hydrolysis of PES, lignin precipitated from acidified and concentrated hydrolysate.

Jalal and Maurer (1997b) sprayed potatoes (variety Improved Red LaSoda) with 4 applications of [*S*-methyl-^{14}C]methamidophos at 2.3 kg ai/ha at intervals of 7 days and harvested tubers 14 days after the last application. The potato tubers were pulverised and extracted with hexane, acetonitrile and aqueous methanol. The post-extraction solids were subjected to a combination of enzymatic and chemical hydrolysis conditions (β-amylase, 0.6 M H_2SO_4, 2 M NaOH) and the released residues characterized by TLC, HPLC, GC-MS and ^{31}P NMR (Table 12).

In contrast with lettuce, the majority of the ^{14}C in potato tubers was associated with post-extraction solids (70%). Aqueous methanol, acetonitrile and hexane extracts contained 21, 8.6 and 0.5% of the ^{14}C, respectively. Methamidophos represented <1% of the TRR, with the majority accounted for by natural products, principally starch.

Table 12. Characterization of radio-labelled residues in potato tubers from a crop treated with four foliar applications of [*S*-methyl-^{14}C]methamidophos at 2.3 kg ai/ha and harvested 14 days after the last spray (Jalal and Maurer, 1997b).

	Extract/hydrosylate	TRR (%)	Residue (mg/kg)
Extracted residues		30	2.2
Methamidophos	Acetonitrile	0.2	0.011
Fructose & glucose [1/]		0.6	0.039
	Acetonitrile	0.2	0.011
	Water-MeOH	0.4	0.028
Sucrose [1/]		3.6	0.25
	Acetonitrile	0.8	0.051
	Water-MeOH	2.8	0.20
Methanesulfonic acid [2/]		3.0	0.22
	Acetonitrile	1.6	0.12
	Water-MeOH	1.4	0.10
Miscellaneous natural products [3/]		9.9	0.71
	Acetonitrile	2.4	0.17
	Water-MeOH	7.5	0.54
Total lipids [4/] (including di- and tri-glycerides)		1.4	0.11
	Hexane	0.5	0.035
	Acetonitrile	0.9	0.070
Amino acids [5/]		11	0.77
	Acetonitrile	1.5	0.11
	Water-MeOH	9.2	0.66
Semi-volatiles [6/]	Acetonitrile	1.1	0.077
Unextracted residues (PES)		70	5.0
Starch & other carbohydrates [7/]		66	4.8
	β-amylase hydrosylate	1.6	0.11
	Acid hydrosylate	65	4.7
Proteins, peptides etc, lignin	Base hydrosylate	2.7	0.19
Post-hydrolysis solid residue (cellulose)		0.6	0.044
Total		100	14

[1/] Co-chromatography and chromogenic reactions (iodine/napthoresorcinol reagent), synthesis of peracetate derivatives.

[2/] TLC and HPLC co-chromatography.

[3/] Differential solubility, TLC, HPLC, gel filtration chromatography and TLC chromogenic reactions (iodine, ninhydrin, napthoresorcinol).

[4/] Co-chromatography (TLC, HPLC) and chromogenic reactions (iodine), saponification and TLC co-chromatography mostly linolenic acid, linoleic and oleic acids. It was reported that the major fatty acids in potato tubers are linolenic acid (51-63% of the total fatty acids) (Gailiard, 1972, 1973). Linoleic (13-24%) and palmitic (16-19%) acids were also found in considerable amounts.

[5/] Co-chromatography and chromogenic reactions (ninhydrin reagent).

[6/] Concentration too low for characterization.

[7/] β-amylase successively removes glucosyl-α-1,4-glucose (maltose) units from soluble oligosaccharides. Acid hydrolysis conditions were strong enough to hydrolyse starch, glycosides and some cell wall polysaccharides but not proteins. Note that potato starch (predominantly amylopectin), due to its structure, is unlikely to be amenable to significant amylase hydrolysis (works best with amylose). TLC co-chromatography of the hydrosylate identified glucose (left over from enzymatic hydrolysis), galactose and xylose. Base hydrolysis conditions were sufficient to hydrolyze, or partially hydrolyze, proteins and cell wall lignin.

Tobacco plants (variety Kentucky 9, *ca.* 1.5 m high) were treated with methamidophos in four treatments at one week intervals, at an application rate of 1.1 kg ai/ha (Shaw and Parker 1983). Plants grown in the laboratory were treated with [*S*-methyl-^{14}C]methamidophos, while those grown in the field were treated with unlabelled methamidophos. In each treatment, sixteen leaves on each of paired tobacco plants were treated, with the plants sampled at 0, 7, 21 and 35 days after the last application, four leaves per pair of plants. Data presented on cured tobacco leaves and smoke will not be discussed here. Methamidophos residues were determined by GC (Table 13). The half-life for methamidophos residues in field and laboratory grown tobacco was *ca.* 5 and 15 days respectively.

Table 13. Decline in TRR and methamidophos residue in leaves with time after the last of four applications of methamidophos at 1.1 kg ai/ha to tobacco plants (Shaw and Parker 1983).

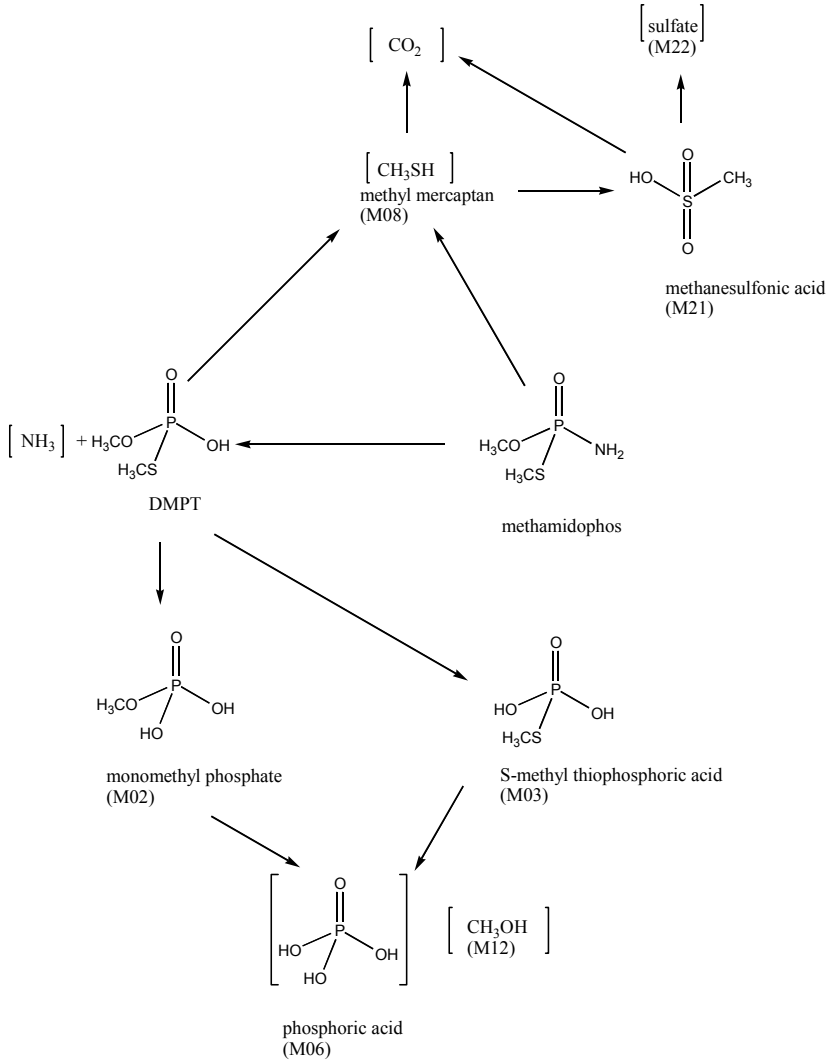
Days after last application	Laboratory		Field
	TRR (mg/kg)	Methamidophos (mg/kg)	Methamidophos (mg/kg)
0	158	137	92
7	426	319	34
21	182	180	6
35	91	71	3

Tutass (1968a) treated the roots and leaves of soil-grown kidney bean, cabbage and tomato seedlings. The root treatment was performed by washing the soil from the roots and supporting the plants in jars, with only the roots immersed, containing ^{14}C-methamidophos dissolved in Hoagland solution.

Autoradiograms used to follow the uptake of ^{14}C by tomato plants with roots immersed in ^{14}C-methamidophos solution for 6 hours indicated that uptake was essentially independent of pH (pH range 3.5 to 7.5). When the ^{14}C-methamidophos was applied directly to the soil, at 1 hour after treatment the petioles were slightly labelled while at two hours after application to the soil the radioactivity had reached the leaves. The plants were initially wilted and required one hour to regain turgor. The movement of ^{14}C suggested that it moved through the xylem. When applied to the leaves of bean, cabbage and tomato plants, the ^{14}C moved towards the margins of the leaves in a pattern suggestive of apoplastic transport. TLC analysis of extracts from bean leaves demonstrated that the ^{14}C at the margins was essentially methamidophos while that at the centre of the leaf was mostly due to more polar compounds.

In plants, methamidophos undergoes hydrolysis of the thioester (–S-CH_3 moiety) which is eventually oxidized to methanesulfonic acid. Further transformation of methanesulfonic acid results in incorporation of the carbon into various natural products such as sugars, starch, lipids and proteins/amino acids. The formation of the putative *S*-methyl phosphorothioate conjugate is expected to result from hydrolysis at the amino group of methamidophos to produce DMPT, which may be subject to further hydrolysis of the ester/thioester. The majority of radioactivity in potato tubers was present in starch, presumably from the incorporation of methamidophos-derived carbon fragments into sugars and transport of them to the tubers.

Figure 2. Proposed pathways of methamidophos metabolism in plants.



Environmental fate in soil

The Meeting received information on the behaviour and fate of methamidophos during soil and solution photolysis, aerobic soil metabolism and field dissipation. Information on the soil adsorption properties of methamidophos was also provided.

Based on the lack of absorption in the UV spectrum of methamidophos above 243 nm, direct photolysis in aqueous media is not expected to occur (Hellpointer 1993). However, indirect photolysis mechanisms may result in some degradation on irradiation.

Photo-degradation on soil

Chopade and Freeseman (1985) irradiated glass slides with [*S*-methyl-^{14}C]methamidophos applied at *ca.* 35 mg/kg to thin layers of sandy loam soil (56% sand, 36% silt, 5.0% clay, pH 5.0, %OM 2.6, CEC 17 meq/100 g, particle density 2.6 g/cm^3) at 33°C for 87 hours. The light source used was a 450 W medium-pressure Hg lamp, filtered through borosilicate glass. Methamidophos degraded with a half-life of *ca.* 63 hours (corrected for dark-control) (Table 14). The major degradation products formed were SMPAA (*S*-methyl phosphoramidothioate = M05, increasing to 24% of the applied ^{14}C by 87 hours) and DMPT (desamino-methamidophos = M01, max 6% of the applied ^{14}C). Unextracted residues increased during irradiation and one-third of the applied radioactivity had volatilised following 87 hours of irradiation. However, there was only a small difference between the losses by volatilization from the dark controls and irradiated samples. The majority of the volatiles trapped were identified by HPLC as dimethyl disulfide (M10), speculated to be formed as a dimer of methyl

mercaptan (M08) and accounting for *ca.* 50% of the volatiles, the remainder was accounted for by methamidophos, SMPAA and DMPT, together with two unknowns.

Table 14. Distribution of ^{14}C in irradiated thin films of soil (Chopade and Freeseman, 1985) [1/].

Hours of irradiation	Extracted				Unextracted	Volatiles
	Total extractable	Methamidophos	DMPT	SMPAA		
0	99 (99)	99 (99)	ND	ND	1 (1)	ND (ND)
24	78 (92)	64 (91)	4 (<1)	10 (<1)	8 (3)	14 (5)
29	72	57	4	11	10	18
45	68 (82)	47 (80)	6 (1)	15 (<1)	11 (5)	21 (13)
63	61 (75)	35 (72)	6 (2)	20 (<1)	14 (7)	25 (18)
87	50 (65)	23 (63)	3 (1)	24 (<1)	17 (9)	33 (26)

[1/] Values in brackets are for the dark control, i.e. non-irradiated samples.
ND = not detected.

Ridlen (1989) irradiated thin layers of a sandy loam soil (56% sand, 30% silt, 14% clay, pH 6.6, %OM 1.1, CEC 10 meq/100 g, particle density 2.6 g/cm^3), treated earlier with [*S*-methyl-^{14}C]methamidophos. The light source was a 450 W medium-pressure mercury lamp, filtered through borosilicate glass (Table 15).

Table 15. Distribution of applied radioactivity after treatment of a soil with 30 mg/kg [*S*-methyl-^{14}C]methamidophos (% applied radioactivity).

Time	Extracted	Methamidophos	Degradates	Unextracted	Volatiles	Total
0	100	100	<1	<1	-	100
115 hours (irradiated)	77	30	47	4	10	91
115 hours (dark control)	93	93	<1	1	6	100

Photo-degradation in solution

Chopade (1985a) reported that [*S*-methyl-^{14}C]methamidophos photodegraded slowly in sterile buffer solutions at pH 5, under both artificial and natural light. Following 5 days of continuous irradiation under a mercury lamp at 33 °C, methamidophos was present at 89% of the initial ^{14}C concentration. Degradation products found were SMPAA (*S*-methyl phosphoramidothioate, M05) and DMPT (desamino-methamidophos, M01), present at 3 and 6%, respectively, of the initial ^{14}C concentration. In the dark controls, 93% remained unchanged and SMPAA, DMPT and dimethyl disulfide accounted for <1, 3 and 2%, respectively, of the initial ^{14}C concentration. Similar results were obtained from solutions exposed to natural light, with 78% of the applied methamidophos remaining after 30 days exposure (August to September in Kansas, temperature 9 - 42°C). SMPAA accounted for 7%, and DMPT 13%, of the initial ^{14}C concentration. In the dark controls, 87% of the ^{14}C remained as unchanged methamidophos after 30 days, with SMPAA, DMPT and dimethyl disulfide accounting for <1, 6 and 6 %, respectively, of the initial ^{14}C concentration.

Aerobic metabolism

Half-lives of aerobic soil degradation of methamidophos were generally ≤ 6 days. The principal mechanism of degradation appears to be microbial metabolism. Observed degradation products included SMPAA and DMPT and, together with methamidophos, these intermediates are further degraded to CO_2.

Leary and Tutass (1968) treated samples of Iowa silt soil with [*S*-methyl-^{14}C]methamidophos and incubated them at 21 and 37 °C for 64 hours. Soxhlet extraction of the samples with acetone extracted 40 and 15% of the applied ^{14}C, from the samples incubated at 21 and 37°C, respectively. The non-extracted radioactivity accounted for 20 and 23% of the applied ^{14}C. Analysis of the extracted ^{14}C by anion- and cation-exchange chromatography and TLC identified DMPT (desamino-methamidophos, M01) as well as minor amounts of methamidophos, amino acids (Rf values and chromogenic reaction with ninhydrin) and carbohydrates. In a separate experiment, the distribution of radioactivity in Iowa silt soil following three days of incubation under aerobic and anaerobic conditions was studied. Under aerobic conditions, approximately 70% of the applied dose was

recovered from traps for volatiles, with 29% remaining in the soil, while under anaerobic conditions 7.6% was associated with volatiles and 85% remained in the soil.

Möllhoff (1974, revised 1991) determined the rate of degradation of methamidophos in two standard soils at 20 °C. Rapid degradation occurred in both soil systems, with estimated half lives (1st order kinetics) of 2.1 and 3.4 days.

Panthani (1989a) treated a sandy soil (sand 96%, silt 6%, clay 2%, %OM 1.9, pH 6.7, CEC 2.9 meq/100 g, bulk density 1.5 g/cm^3) with [*S*-methyl-^{14}C]methamidophos to give a final concentration of 9 mg/kg. After treatment, the soil moisture content was approximately 75% of field capacity. The soil was maintained at 25°C under a flow of CO_2-free air that was passed through the soil and into methanol and NaOH traps, to collect evolved volatiles. Soil samples were analyzed at 0, 1, 2, 4 and 10 days by combustion/LSC; extracts were also analyzed by TLC and HPLC (Table 16). Methamidophos was rapidly degraded with a half-life of approximately 6 days.

Table 16. Characterization of ^{14}C in the soil (% applied radioactivity) (Panthani 1989a).

Incubation period (days)	Soil					MeOH trap	NaOH trap [1/]	Total
	Extracted				Unextracted			
	Total	Methamidophos	DMPT	SMPAA				
0	99	97	1.1	-	<1	-	-	100
1	95	85	5.8	5		0.26	0.52	101
2	93	86	3.7		9.2	0.85	1.4	104
4	75	57	9.7		10	3.5	9.0	97
10	43	8.4	5.5	31	21	28	5.1	97

[1/] Approximately 88, 77, 66 and 43% of the ^{14}C in the NaOH traps on days 1, 2, 4 and 10 was as $^{14}CO_2$.

Panthani (1989b) treated a sandy loam (sand 56%, silt 32%, clay 12%, %OM 0.9, pH 7.5, CEC 8.1 meq/100 g, field capacity at 0.3 bar 10%, bulk density 1.4 g/cm^3) with [*S*-methyl-^{14}C]methamidophos to give a final concentration of 6.5 mg/kg. After treatment, the soil moisture content was approximately 75% of field capacity. The soil was maintained in the dark at 25°C for 5 days. Evolved CO_2 and volatile compounds were collected in methanol and NaOH traps. Soil samples were analyzed by combustion/LSC with extracts also analyzed by TLC and HPLC (Table 17). Methamidophos was rapidly degraded, with a half-life of approximately 14 hours.

Based on TLC analysis of acetonitrile extracts of the soil, the parent compound represented 93% (6.0 mg/kg) of the applied radioactivity at 0 days, decreasing to 71% (4.7 mg/kg) in 6 hours, to 1% (0.06 mg/kg) in 2 days and was less than the limit of quantification by 5 days. The major degradation product was radio-labelled $^{14}CO_2$, which accounted for 49% of the applied radioactivity at 5 days. The major non-volatile degradation product, SMPAA (*O*-desmethyl methamidophos), represented 1% (0.06 mg/kg) of the applied radioactivity at 0 days, increasing to a maximum concentration of 27% at 1 day post-treatment but then decreased to 11% (0.72 mg/kg) by 2 days and was not detected at 5 days. Volatile organic compounds accounted for a maximum of 6% of the applied radioactivity at 2 days. Characterization of the volatile residues, other than CO_2, by GC-FPD analysis detected methyl mercaptan, dimethyl sulfide and dimethyl disulfide. Unextracted ^{14}C reached 31% of the applied radioactivity at 5 days.

Table 17. Characterization of ^{14}C in the soil (% applied radioactivity) (Panthani 1989b).

Incubation period (days)	Soil					CO_2	Volatile organics	Total
	Extracted				Un-extracted			
	Total	Methamidophos	SMPAA	others				
0	99	92	0.92	4.6	1.7	-	-	100
0.25	83	72	6.5	5.1	9.5	1.8	1.4	95
1	59	27	27	4.6	20	7.2	5.4	91
2	15	0.92	11	3.8	30	31	6.3	83
5	2.6	0	0	2.1	31	49	5.1	87

Stupp (2002a, 2002b) studied the rate of aerobic degradation of methamidophos and SMPAA (*O*-desmethyl methamidophos, M05) in three soils: Laacherhof A III (silt loam: sand 37%, silt 51%, clay 12%, pH_{CaCl2} 6.5, %OM 1.4, CEC 8 meq/100 g, bulk density 2.6 g/cm^3), Höfchen am Hohenseh

4a (silt: sand 8.5%, silt 85%, clay 10%, pH_{CaCl2} 6.7, %OM 3.6, CEC 15 meq/100 g, bulk density 2.1 g/cm^3) and Laacherhof AXXa (sandy loam: sand 74%, silt 23%, clay 5%, pH_{CaCl2} 6.3, %OM 1.8, CEC 8 meq/100 g, bulk density 2.5 g/cm^3). The initial application rate in the experiment on methamidophos was 0.84 mg/kg soil DW, while that for the experiment on SMPAA was 0.925 mg/kg soil DW basis. In both experiments, samples were maintained under aerobic conditions in the dark at 20 °C and at 50% maximum water holding capacity for periods of up to 30 hours. At intervals, samples were extracted with water/acetonitrile/formic acid (800/200/1) and the extracts were analyzed by LC-MS/MS (Table 18). The rate of degradation of both compounds was rapid, with residues declining from 79-86% and 86-91% of the application rate, for methamidophos and SMPAA respectively, at 0 hours to be <LOD after 24 hours incubation. The rate of degradation appeared to correlate with soil microbial activity.

Table 18. Degradation rate of methamidophos in several soils (Stupp, 2002a, 2002b).

Soil classification	Silt loam	Sandy loam	Silt
Methamidophos			
K (h^{-1})	0.221	0.252	0.381
t½ (DT_{50}) days	3.1	2.8	1.8
Initial soil biomass/ soil microbial activity (mg C microbial/kg soil DW)	275	301	800
SMPAA			
K (h^{-1})	0.143	0.208	0.368
t½ (DT_{50}) days	4.9	3.3	1.9
Initial soil biomass/ soil microbial activity (mg C microbial/kg soil DW)	275	301	800

Anaerobic metabolism

Panthani (1989c) treated a sandy loam (sand 56%, silt 32%, clay 12%, %OM 0.9, pH 7.5, CEC 8.1 meq/100 g, field capacity at 0.3 bar 10%, bulk density 1.4 g/cm^3) with [*S*-methyl-^{14}C]methamidophos, to give a final concentration of 6.5 mg/kg. After treatment, the soil moisture content was approximately 75% of field capacity. The soil was maintained in the dark at 25°C under an oxygen atmosphere for 14 hours, after which the system was converted to an anaerobic one by flushing with nitrogen. Evolved CO_2 and volatile compounds were collected in methanol and NaOH traps. Soil samples were analyzed by combustion/LSC and extracts were analyzed by TLC and HPLC (Table 19).

Based on TLC analysis of the soil extracts, the parent compound was present at 97% (6.3 mg/kg) of the applied radioactivity at 0 days, decreasing to 41% (2.6 mg/kg) at 14 hours, 0.3% (0.02 mg/kg) at 16 days and was less than the limit of quantification at 61 days. The major degradation product was SMPAA, which accounted for 35% of the applied radioactivity at 16 days post-treatment. Volatile organic compounds accounted for a maximum of 15% of the applied radioactivity at 31 days; GC-FPD analysis detected methyl mercaptan, dimethyl sulfide, and dimethyl disulfide. Unextracted ^{14}C increased to a maximum of 22% of the applied radioactivity at 61 days.

Table 19. Characterization of ^{14}C in the soil (% applied radioactivity) (Panthani 1989c).

Incubation period (days)	Soil					CO_2	Volatile organics	Total
	Extracted				Un-extracted			
	Total	Methamidophos	SMPAA	others				
0	99	97	0	2	1	0	0	100
0.58	66	41	22	6	15	3	4	87
16	37	0	35	1	19	13	11	80
31	37	0	35	1	19	8	15	79
61	36	0	34	2	22	7	13	78

Mobility

Methamidophos and DMPT are only weakly adsorbed by soils and can be classified as mobile. The rate of degradation, both in the laboratory and in the field, is such that residues are not expected to persist at detectable levels for more than a few days. Methamidophos is not persistent.

In a study of the mobility of ^{14}C-methamidophos on TLC plates prepared with six soils, ranging from sand to silty clay, there was little adsorption of methamidophos, as demonstrated by the Rf values of 0.92-0.98 (Thornton *et al.* 1976).

Flint and Shaw (1972) studied the adsorption of [S-methyl-^{14}C]-methamidophos on a loam (40% sand, 42% silt, 18% clay, pH 7.7, %OM 1.4, bulk density 1.4 g/cm^3), a silty clay loam (8% sand, 54% silt, 38% clay, pH 6.3, %OM 2.0, bulk density 1.3 g/cm^3) and a "high organic content" silty clay loam (6% sand, 54% silt, 40% clay, pH 6.1, %OM 4.4, bulk density 1.4 g/cm^3), using batch equilibrium studies. Methamidophos was only weakly adsorbed by the soils incubated at 15 °C, such that it was not possible to determine a Freundlich adsorption coefficient for the loam soil and only two data points were available to determine the K_{ads} for silty clay loam soil. The calculated values of K_{ads} and n were 0.05 and 1.5, respectively, for the silty clay loam and 0.12 and 5.9, respectively, for the "high organic content" silty clay loam. The adsorption was too low at 30 °C to determine any K_{ads} values.

The same soils as above were used to determine that the optimum time for equilibration at 15°C for the adsorption studies was 2 to 4 hours (Shaw 1979). The "high organic content" silty clay loam was the only soil to adsorb methamidophos, though only slightly, and was used to determine 1/n and K_{ads} values of 1.28 and 0.12, respectively, by the Freundlich equation.

Batch equilibrium studies using [*S*-methyl-^{14}C]acephate, [*O*-methyl-^{14}C]methamidophos and [*S*-methyl-^{14}C]*O,S*-dimethyl phosphorothioate (DMPT) were conducted using five soils that ranged in texture from sand to clay loam (Pack and Verrips, 1988). In four of the soils, acephate, methamidophos and DMPT were not adsorbed sufficiently to permit the calculation of Freundlich adsorption coefficients (Freundlich K_{ads}). For the clay loam soil (sand 38%, silt 30%, clay 32%, %OM 3.3, CEC 20 meq/100g, pH 5.8 density 1.3 g/cm^3), the reported adsorption values for parent acephate and its degradation products are listed in the following table:

Acephate			Methamidophos			DMPT		
K_{ads} (ml/g)	1/n	r^2	K_{ads} (ml/g)	1/n	r^2	K_{ads} (ml/g)	1/n	r^2
0.090	1.06	0.96	0.029	0.64	0.93	0.030	0.69	0.92

Calculated K_{oc}s for acephate, methamidophos, and DMPT in this clay loam soil were 4.7, 1.5, and 1.6 ml/g, respectively. The minimal adsorption of acephate, methamidophos and DMPT precluded the determination of desorption values.

The dissipation and mobility of methamidophos was studied in two potato fields in Chualar and Fresno California, USA (Grace and Cain, 1990). Post-emergence applications at 1.1 kg ai/ha were made to two plots at each site: a single application to the first and six applications at approximately 7-day intervals to the second. The surface soil, 0-0.9 m, was characterized as loam sand, sand and sandy loam for the Chualar site and sandy loam/loamy sand for the Fresno site. Residues of methamidophos (no metabolites) were monitored in soil cores to a maximum of 122 cm depth, each core divided into 15 cm depths, for 14 days after the last application. Rainfall and irrigation during the study ranged from 5.8-16 cm. The only soil cores in which residues were detected were the 0-15 cm cores and only on day 0; the exception being the Chualar multiple application plot, in which low levels of methamidophos were detected on day 3 after the last spray.

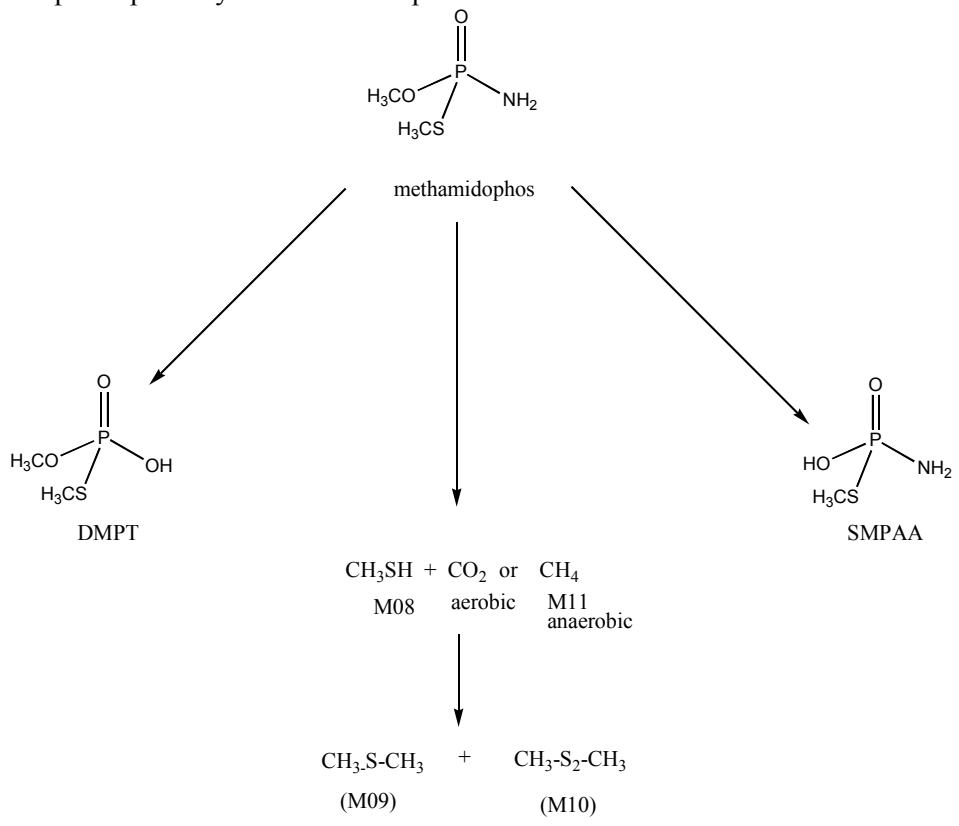
Babczinski and Sommer (2002) studied the leaching behaviour of methamidophos and its major metabolite SMPAA (*S*-methyl-phosphoramidothioate, M05) in three different soils: a sandy loam (72% sand, 23% silt, 5.0% clay, pH 7.2, %OM 1.8, CEC 8 meq/100 g, microbial biomass 352 mg microbial C/kg, bulk density 2.5 g/cm^3), a silt loam (37% sand, 51% silt, 12% clay, pH 7.4, %OM 1.4, CEC 8 meq/100 g, microbial biomass 301 mg microbial C/kg, bulk density 2.6 g/cm^3) and a silt (8.5% sand, 81% silt, 10% clay, pH 7.6, %OM 3.6, CEC 15 meq/100 g, microbial biomass 707 mg microbial C/kg, bulk density 2.1 g/cm^3). Duplicate soil columns, with methamidophos and M05 applied at rates equivalent to 1.2 kg methamidophos/ha, were leached with 1 litre of 0.01 M $CaCl_2$ solution over a period of 5 days (equivalent to 51 cm rainfall) and the mobility of the compounds through the soil monitored by LC-MS/MS of the leachate fractions. The degradation of both methamidophos and SMPAA on the non-sterile soil columns was too fast to permit calculation of K_d

or K_{oc} values. Repeating the experiment with sterile columns permitted calculation of K_{oc} values. The calculated K_{oc} for methamidophos was 8 for all three soils. In the case of SMPAA, K_{oc} values of 7 and 13 were obtained for the sandy loam and silt loam soils. Matrix interferences prevented the determination of a K_{oc} value for the silt soil.

Obrist (1979) studied the soil-column leaching of [S-methyl-^{14}C]methamidophos in Kansas sandy loam (sand 58%, silt 32%, clay 10%, %OM 2.8, pH 5.1, CEC 15, bulk density 1.3 g/cm^3, particle density 2.4 g/cm^3). A solution of ^{14}C-methamidophos was applied to the soil to give a final concentration of 10 mg methamidophos/kg soil and the residue was aged for 30 days before the treated soil was applied to the surface of 30 cm untreated soil columns. Characterization of the aged residue in the treated soil indicated that only 21% of the applied radioactivity remained at 30 days, of which less than 2% could be extracted by refluxing with dichloromethane/methanol (7:3). The solvent-extracted residue was identified by TLC as compounds more polar than methamidophos. The unextracted soil radioactivity (20% of the applied dose) was associated with fulvic acid (47%), humic acid (22%) and humin (31%). Following application of soil containing aged residues to the tops of columns of untreated soil, the columns were eluted with oxygen-saturated rainwater equivalent to approximately 12.5 mm rainfall, each day for 45 days. Only 5.4% of the ^{14}C was recovered in the leachate, the majority of it remaining associated with the soil depth 0-1.25 cm, mostly comprising the treated soil. No ^{14}C was detected in soil column sections below 7.5 cm in depth. Approximately 10% of the ^{14}C was lost by volatilization.

Tutass (1968b) studied the soil column leaching of methamidophos in three soils (Mount Holly muck, Fresno sandy loam and Moorestown loam). Little of the compound was retained by the soils. The mean total percentage recovery of the applied methamidophos was low for all three soils, being in the range 9.5-25%, which was probably a result of microbial decomposition of methamidophos.

Figure 3. Proposed pathways of methamidophos metabolism in soil.



Environmental fate in water-sediment systems

The Meeting received information on the behaviour of methamidophos during aqueous sterile hydrolysis and on the fate of methamidophos in water-sediment systems.

Aqueous hydrolysis

Methamidophos is stable to hydrolysis at low pH but is readily degraded at high pH and thus the rate of hydrolysis is pH-dependent. Chopade (1985a) studied the hydrolysis of methamidophos in sterile aqueous buffered solutions at 12 mg/l, incubated at 25 °C in the dark. Methamidophos was stable at pH 5 (<9% degraded after 30 days incubation) but significant degradation occurred at pH 7 and 9, with calculated hydrolysis half-lives of 27 and 3.2 days, respectively. The predominant degradation product after 30 days at pH 7 was dimethyl disulfide (M10, 41%) while at pH 9 after 7 days, both dimethyl disulfide (M10, 26%) and SMPAA (*O*-desmethylmethamidophos = M05, 51%) were formed. Small amounts (≤1%) of DMPT were detected at all pHs.

Aerobic sediment/water

The aerobic degradation and metabolism of methamidophos was studied in two water/sediment systems (Brumhard *et al.,* 1995). The sediments, collected from a drainage ditch close to an orchard (Ijzendoorn, NL) and from a fish pond (Lienden, NL), were classified as loamy silt (Ijzendoorn) and loamy sand (Lienden), with organic carbon contents of 3.8 and 0.42%, respectively, and a pH (in $CaCl_2$) of 7.2 in both cases. Test vessels, with [*S*-methyl-^{14}C]methamidophos applied at 3.7 mg/l, were incubated in the dark under aerobic conditions at 22 °C for 60 days (Table 20).

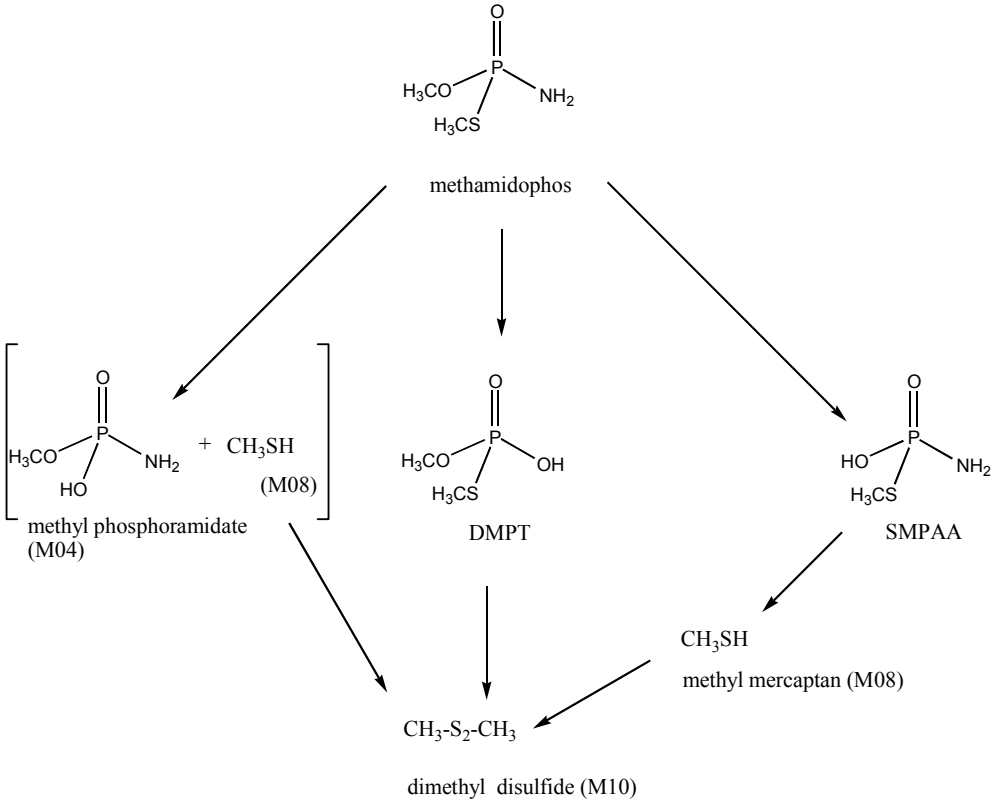
Table 20. Distribution of ^{14}C from [*S*-methyl-^{14}C]methamidophos following incubation with Ijzendoorn and Leinden water/sediment systems (% applied ^{14}C) (Brumhard *et al.,* 1995).

Incubation period (days)	$^{14}CO_2$ [1/]	Other volatiles	H_2O [2/]	Extracted from sediment	Un-extracted in sediment	Total recovery
Ijzendoorn						
0	ND	ND	ND	6.2	1.0	-
7	20	1.5	35	8.7	23	89
14	42	2.8	9.2	4.4	25	83
32	60	2.5	0.9	2.0	30	96
60	66	3.4	0.4	1.8	23	95
Leinden						
0	ND	ND	ND	2.0	0.6	-
7	8.2	1.7	68	3.3	11	93
14	27	4.3	33	2.6	17	86
32	56	3.8	5.2	1.3	20	89
60	66	5.5	3.3	1.0	16	93

[1/] Radioactivity collected in CO_2 traps plus $^{14}CO_2$ in the aqueous phase.
[2/] Radioactivity in the aqueous phase (minus $^{14}CO_2$ in the aqueous phase).

Although metabolites other than CO_2 were not characterized, no individual metabolite comprised more than 10% of the total radioactive residue at any time point sampled. The half-life for degradation of methamidophos in the two water-sediment systems was estimated to be 4 and 6 days, respectively, for the Ijzendoorn and Lienden sediments.

Figure 4. Proposed pathways for fate of methamidophos in water-sediment systems.



METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received information on methods for analysis of methamidophos residues in crops and animal tissues. Typically, crops and animal commodities were extracted with ethyl acetate/sodium sulfate, with clean-up using a silica gel column. Dry crops required the addition of water prior to extraction. Oily crops required an initial clean-up by partitioning between acetonitrile and hexane, prior to the silica gel column. Detection was by GC, using a thermionic detector.

There was good agreement between the results obtained using radiometric methods in metabolism studies for methamidophos in lettuce and potato and the same samples analyzed using method RM-12A-9 (Jalal and Maurer, 1997a, 1997b) (Table 21).

Table 21. Comparison of residues of methamidophos determined by method RM-12A-9 and the radiometric results (Jalal and Maurer, 1997a, 1997b).

Crop	Residue (mg/kg)	
	GC (RM-12A-9)	Radiometric results
Lettuce	8.3 [1/]	9.1
Potato	0.011 [2/]	0.011

[1/] Not corrected for analytical recovery of 87% from a sample fortified at 10 ppm.
[2/] Not corrected for analytical recovery of 98% from a sample fortified at 0.02 ppm.

Methods used in residues trials

Table 22 summarizes the various methods used in the residue trials. They were all similar, with small modifications in extraction solvent, clean-up steps and GC detection method.

Table 22. Summary of major analytical methods used for the determination of methamidophos in various matrices.

Method, (reference)	Matrix	Extraction and clean-up	Detection	LOQ (mg/kg)
RM-10 (Anonymous 1968b, 1973a)	Crops	Extraction with ethyl acetate (dried with anhydrous Na_2SO_4). Clean-up on a silica gel column.	GC, thermionic detector	0.05
Bayer method no. 00137 (Leary, 1971)	Potato, tomato, sugar beet	Extraction depended on crop type. Water was added to dry crops prior to extraction with ethyl acetate. Oily crops were extracted with acetonitrile followed by a hexane partition. Extracts cleaned-up on a silica gel column.	GC, thermionic detector	0.05
Bayer method no. 00137/M013 (Anonymous, 1973b)	Crops	Extraction depended on crop type. Dry crops were extracted twice with ethyl acetate. Oily crops and oil were extracted with acetonitrile followed by a hexane partition step and silica gel clean up step omitted.	GC, thermionic detector	0.1
Bayer method no. 00137/M001 (Ohs, 1987)	Crops, soil, animal tissues	As per Leary (1971).	GC using a megabore column and FPD	0.01-0.05
Leary, 1974	Crops	Extraction depended on crop type. Dry crops were extracted with ethyl acetate (anhydrous Na_2SO_4). For oily crops, acetonitrile was used followed by hexane partition. Clean-up was by a silica gel column.	GC, thermionic detector	0.01
RM-12A and modifications (RM-12A-4, Anonymous, 1974a)	Crops (oily, dry), soil, tissues	Extraction depended on the matrix. Dry crops, brain and liver tissues were extracted with ethyl acetate (anhydrous Na_2SO_4). Oily crops, heart, kidney, muscle and fat were extracted with acetonitrile followed by hexane partition, after addition of water. Soil was extracted with water-saturated ethyl acetate. Clean-up of extracts was by silica gel column.	GC, thermionic detector	
(RM-12A-5a Slagowski and Leary, 1979) (RM-12A-7a Slagowski and Leary, 1982)	Crops (oily, dry), oil, soil, tissues, cotton lint)	Extraction step depended on the matrix. Crops/dry crops were extracted with ethyl acetate (anhydrous Na_2SO_4), adding a small quantity of water to dry crops. Oily crops and oils were extracted with acetonitrile after addition of water, followed by a hexane partition. Cotton lint was extracted with acetone after first wetting with water. Cleaned-up extracts using gel permeation chromatography.	GC, thermionic detector	0.01
Bayer method 00219 (McNamara and Stanley, 1975)	Processed tomato and sugar beet	Samples were extracted with chloroform/methanol (1:1), followed by partitioning with either acetonitrile/hexane or benzene/water. Clean-up on a silica gel column.	GC, thermionic detector	0.01
00219/M001 (Anonymous, 1982a)	Tomato	As above but extraction solvent was ethyl acetate instead of chloroform/methanol.	GC, thermionic detector	0.01
Luke *et al,*. 1975 JAOAC	Crops	Extracted residues with acetone, followed by a with dichloromethane/petroleum ether partition step. Repeated the partitioning of the water phase (after adding NaCl) with dichloromethane.	GC, thermionic detector	0.5
Bayer method 00155 (Möllhoff, 1971)	Crops	Extraction depended on crop type. Wet crops were extracted with acetone ($NaCO_3$). Cotton seed was extracted with acetone/water (2:1) and, for soil, acetone/water [1/]. Clean-up by partition of the aqueous extract with hexane (discarded), addition of NaCl and partition with chloroform then chloroform/acetone (9:1 v/v). Chloroform was removed and extracts redissolved in acetone.	GC FPD, P-mode	0.01 (0.05 for cottonseed)
DFG Method 365; Bayer method 00156 (Möllhoff, 1976)	Crops	Wet crops extracted with acetone, followed by a partition step with dichloromethane after NaCl addition. For dry crops, used acetone/water. In both cases, transferred the residue to dichloromethane after NaCl addition. Clean-up on a silica gel column.	GC, thermionic detector	0.01

Method, (reference)	Matrix	Extraction and clean-up	Detection	LOQ (mg/kg)
DFG multi-residue method S-19; Bayer method 00086 (Specht and Their, 1989)	Crops	Residues extracted with acetone/water, 2:1, taking into account the water content of the plant matrix, and transferred to dichloromethane after NaCl addition. Clean-up using gel permeation chromatography, eluting with cyclohexane and ethyl acetate. Additional clean-up with a silica gel column was required if using GC-ECD	GC-NPD or GC-ECD or GC-FPD	0.01
Spanish multi-residue method IA (Anonymous, 1995)	Crops	Residues extracted with ethyl acetate (anhydrous Na_2SO_4). Clean-up by gel permeation chromatography, elution with cyclohexane and ethyl acetate	GC-thermionic, GC-FPD	0.01
MET-95-01, Anding, 1995; Anonymous, 1988a	Crops	Residues extracted with ethyl acetate (anhydrous Na_2SO_4).	GC-FPD	0.01
Bayer method no. 00350 (Blaß and Philipowski, 1995)	Crops	Wet crops were extracted with acetone. For dry crops, acetone/water was used. Clean-up on a diatomaceous earth column washed with *n*-hexane, eluted with ethyl acetate/ethanol, followed by a silica gel column eluted with dichloromethane/acetone.	GC-FPD (megabore column)	0.01
Tucker (1973) based on RM-12-2	Bovine tissues, milk	Different extraction steps were used for milk (ethyl acetate); kidney, muscle and fat (acetonitrile after wetting with water, Na_2SO_4 added, and hexane partition); liver (ethyl acetate and Na_2SO_4 followed by acetonitrile/hexane partition).	GC (detector not reported)	0.01 (tissues) 0.005 (milk)
Method 31093 (Stanley, 1971)	Bovine tissues, milk	In the case of milk, blended with acetone, phases separated with chloroform. Transferred the residue to SkellysolveB/acetonitrile to clean-up the extract. Tissues extracted with acetonitrile/Skellysolve B and clean-up on a silica gel column	GC, thermionic detector	0.01 (milk) 0.1 (tissues)
Modified method 31093 (Ackerman *et al.,* 1975b)	Poultry tissues, eggs	Tissues and eggs extracted with acetonitrile/pentane, adding water and Na_2SO_4 to avoid severe emulsion formation. Clean-up on a silica gel column.	GC-FPD	0.02 (tissues) 0.05 (eggs)
Modified method 31093 (Anonymous, 1975a, 44526)	Poultry tissues, eggs	As above.	GC, thermionic detector	0.02
Method I506 (Stanley and Murphy, 1982 MR80594)	Soil	Residues in soil were subject to Soxhlet extraction with chloroform/methanol. Clean-up was by dilution with water, partition against benzene (discarded), addition of NaCl and partition into chloroform/acetone (2:1 v/v) and evaporation of the chloroform.	GC-FID	
Modification of method I506 (Grace and Cain, 1990, MR100166)	Soil	As above but with benzene replaced by toluene.	GC-NPD	0.01

[1/] The method for soil is identical with method 00156 (Möllhoff, 1976), which is the published version of method RA-315 (Möllhoff, 1973).

Satisfactory recoveries were obtained using the above methods on a wide range of crops. Recoveries from samples of broccoli, Brussels sprouts, cabbages, cauliflowers, lettuce and potatoes, fortified at 0.05-0.1 mg/kg and analyzed with Method RM-10, were 64-108% (Anonymous, 1973a 35291). From cauliflowers, paprika, broccoli, apples (fruit, juice, sauce, pomace), tomatoes, kale, potatoes, maize, peaches (fruit, jam, preserve, juice), rapeseed and rice, fortified at 0.01-1 mg/kg and analyzed using Method 00350, recoveries were 61-113% (Blaβ, 1996a TMN-0251D; Blaβ, 1996b TMN-0212F; Blaβ, 1998 MR-571/97; Blaβ, 1999 TMN-0212G; Blaβ, 2000 MR-129/00; Blaβ and Philipowski, 1995 TMN-0212H). Method 00219 yielded recoveries of 78-99% from samples of peanut kernels, peanut forage, sorghum grain, rapeseed and dry sugar beet pulp, fortified at 0.05-0.1 mg/kg (McNamara and Stanley, 1975 TMN-216; Anonymous, 1973b TMN-224A). For method 00137, recoveries were 56-120% from beet (roots and leaves), potatoes, rice (forage, grain, glumes), Chinese cabbages, broccoli, cabbages, cauliflowers, celery, collards, lettuce, mustard, spinach,

tomatoes and turnips, fortified at 0.01-5 mg/kg (Ohs, 1987 TMN-0210B; Ohs, 1988 TMN-0210D; Leary, 1971 TMN-220). The low value of 56% recovery represented a single recovery from sugar beet roots. The method of Anding (1995) was validated for apples (Grolleau, 1997a), sugar beet (Grolleau, 1997b), maize (Grolleau, 1997c) and potato (Grolleau, 1997d), with recoveries of 69-124% from samples fortified at 0.01-40 mg/kg. Recoveries using the Spanish multi-residue method IA were 79-92% from samples of peppers (Díaz and Gámon, 1995) and 76-83% from samples of peaches (Díaz and Gámon, 1996a) fortified at 0.01-0.5 mg/kg.

Samples of animal tissues (cattle liver, kidney, fat, meat and milk, chicken liver, kidney, skin, fat, muscle and eggs), fortified with methamidophos at 0.02-0.1 mg/kg and analyzed according Method 31093 produced recoveries of 64-120% (Stanley, 1971 TMN-210; Anonymous 1975a and 1975b TMN-0239A, TMN-0241A; Ackerman *et al,*. 1975b TMN-0241B). Cattle tissues and milk analyzed with Method RM-12A-2 gave recoveries of 64-120% from samples fortified at 0.025-0.1 mg/kg (Tucker, 1973, 3.0/44).

Enforcement Methods

Specht and Thier (1989) described DFG method S-19, for analysis of methamidophos residues in plant material (Bayer method No. 00086), later updated by Thier and Kirchoff (1992). Plant material was extracted with acetone and water, with the quantity of water added adjusted according to the water content of the plant material, to ensure a final acetone to water ratio of 2:1 (v/v). The extract was filtered, saturated with sodium chloride and partitioned with dichloromethane or, in a revised version, ethyl acetate/cyclohexane (Weeren *et al.,* 1999). Clean-up of the organic phase was by gel permeation chromatography for analysis by GC (ECD, NPD or FPD), using a wide-bore capillary column. If electron-capture detection was used, an additional clean-up step was required, using a silica gel column. The LOQ was 0.01 mg/kg. The average percentage recoveries ± %RSD, for samples spiked at 0.01 and 0.1 mg/kg and analyzed using GC-FPD, were 80 ± 10% from tomatoes, 85 ± 8.6% from oranges, 84 ± 7.6% from egg plants, 95 ± 6.0% from maize kernels and 88 ± 4.4% from tobacco leaves (Pelz and Linkerhägner, 2002a). Comparable results were obtained from confirmatory analyses performed by GC-NPD. In an independent laboratory validation of DFG method S-19 (extended revision) (Bayer method no. 00086/M042), Preu (2002a) chose tomatoes and oranges as representative matrices for validation. Average percentage recoveries ± %RSD, from samples spiked at 0.01 and 0.1 mg/kg and analyzed by GC-FPD, were 75 ± 9.2% from tomatoes and 74 ± 10% from oranges. A low mean recovery was obtained from orange samples spiked at 0.1 mg/kg, 67 ± 3.0%. However, this was not considered significantly below the 70-110% target range. Recoveries of 85-95% were obtained from apple and pear samples fortified at 0.01 mg/kg (Pelz, 1993). Weber and Pelz (2002) validated the revised DFG method S 19 for the determination of methamidophos residues in milk, meat, egg and fat. The average percentage recoveries from samples of milk (3.5% fat), bovine muscle and egg spiked at 0.01 and 0.1 mg/kg and samples of bovine fat spiked at 0.02 and 0.2 mg/kg and analyzed using GC-FPD, were: 64 ± 14% from milk; 80 ± 9.1% from bovine meat; 65 ± 7.2% from egg and 99 ± 2.4% from bovine fat. In an independent laboratory validation, using the method to analyze animal tissues the average percentage recoveries, from samples spiked at 0.01 and 0.1 mg/kg and using GC-FPD, were 7 1 ± 6.2% from cows' milk and 71 ± 9.4% from bovine muscle (Preu, 2002b). In all cases, the results of the confirmatory procedure using GC/NPD showed good correlation with those obtained using GC-FPD.

Stanley and Murphy (1982) developed a method for methamidophos residues in soils, i.e. method I506 (MR80594). Soil was extracted with chloroform/methanol by Soxhlet extraction and the extract evaporated to dryness. The residue was dissolved in water, washed with benzene and extracted with chloroform/acetone (2:1), after addition of NaCl. The extract was evaporated and dissolved in acetone for analysis by GC-NPD. Recoveries from soils fortified with methamidophos at 1 mg/kg were 84-95%. In a modification of this method, replacing benzene with toluene, using a different GC column and a nitrogen-phosphorus detector, recovery percentages at fortification levels of 0.01, 0.02 and 0.05 mg/kg were 75-114% (Grace and Cain, 1990). The limit of quantification was 0.01 mg/kg.

The applicability of the revised DFG method S-19 for the determination of residues of methamidophos in soils was validated by Pelz and Linkerhägner (2002b), also reported as Bayer method no. 00086/M043. Soil samples were extracted with acetonitrile, using an accelerated solvent extractor. Clean-up of the evaporated residue was by gel permeation chromatography, with detection of the residues by GC-FPD. Mean recoveries ± RSD from soil samples fortified at 0.01 and 0.1 mg/kg were 91 ± 10% and 88 ± 10%, respectively, for the two fortification levels. For confirmation, samples were also analyzed by GC-NPD. There was good correlation between the two sets of results, with average recoveries of 93 and 90% from samples fortified with methamidophos at 0.01 and 0.1 mg/kg and analyzed using GC-FPD and 103 and 74% for samples analyzed by GC-NPD. The limit of quantification was 0.01 mg/kg.

Burger (1987) determined methamidophos residues in water by drawing water samples through an extraction column filled with modified silica gel, eluting the residue with acetone and with detection by GC-FPD using cold on-column injection (method 2287/1811799/00). Recoveries from water samples fortified at 0.1-100 µg/l were 80-100%. The limit of quantification was 0.1 µg/l.

According to method 00138 (old method no. I 900; Ohs, 1989), NaCl was added to water samples prior to extraction of the residues by phase partition with ethyl acetate on diatomaceous earth. Detection of the residues was by GC-FPD (phosphorus mode). Satisfactory recoveries were obtained from water samples fortified at 0.2-100 µg/l. Validation of the method by Koenig (1989) established the limit of quantification as 0.05 µg/l. Recoveries from water samples spiked at 0.05 µg/l were 82-110%.

In Bayer method 00747 (Sommer, 2002), water samples were acidified with acetic acid to pH 4 for analysis by direct injection HPLC-MS/MS. The limit of quantitation was 0.05 µg/l.

Stability of residues in stored analytical samples

The Meeting received information on the stability of methamidophos residues during storage of analytical samples at freezer temperatures. Test data were provided for beef, broccoli, Brussels sprouts, cabbages, cauliflowers, celery, chicken, eggs, lettuce, milk, peanuts, peppers, potatoes, rape seed, sugar beet, tomatoes and sorghum.

Leary (1968) tested the freezer storage stability of field-incurred residues of methamidophos in chopped broccoli, lettuce, cabbages, cauliflowers and Brussels sprouts samples (Table 23). The samples were stored at approximately –20 °C for intervals up to 9 months. The analytical method used was RM-10.

Table 23. Freezer storage stability data for field-incurred methamidophos residues in macerated crop samples stored at approximately -20°C for intervals up to 9 months (Leary, 1968).

Crop	Days after last application	Storage interval (months)	Residue (mg/kg)	Procedural recovery (%)
Broccoli	3	0	1.4	
		6	1.2	98
		9	1.1	92
	7	0	1.3	
		6	1.2	98
		9	1.0	92
Lettuce	3	0	0.17	
		2	0.15	96
	7	0	0.08	
		2	0.04	96
Cabbages	3	0	0.07	
		5	0.08	95
		8	0.08	98
	7	0	0.05	
Cabbages, continued	7	5	0.05	95
		8	0.06	98
Cauliflowers	3	0	0.67	
		3	0.58	96

Crop	Days after last application	Storage interval (months)	Residue (mg/kg)	Procedural recovery (%)
		6	0.51	103
	7	0	0.51	
		3	0.64	96
		6	0.35	103
Brussels sprouts	3	0	0.71	
		2	0.60	108
		5	0.50	94
	7	0	0.67	
		2	0.43	108
		5	0.38	94

A series of summary reports detailed the storage stability of residues in samples of celery, peppers, sugar beet (tops and roots), peanut meat and vines, sorghum grain and forage and rapeseed, fortified with methamidophos at 1 mg/kg and stored at –18 to -23°C for various intervals (Table 24). Method 00137 was used for analysis of celery, peppers, sugar beet tops and roots and the sorghum forage stored for 10 months. Method 00219 was used for the other samples.

Table 24. Freezer storage stability data for methamidophos residues in macerated crop samples fortified at 1 mg/kg and stored at approximately -20°C.

Matrix	Storage interval (days)	Residue (mg/kg)	Reference
Celery	782	1.0, 1.1	44726
Peppers	782	0.78, 1.2	
Sugar beet (tops)	769	0.54, 0.77	48995
Sugar beet (roots)	769	0.58, 0.57, 0.71	
Peanut meal	46	0.92, 0.92	46491
	760	0.55, 0.58	
Peanut vines	42	0.98	46493
	60	0.84, 0.82	
	90	0.78, 0.83	
Peanut forage	760	0.85, 0.81	53942
Sorghum grain	28	0.95	46494
	43	1.0	
	62	0.89, 0.92	
	90	0.84, 0.82	
	205	0.97, 0.95	
Sorghum forage	31	0.82, 0.87	46492
	60	0.92, 0.90	
Sorghum forage	298	0.94, 1.24	46744
Rapeseed	46	0.92, 0.86	46495
	758	0.71, 0.63	

Williams (1994) tested the freezer storage stability of methamidophos residues in potato and tomato commodities. Samples were homogenised and fortified with methamidophos at 1.0 mg/kg and stored in glass jars, sealed with Teflon®-lined screw caps, at approximately –20 °C (typical temperature –20°C, range -8 to –34°C) for intervals up to 24 months. Sample sizes in the storage jars were 10 g. At each analysis interval, methamidophos residues were measured on one replicate stored sample, one procedural recovery was determined (at 0.3 mg/kg) and one control (unfortified) sample was analyzed. Method 00137 was used to analyze the samples and had been validated with material fortified at 0.3 to 1.0 mg/kg. Analytical recoveries were 99-105% from potato tubers, 73-81% from potato granules, 86-97% from potato dry peel, 80-85% from tomatoes, 67-88% from tomato purée and 102-114% from tomato dry pomace. Residues were stable during the storage periods examined (Table 25).

Table 25. Freezer storage (–20°C) stability data of methamidophos in fortified potato and tomato commodities (Williams, 1994) [1/].

Storage interval (months)	Procedural recovery (%)	Residue (mg/kg)		
		Potato RAC [2/]	Granules	Dry peel
0	-	0.99, 1.1	0.81, 0.73	0.97, 0.94
1	76	0.89, 0.76	0.32, 0.55	0.81, 0.86
3	100	0.93, 0.94	1.1, 0.92	0.91, 0.85
6	100	0.99, 0.92	0.95, 0.91	0.94, 0.94
12	105	1.0, 0.90	0.89, 0.86	0.88, 0.90
18	90	1.1, 1.1	0.97, 1.0	1.0, 1.0
24	98	1.1, 0.82	0.98, 0.99	1.0, 1.1
		Tomato RAC [2/]	Purée	Dry pomace
0	-	0.78, 0.82	0.88, 0.79	1.0, 1.1
1	79	0.80, 0.83	0.92, 0.86	0.88, 0.85
3	94	0.93, 0.94	0.93, 0.93	0.84, 0.86
6	96	0.87, 0.96	0.88, 0.92	0.61, 0.79
12	96	1.0, 0.93	0.93, 0.97	0.68, 0.67
18	111	0.99, 0.99	1.0, 0.91	0.76, 0.86
24	91	0.96, 0.95	0.95, 0.93	0.71, 0.69

[1/] Low-level residues were detected in control samples of potato at 1 month (0.04 mg/kg) and 18 months (0.14 mg/kg), in potato granules at 24 months (0.13 mg/kg), in potato dry peel at 18 months (0.06 mg/kg) and 24 months (0.2 mg/kg) and in tomato at 18 months storage (0.06 mg/kg).

[2/] RAC = raw agricultural commodity.

A series of summary reports detailed the storage stability of methamidophos in samples of cattle and poultry commodities, fortified at 1 mg/kg for tissues and 0.1 mg/kg for milk (Table 26). Samples were stored at –18 to –23°C for various intervals, with analysis by Chemagro method 31093 in the cases of cattle liver, muscle, fat and milk and by modified Chemagro method 31093 (Anonymous, 1975b) in the cases of chicken liver, heart/gizzard and eggs.

Table 26. Freezer storage stability data for methamidophos residues in macerated animal tissue and milk samples stored at approximately -20°C.

Matrix	Storage interval (days)	Residue (mg/kg)	Reference
Cattle liver	0	1.1, 1.1	34238
	95	0.26, 0.29	
	96	0.25, 0.30	
Cattle steak (muscle)	0	0.79, 0.89	
	93	0.97, 1.0	
Cattle fat	0	0.92, 0.95	
	92	0.98, 1.0	
Cattle milk	0	0.078, 0.105	
	96	0.095, 0.097	
Poultry liver	0	0.73, 0.66	44958
	30	0.67	
	60	0.52, 0.41	
Poultry heart/gizzard	0	0.98, 0.97	
	30	0.85	
	60	0.86	
Eggs	0	0.67, 0.72	
	30	0.62	
	60	0.62, 0.74	
	90	0.85	

Stanley (1982a, 1982b, 1982c) studied the freezer storage stability of methamidophos in three different soils (Table 27). Samples were stored at –17 to –23°C. The soil residues were stable for one year in the freezer.

Table 27. Freezer storage stability data for soil samples fortified at 1 mg/kg with methamidophos and stored at approximately -20°C (Stanley 1982a,1982b, 1982c).

Storage interval (days)	Residue (mg/kg)		
	Sandy loam	Silt loam	High organic silt loam
0	0.87, 0.96, 1.0	0.86, 0.82, 0.99	0.84, 0.96, 0.88
90	1.1, 1.1	0.65, 0.70	0.90, 0.86
188	0.85, 0.82	1.2, 1.0	1.0, 1.1
368	1.0, 1.1	0.72, 0.80	0.80, 0.71
1111-1119	0.74, 0.96	0.39, 0.38	0.43, 0.53, 0.53

The available storage stability data indicate that the residues of methamidophos are stable under frozen storage conditions (-20°C) in/on the following commodities (storage interval in parentheses): broccoli (9 months); lettuce (2 months); cabbage (8 months); cauliflower (6 months); Brussels sprouts (5 months); celery, peppers and peanut forage (26 months); sorghum grain (6 months); sorghum forage (10 months); rapeseed, potatoes (tubers, granules, dry peel) and tomatoes (fruit, purée, dry pomace) (24 months); bovine milk, meat, fat and poultry eggs (3 months) and poultry liver and heart/gizzard (2 months).

Methamidophos in cattle liver was not stable, with only 26% of residues remaining after 3 months frozen storage.

USE PATTERN

Methamidophos is registered in many countries as a broad-spectrum organophosphate insecticide on a range of crops. It has uses on citrus, pome and stone fruits, vines, potatoes, brassicas, beet, cotton, maize and other crops, for control of a wide range of chewing and sucking insects.

Information on registered uses was made available to the Meeting and those of relevance to this evaluation are summarized in Table 28.

Table 28. Registered methamidophos uses.

Crop	Country	Form	Application				PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No. (notes)	
Apricot	France	400 g/l SL	foliar		0.05		14
Apricot	Italy	228 g/l SL	foliar		0.03 - 0.06	2	35
Apricot	Spain	500 g/l SL	foliar		0.05 - 0.08	note 1	PF+10
Beet	Italy	228 g/l SL	foliar	0.4 - 0.57			21
Brassicas	Australia	580 g/l SL	foliar	0.6 - 1.2	0.06 - 0.11		7
Brassicas	Brazil	600 g/l SL	foliar	0.3 - 0.6	0.06		
Brassicas	New Zealand	600 g/l SL	foliar	0.6 - 0.9			7
Brassicas	South Africa	585 g/l SL	foliar		0.06		21
Broccoli	Canada	480 g/l SL	foliar	0.53 - 1.1			14
Broccoli	Mexico	600 g/l SL	foliar	0.6 - 0.9			21
Brussels sprouts	Canada	480 g/l SL	foliar	0.53 - 1.1	0.05 - 0.55		14
Brussels sprouts	Mexico	600 g/l SL	foliar	0.6 - 0.9			14
Cabbage	Canada	480 g/l SL	foliar	0.53 - 1.1			7
Cabbage	Germany	605 g/l SL	foliar	0.36		2	14
Cabbage	Greece	600 g/l SL	foliar		0.06 - 0.08		21
Cabbage	Korea	100 g/l SL	foliar	0.1 - 0.15	0.01	3	35
Cabbage	Mexico	600 g/l SL	foliar	0.6 - 0.9			35
Cabbage	Thailand	600 g/l SL	foliar	0.45 - 0.9			21
Capsicum	Australia	580 g/l SL	foliar	0.32 - 0.4	0.03 - 0.04		14
Capsicum	Australia	580 g/l SL	foliar	1.2	0.11		14
Capsicum	Brazil	600 g/l SL	foliar	0.3 - 0.6	0.06		
Cauliflower	Canada	480 g/l SL	foliar	0.53 - 1.1			7
Cauliflower	Germany	605 g/l SL	foliar	0.36		2	21
Cauliflower	Greece	600 g/l SL	foliar	0.6 - 0.9	0.06 - 0.08		21
Cauliflower	Mexico	600 g/l SL	foliar	0.6 - 0.9			28
Cotton	Argentina	580 g/l SL	foliar	0.2 - 0.6		proposed label	21
Cotton	Brazil	600 g/l SL	foliar	0.21 - 0.42			21

Crop	Country	Form	Application				PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No. (notes)	
Cotton	Brazil	600 g/l SL	foliar	0.6 - 1.2		note 2	21
Cotton	Columbia	600 g/l SL	foliar	0.3 - 0.9	0.03 - 0.09		
Cotton	Ecuador	600 g/l SL	foliar	0.3 - 0.9			21
Cotton	Greece	600 g/l SL	foliar	0.6 - 1.5			21
Cotton	Mexico	600 g/l SL	foliar	0.6 - 1.2			50
Cotton	Spain	500 g/l SL	foliar		0.05 - 0.08		35
Cotton	Thailand	600 g/l SL	foliar	0.45 - 0.9			21
Cotton	USA	400 g/l SL	foliar	0.11 - 0.56		1 - 2	50
Cotton	USA	400 g/l SL	foliar	0.56 - 1.12		1 - 2	50
Eggplant	Greece	600 g/l SL	foliar	0.6 - 0.9	0.06 - 0.08		21
Eggplant	Mexico	600 g/l SL	foliar	0.6 - 0.9			14
Eggplant	Thailand	600 g/l SL	foliar	0.45 - 0.9			21
Fodder beet	Germany	605 g/l SL	foliar	0.36 - 0.48		3	28
Fruit	Thailand	600 g/l SL	foliar	1.1 - 2.2			21
Kohlrabi	Germany	605 g/l SL	foliar	0.36		2	14
Maize	Greece	600 g/l SL	foliar	0.6 - 0.8	0.06 - 0.08		21
Maize	Italy	228 g/l SL	foliar		0.09 - 0.11		21
Maize	Spain	500 g/l SL	foliar		0.05 - 0.08		35
Nectarine	France	400 g/l SL	foliar		0.05	2	21
Nectarine	Spain	500 g/l SL	foliar		0.05 - 0.08	note 1	PF+10
Peach	Australia	580 g/l SL	foliar		0.03		21
Peach	France	400 g/l SL	foliar		0.05	2	14 - 21
Peach	Italy	228 g/l SL	foliar		0.03 - 0.06	2	35
Peach	Portugal	600 g/l SL	foliar	0.6	0.06	1	35
Peach	South Africa	585 g/l SL	foliar		0.06	2	28
Peach	Spain	500 g/l SL	foliar		0.05 - 0.08	note 1	PF+10
Peppers	Mexico	600 g/l SL	foliar	0.6 - 0.9			14
Peppers	Venezuela	600 g/l SL	foliar	0.6			15
Peppers (hot)	Mexico	600 g/l SL	foliar	0.6 - 0.9			21
Plums	France	400 g/l SL	foliar		0.05		14
Plums	Italy	228 g/l SL	foliar		0.03 - 0.06	2	21
Plums	Spain	500 g/l SL	foliar		0.05 - 0.08	note 1	PF+10
Potato	Argentina	580 g/l SL	foliar	0.46		proposed label	14
Potato	Australia	580 g/l SL	foliar	0.32 - 0.4	0.03 - 0.04		7
Potato	Brazil	600 g/l SL	foliar	0.3 - 0.6	0.06		21
Potato	Canada	480 g/l SL	foliar	0.9 - 1.1			14
Potato	Chile	600 g/l SL	foliar	0.3 - 0.6			15
Potato	Columbia	600 g/l SL	foliar	0.3 - 0.9	0.09 - 0.12		
Potato	Ecuador	600 g/l SL	foliar	0.3 - 0.6			21
Potato	Germany	605 g/l SL	foliar	0.48 - 0.6		7	14
Potato	Germany	605 g/l SL	foliar	0.73		1 (early season)	14
Potato	Greece	600 g/l SL	foliar		0.045 - 0.09		21
Potato	Italy	228 g/l SL	foliar	0.57			21
Potato	Mexico	600 g/l SL	foliar	0.6 - 0.9			14
Potato	New Zealand	600 g/l SL	foliar	0.48 - 0.6			7
Potato	Peru	600 g/l SL	foliar	0.6 - 1.2	0.12 - 0.18		14
Potato	Portugal	600 g/l SL	foliar	0.72	0.07	2	14
Potato	South Africa	585 g/l SL	foliar	0.15 - 0.3			14
Potato	USA	400 g/l SL	foliar	0.84 - 1.1	0.054 - 0.36	4.5kg ai/ha/season	14
Soya bean	Argentina	580 g/l SL	foliar	0.46 - 0.7		proposed label	30
Soya bean	Brazil	600 g/l SL	foliar	0.15 - 0.3			23
Soya bean	Brazil	600 g/l SL	foliar	0.3 - 0.5			23
Soya bean	Ecuador	600 g/l SL	foliar	0.45 - 0.6			21
Soya bean	Mexico	600 g/L SL	foliar	0.6 - 0.9			14
Sugar beet	Chile	600 g/l SL	foliar	0.3 - 0.6			15
Sugar beet	Germany	605 g/l SL	foliar	0.36 - 0.48		3	28
Tomato	Chile	600 g/l SL	foliar	0.3 - 0.6			15
Tomato	Columbia	600 g/l SL	foliar	0.3 - 0.9			

Crop	Country	Form	Application				PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No. (notes)	
Tomato	Ecuador	600 g/l SL	foliar	0.3 - 0.6			21
Tomato	Greece	600 g/l SL	foliar	0.6 - 0.9			21
Tomato	Mexico	600 g/l SL	foliar	0.6 - 0.9			7
Tomato	New Zealand	600 g/l SL	foliar	0.6 - 0.9			3
Tomato	Peru	600 g/l SL	foliar	0.6 - 1.2	0.12 - 0.18		14
Tomato	South Africa	585 g/l SL	foliar	0.3 - 0.6	0.058		3
Tomato	Thailand	600 g/l SL	foliar	0.45 - 0.9			21
Tomato	USA	400 g/l SL	foliar	0.84 - 1.1		5 (note 2)	7
Tomato (processing)	USA	400 g/l SL	foliar	0.84 - 1.1		2	14
Tomato	Venezuela	600 g/l SL	foliar	0.6			15
Tomato (field crops)	Australia	580 g/l SL	foliar	0.3-0.6	0.03-0.06		4
Tomato (field crops)	Australia	580 g/l SL	foliar	1.2	0.11		4
Tomato (field crops)	Brazil	600 g/l SL	foliar	0.3 - 0.6	0.06		21
Tomato (field crops)	Portugal	600 g/l SL	foliar	0.9 - 1.2	0.18 - 0.24	2	28
Vegetables	Ecuador	600 g/l SL	foliar	0.3 - 0.6			21

Note 1. Recommended use is for applications up to 10 days after petal fall (PF).

Note 2. Restricted use. Maximum of 5.6 kg ai/ha per season for non-processed crops.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for the following crops.

Table 30	Nectarines	Italy, Portugal
Table 31	Peaches	France, Italy, Spain
Table 32	Broccoli	Belgium, Germany, UK
Table 33	Cauliflowers	Canada, Germany
Table 34	Cabbages	Canada, Germany
Table 35	Tomatoes	Brazil, France, Greece, Italy, Mexico, Spain, Turkey, USA.
Table 36	Peppers	Brazil, Italy, Mexico, Spain, USA
Table 37	Soya beans	Brazil
Table 38	Potatoes	France, Germany, Greece, Italy and Spain.
Table 39	Potatoes	Canada, USA
Table 40	Sugar beet	France, Germany, Greece, Italy and Spain
Table 41	Fodder beet	Germany
Table 42	Maize	Germany, Greece, Spain
Table 43	Cotton seed	Brazil, India, USA

Recent trials were generally well documented, with full laboratory and field reports. Laboratory reports generally included method validation data, including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials, together with dates of analyses or duration of residue sample storage. Although trials included control plots, no control data are recorded in the tables, except where residues in control samples exceeded the LOQ (or in the earlier trials, the LOD). Residue data from the trials are recorded unadjusted for recovery.

Where residues were not detected they are shown as below the LOQ or, in the case of the early trials, below the LOD (e.g. <0.1 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are <u>double underlined</u>.

Conditions of the supervised residue trials are summarised in Table 29. Most trial designs, particularly in the earlier studies, used non-replicated plots. Multiple residues are recorded in the data

tables where the trial design included replicate plots and where separate samples have been identified as being from these replicate plots.

Intervals of freezer storage between sampling and analysis were recorded and were within the acceptable proven storage stability duration.

Table 29. Summary of sprayers, plot sizes and field sample sizes in the supervised trials.

Crop	Country	Year	Sprayer	Plot size	Sample size (min)
Broccoli	Belgium	1997	2m boom	17 m^2	2.4 kg
Broccoli	Germany	1997	1.5-2m boom	60-72 m^2	12 units, 1.4-2 kg
Broccoli	UK	1997	2.5m boom	100 m^2	12 units, 2.8 kg
Cabbage	Canada	1972			
Cabbage	Germany	1974		14 m^2	2-5 kg
Cabbage	Germany	1975		0.5 ha	3-4 heads
Cabbage	Germany	1976, 77		22-30 m^2	10-12 heads
Cauliflower	Canada	1972	hand sprayer		
Cauliflower	Germany	1976-78		10-50 m^2	8-10 heads
Cauliflower	Germany	1995	hand lance knapsack [2/]	10-50 m^2	12 heads
Cottonseed	Brazil	1988		80 m^2	
Cottonseed	India	1979		20 m^2	0.1 kg seed
Cottonseed	USA	1969, 73	4m tractor boom, aircraft	465 m^2, 2 x 6-23m rows	
Cottonseed	USA	1997	boom sprayers (tractor, research)	112-1030 m^2	
Fodder beet	Germany	1987		50-150 m^2	5.8-9 kg
Maize	Germany	1971, 73		100 m^2, 0.4ha	2.5-4kg
Maize	Greece	1996	knapsack hand lance [2/]	86-172 m^2	2 kg
Maize	Spain	1995	1.5-2.5m research boom	30-80 m^2	1.5-2 kg min
Nectarines	Italy	1995	hand gun knapsack [1/]	6 trees	
Nectarines	Portugal	1999	hand lance knapsack [2/]	65 m^2	1kg
Peaches	France	1995	hand gun knapsack [1/]	4 trees	
Peaches	Italy	1994	hand gun knapsack [1/], motorised lance[2]	4 trees	24 fruit
Peaches	Italy	1999	hand lance[2]	4 trees	24 fruit
Peaches	Spain	1994	hand gun knapsack[1]	4 trees	15-32 fruit
Peaches	Spain	1995	motorised hand lance[2]	4 trees	12 fruit
Peaches	Spain	1999	hand gun knapsack[1]	4 trees	24 fruit
Peppers	Brazil	1987			1.5-2 kg
Peppers	Italy	1997	2.5m boom	80 m^2	24 fruit
Peppers	Mexico	1996	motorised sprayer, tractor sprayer	6-8 x 10-40m rows	
Peppers	Spain	1994-95	motorised hand gun [1/] knapsack	15 plants	12-15 fruit
Peppers	Spain	1997	hand gun knapsack [1/]	25-40 m^2	2-5 kg
Peppers	USA	1987		45-220 m^2	
Potatoes	Canada	1972			
Potatoes	France	1979		13-36 m^2	2.5kg
Potatoes	Germany	1974, 77		12-7600 m^2	10 plants
Potatoes	Germany	1987, 88		25-100 m^2	4-17 kg
Potatoes	Greece	1996	2.5m boom	120 m^2	3.4-6 kg
Potatoes	Italy	1995	2.5m boom	30 m^2	1.3-3.2 kg
Potatoes	Spain	1985		25-80 m^2	3-7.7 kg
Potatoes	Spain	1995	2.5m boom	50 m^2	2.7-3.5 kg
Potatoes	USA	1987		4-20 x 12-100m rows	
Potatoes	USA	1996, 97	boom sprayers (tractor, research)	4-6 x 10-60m rows	
Soya beans	Brazil	1988			2 kg
Soya beans	Brazil	2000, 01	2.5-3m boom	24-50 m^2	1-1.5 kg
Sugar beet	France	1976		25-72 m^2	2.5-8 kg
Sugar beet	France	1984		37-50 m^2	6-7 kg
Sugar beet	France	1995	2.3m research boom	27-66 m^2	3.5 kg (roots), 2.5 kg (tops)
Sugar beet	Germany	1970, 71		250-2000 m^2	3-4.5 kg
Sugar beet	Germany	1975, 76		40-250 m^2	1.3-4.4 kg
Sugar beet	Greece	1995, 96	2.5m research boom	144 m^2	1.4-1.6 kg
Sugar beet	Italy	1989		60 m^2	3.3-6.5 kg
Sugar beet	Spain	1995, 96	2.5m research boom	30-100 m^2	3.3-4.1 kg

Crop	Country	Year	Sprayer	Plot size	Sample size (min)
Tomatoes	Brazil	1998			1.5 kg
Tomatoes	France	1988	hand gun knapsack [1/]	4.5 m^2, 6 plants	10 fruit, 1 kg
Tomatoes	Greece	2002	hand gun knapsack [1/]	50 m^2	30 fruit
Tomatoes	Italy	2002	hand lance knapsack [2/]	21 m^2	35 fruit
Tomatoes	Mexico	1973			
Tomatoes	Spain	1986	knapsack sprayer	31 m^2	1.5 kg
Tomatoes	Turkey	1989		70 m^2	0.5 kg
Tomatoes	USA	1981	aircraft	75 m^2	
Tomatoes	USA	1996	research sprayer, tractor & boom	50-560 m^2	2 kg

[1/] The term 'hand gun' is used to describe sprayers with a single-nozzle hand-held wand.
[2/] The term 'hand lance' is used when the hand-held wand consists of more than one nozzle.

Table 30. Methamidophos residues in nectarines resulting from supervised trials in Italy and Portugal.

NECTARINES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Italy, Metaponto, 1995, (Fantasia), RA-2112/95, 0639-95, (506397)	SL	0.75	0.05	1500	2	-0	with stone 0.08
						0	with stone 0.45
						7	with stone 0.15
						10	pulp 0.20
						14	pulp 0.06
						21	pulp 0.07
Italy, Ravenna, 1995, (Stark Red Gold) RA-2112/95, 0408-95, (504084)	SL	0.75	0.05	1500	2	-0	with stone 0.17
						0	with stone 0.56
						7	with stone 0.25
						10	with stone 0.24
						14	pulp 0.16
						21	pulp 0.10
Portugal, Lisboã, 1999, (E. King 2), RA-2160/99, 0662-99, (R1999 0662/5), whole fruit basis	SL	0.33	0.033	1000	2	-0	0.26
						0	0.62 c0.01
						7	0.25
						14	0.12
						21	0.07
						28	0.03
						35	0.02
						42	<0.01

c = result obtained from control sample.

Table 31. Methamidophos residues in peaches resulting from supervised trials in France, Italy and Spain.

PEACHES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
France, Avignon, 1995, (Meryl Gen Free) RA-2112/95, 0627-95, (506273)	SL	0.75	0.05	1500	2	- 0	with stone 0.78
						0	with stone 1.8
						7	with stone 0.87
						10	pulp 0.70
						14	pulp 0.44
						21	pulp 0.46
France, Avignon, 1995, (O'Harry) RA-2112/95, 0628-95, (506281)	SL	0.75	0.05	1500	2	- 0	with stone 0.15
						0	with stone 0.66
						7	with stone 0.54
						10	with stone 0.42
						14	with stone 0.41
						21	with stone 0.29
France, Avignon, 1995, (Primrose), RA-2112/95, 0017-95, (500178)	SL	0.75	0.05	1500	2	- 0	with stone 0.81
						0	with stone 2.4
						7	with stone 1.1
						10	pulp 0.66
						14	pulp 0.49
						21	pulp 0.38

PEACHES Location, year (variety), reference	Application Form	kg ai/ha	kg ai/hl	water, l/ha	no.	PHI days	Residues, mg/kg methamidophos
Italy, Monte S Biagio, 1994, (local variety) RA2107/94, 0675-94, (406759)	SL	0.6	0.05	1200	2	- 0 0 7 14 21 28	0.41 0.95 0.55 0.28 0.27 0.13
Italy, Monte S Biagio, 1994, (local variety) RA2107/94, 0672-94, (406724)	SL	0.68	0.057	1200	2	- 0 0 7 14 21 28 28	0.19 1.1 0.41 0.26 0.16 0.11 washed 0.08 juice 0.05 jam 0.1 preserve 0.09
Italy, Ravenna, 1994, (Red Haven), RA2107/94, 0674-94, (406740)	SL	0.6	0.05	1200	2	- 0 0 21 28	0.12 0.39 0.09 0.04
Italy, Ravenna, 1994, (Red Haven), RA2107/94, 0673-94, (406732)	SL	0.68	0.057	1200	2	- 0 0 21 28	0.13 0.67 0.12 0.06
Italy, Emilia Romagna, 1999, (Elegant lady) RA-2160/99, 0664-99, (R1999 0664/1)	SL	0.38	0.038	1000	2	- 0 0 7 14 21 28 35 42	0.11 0.34 0.10 0.07 0.01 0.01 <0.01 <0.01
Italy, Emilia Romagna, 1999, (Fayette), RA-2160/99, 0666-99, (R1999 0666/8)	SL	0.48	0.038	1250	2	- 0 0 7 14 21 28 35 42	0.35 0.64 0.34 0.39 0.16 0.07 0.04 0.02
Spain, Barcelona, 1994, (July Lady), RA2107/94, 0403-94, (404039)	SL	0.6	0.048	1250	2	- 0 0 6 14 21 28 28	0.26 1.3 0.59 0.27 0.09 0.09 washed 0.05 jam 0.03 preserve 0.02 juice 0.02
Spain, Figueres, 1994, (Baby Gold 9), RA2107/94, 0404-94, (404047)	SL	1.1 0.9	0.06 0.048	1900	1 +1	- 0 0 21 28	0.6 1.00 0.27 0.15
Spain, Valencia, 1995, (Maycrest), ER96ESP002, 0001-A	SL	0.74	0.048	1540	1	28 35	0.02 0.04
Spain, Valencia, 1995, (Maycrest), ER96ESP002, 0002-A	SL	0.77	0.048	1600	1	28 35	0.01 0.03
Spain, Valencia, 1995, (Maycrest), ER96ESP002, 0001-B	SL	1.1	0.072	1540	1	28 35	0.01 0.01
Spain, Valencia, 1995, (Maycrest), ER96ESP002, 0002-B	SL	1.2	0.072	1600	1	28 35	0.03 0.02

PEACHES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Spain, Barcelona, 1999, (Firered) RA-2160/99, 0663-99, (R1999 0663/3)	SL	0.45	0.033	1350		- 0	0.2
						0	1.0
						7	0.43
						14	0.2
						21	0.17
						29	0.05
						36	0.03
						42	0.02

Table 32. Methamidophos residues in broccoli resulting from supervised trials in Belgium, Germany and the UK.

BROCCOLI	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Belgium, Katelijne-Waver, 1997, (Fiesta), RA-2103/97, (706116)	SL	0.36	0.06	600	2	0	whole plant 7.5
						21	<0.01
Germany, Höfchen, 1997, (Green Valiant), RA-2103/97, (701432	SL	0.36	0.06	600	2	- 0	whole plant 0.04
						0	whole plant 5.0
						7	whole plant 0.61
						14	<0.01
						21	<0.01
						28	<0.01
Germany, Laacherhof, 1997, (Green Valiant), RA-2103/97, (701440)	SL	0.36	0.06	600	2	0	whole plant 4.3
						21	<0.01
UK, Bury St Edmunds, 1997, (Fiesta), RA-2103/97, (706108)	SL	0.36	0.06	600	2	-0	whole plant 0.6
						0	whole plant 2.8
						7	0.14
						14	0.03
						21	<0.01
						28	<0.01

Table 33. Methamidophos residues in cauliflowers resulting from supervised trials in Canada and Germany.

CAULIFLOWERS	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Canada, British Columbia, 1972, (Snowball Y), 35386	SL	1.1	0.14	814	8	0	0.48
						3	0.23
Canada, Ontario, 1972, (Snow Mound), 35385	SL	1.1	0.12	935	8	0	4.1
						3	2.0
						7	0.08
						14	0.03
Germany, Laacherhof, 1976, (Delfter Markt), 6701-76	SL	0.36	0.06	600	3	0	0.55
						7	0.02
						14	<0.01
						21	≤0.01
						28	<0.01
Germany, Laacherhof, 1976, (Malinus), 6703-76	SL	0.36	0.06	600	3	0	0.55
						7	0.07
						14	0.01
						21	≤0.01
						28	<0.01
Germany, Laacherhof, 1976, (Vedeslez), 6702-76	SL	0.36	0.06	600	3	0	0.7
						7	0.1
						14	0.04
						21	0.01
						28	<0.01

CAULIFLOWERS	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Germany, Höfchen, 1978, (Delfter Markt), 6700-78	SL	0.36	0.06	600	2	0	2.2
						3	1.1
						7	0.56
						14	0.31
						21	0.04
						28	<0.01
Germany, Laacherhof, 1978, (Delfter Markt), 6701-78	SL	0.36	0.06	600	2	0	0.37
						3	0.25
						7	0.07
						14	0.02
						21	0.01
						28	<0.01
Germany, Pfalz, 1978, (Delira), 6702-78	SL	0.36	0.06	600	2	0	0.55
						7	0.01
						14	<0.01
						21	≤0.01
						28	<0.01
Germany, Monheim, 1995, (Nautilus), RA-2014/95, (500259)	SL	0.36	0.06	600	1	0	whole plant except roots: 5.9
						21	≤0.01
Germany, Worms-Heppenheim, 1995, (Nautilus), RA-2014/95, (504343)	SL	0.36	0.06	600	1	0	whole plant except roots: 5.9
						21	≤0.01

Table 34. Methamidophos residues in cabbages resulting from supervised trials in Canada and Germany.

CABBAGES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Canada, British Columbia, 1972, (Golden Acre), 35383	SL	1.1	0.14	814	8	0	0.12
						3	0.08
						7	0.04
						14	0.04
Canada, Ontario, 1972, (King Cole), 35382	SL	1.1	0.12	935	8	0	0.38
						3	0.12
						7	0.60
						14	0.02
Canada, Ontario, 1972, (Wisconsin Golden Acre), 35384	SL	1.1	0.12	935	8	0	0.77
						3	1.6
						7	0.62
						14	0.15
Germany, Laacherhof, 1974, (Fruehwirsing Draeco HRZ) - Savoy cabbage, 6706-74	EC	0.3	0.05	600	2	0	3.0
						8	0.02
						14	0.01
						22	<0.01
Germany, Laacherhof, 1974, (Langendyker Frueher), Savoy cabbage, 6708-74	EC	0.3	0.05	600	2	0	4.8
						8	<0.01
						14	≤0.01
						22	<0.01
Germany, Laacherhof, 1974, (Marner Fruehwirsing G-F) - Savoy cabbage, 6707-74	EC	0.3	0.05	600	2	0	5.0
						8	0.01
						14	≤0.01
						22	<0.01
Germany, Burg Fehmarn, 1975, (Wirosa) - Savoy cabbage, 6704-75	EC	0.3	0.05	600	3	0	2.0
						8	0.9
						14	0.07
						22	0.02
						29	<0.01

CABBAGES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Germany, Laacherhof, 1976, (Fruhwirsing Praeco HKZ) - Savoy cabbage, 6700-76	SL	0.36	0.06	600	3	0	1.2
						7	0.9
						14	0.04
						21	0.03
						28	0.03
Germany, Leverkusen, 1976, (Juliwirsing) - Savoy cabbage, 6710-76	EC	0.3	0.05	600	3	0	0.98
						7	0.2
						14	0.09
						21	0.02
						28	<0.01
Germany, Laacherhof, 1977, (Dithmarscher Fruhstamm 49) - White cabbage, 6701-77	SL	0.36	0.06	600	3	0	<0.01 [1/]
						7	0.16
						14	0.03
						21	0.02
						28	0.05
Germany, Laacherhof, 1977, (Fruhwirsing Praeco HKZ) - Savoy cabbage, 6700-77	SL	0.36	0.06	600	3	0	1.95
						7	0.23
						14	0.20
						21	0.06
						28	0.07

[1/] No explanation given for the day 0 reported residue.

Table 35. Methamidophos residues in tomatoes resulting from supervised trials in Brazil, France, Greece, Italy, Mexico, Spain, Turkey and the USA.

TOMATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Brazil, São Paulo, 1998, 639/88 (BRA-639-88-A)	SL	0.6	0.06	1000	3	0	0.03
						4	<0.01
						7	<0.01
						14	<0.01
						21	<0.01
Brazil, São Paulo, 1998, 639/88 (BRA-639-88-B)	SL	1.2	0.12	1000	3	21	<0.01
France, St Croix du Monde, 1988, (Rio Grande), TE 2376 (TE 2376-A)	SC	0.44	0.06	733	2	20	0.05
France, St Croix du Monde, 1988, (Rio Grande), TE 2376 (TE 2376-B)	SC	0.44	0.06	733	1	20	0.03
France, St Croix du Monde, 1988, t Croix du Monde, (Rio Grande), TE 2376 (TE 2376-C)	SC	0.44	0.06	733	1	13	0.04
France, Villandrout, 1988, (St Pierre), TE 2392 (TE 2392-A)	SL	1.3	0.06	2187	1	14	<0.02
						21	0.07
France, Villandrout, 1988, (St Pierre), TE 2392 (TE 2392-B)	SL	1.3	0.06	2187	2	21	<0.02
Greece, GR-Vasilica, 2002, RA-2012/02 (R2002 0001/3)	SL	0.39	0.06	650	3	-0	0.03
						0	0.15
						3	0.18
						7	0.11
						10	0.04
Italy, Trinitapoli, 2002, RA-2012/02 (R2002 0321/7)	SL	0.6	0.06	1000	3	-0	0.08
						0	0.4
						3	0.14
						7	0.08
						10	0.08

TOMATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Mexico, Sinaloa, 1973, (Tropic), 37313	EC	0.6			7	0	0.1
						1	0.09
						3	0.08
						5	0.15
						7	0.08
						14	0.06
						21	0.03
							c0.01
Mexico, Sinaloa, 1973, (Tropic), 37312	EC	0.45			1	0	0.16
		0.6			+6	1	0.13
						3	0.16
						5	0.09
						7	0.11
						14	0.07
						21	0.03
Spain, Alicante, 1986, (Bornia), TE-2233 (TE 2233-A)		0.72	0.045	1600	2	1	<0.02
						3	<0.02
						7	<0.02
Spain, Alicante, 1986, (Bornia), TE-2233 (TE 2233-B)		0.72	0.045	1600	4	1	0.03
						3	0.04
						7	<0.02
Spain, Alicante, 1986, (Bornia), TE-2234 (TE 2234-A)	SC	1.0	0.06	1666	2	1	0.22
						3	0.44
						7	0.14
Spain, Alicante, 1986, (Bornia), TE-2234 (TE 2234-B)	SC	1.0	0.06	1666	4	1	0.15
						3	0.07
						7	0.12
Turkey, Bursa, 1989, (Lerica), 0042-89	SL	0.6	0.06	1000	2	0	0.17
						7	0.1
						14	<0.01
USA, California, 1981, (315), 80510	SL	1.1	1.2	93.5	5 air	0	0.07
						3	0.12
						8	0.07
						11	0.08
						15	0.06
USA, California, 1981, (Macero II), 80509	SL	1.1	1.2	93.5	5 air	0	0.11
						3	0.07
						8	0.05
						11	0.03
						15	0.02
USA, California, 1996, (Aztex), 108061 (457-MN116-96D)	SC	1.1	0.3	371	5	8	0.24
						16	0.14
						22	0.1
						29	0.08
USA, California, 1996, (Gargiulo), 108061 (458-MN118-96D)	SC	1.1	0.3	375	5	7	0.56
						14	0.36
						21	0.2
						27	0.22
USA, California, 1996, (Maxi Rio), 108061 (457-MN114-96D)	SC	1.1	0.28	397	5	8	0.16
						13	0.06
						23	0.08
						30	0.1
USA, California, 1996, (Maxi Rio), 108061 (457-MN115-96D)	SC	1.1	0.3	372	5	8	0.36
						16	0.24
						22	0.1
						29	0.18
USA, California, 1996, (Red Sun), 108061 (457-MN112-96D)	SC	1.1	0.3	371	5	7	0.22
						15	0.08
						22	0.08
						30	0.08

TOMATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
USA, California, 1996, (Roma), 108061 (455-MN107-96D)	SC	1.1	0.4	280	5	7	0.86
						14	0.63
						21	0.58
						28	0.32
USA, California, 1996, (Santiago), 108061 (455-MN111-96D)	SC	1.1	0.3	374	5	9	0.14
						16	0.07
						21	0.05
						29	0.03
USA, California, California, 1996, (Shady Lady), 108061 (FCA-MN106-96D)	SC	1.1	0.33	342	5	7	0.36
						14	0.16
						21	0.12
						28	0.1
USA, California, 1996, (Tango), 108061 (455-MN110-96D)	SC	1.1	0.3	374	5	6	0.26
						13	0.12
						20	0.15
						26	0.12
USA, California, 1996, (UC-82B), 108061 (455-MN108-96D)	SC	1.1	0.4	280	5	7	0.31
						14	0.3
						21	0.24
						28	0.1
USA, California, 1996, (UC-82B), 108061 (455-MN109-96D)	SC	1.1	0.4	280	5	7	0.12
						16	0.06
						23	0.04
						28	0.04
USA, Florida, 1996, (Agri-Set), 108061 (VBL-MN103-96D)	SC	1.1	0.37	300	5	5	1.5
						10	1.21
						15	1.04
						20	0.67
USA, Indiana, 1996, (Heinz 8813), 108061 (HIN-MN105-96D)	SC	1.1	0.41	270	5	7	1.3
						14	1.4
						21	0.84
						28	0.54
USA, New Jersey, 1996, (Better Boy), 108061 (856-MN102-96D)	SC	1.1	0.38	296	5	7	0.42
						14	0.32
						21	0.21
						28	0.12
USA, Pennsylvania, 1996, (Better Boy), 108061 (353-MN104-96D)	SC	1.1	0.27	400	5	7	0.14
						14	0.05
						21	<0.02
						29	<0.02
USA, Pennsylvania, 1996, (Better Boy), 108061 (856-MN101-96D)	SC	1.1	0.4	280	5	7	1.3
						14	0.95
						21	0.8
						28	0.4

Table 36. Methamidophos residues in sweet peppers resulting from supervised trials in Brazil, Italy, Spain and the USA and hot peppers in Mexico.

PEPPERS	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Brazil, São Paulo, 1987, Sweet peppers, 646/88 (BRA-646-88-A)	SL	0.6	0.06	1000	4	0	0.06 c0.01
						14	0.02
						21	<0.01
						28	<0.01
Brazil, São Paulo, 1987, Sweet peppers, 646/88 (BRA-646-88-B)	SL	1.2	0.12	1000	4	21	0.01
Italy, Latina, 1997, (Gordo) Sweet peppers, RA-2015/97 (704571)	SL	1.2	0.12	1000	3	-0	0.05
						0	0.53
						3	0.18
						7	0.17
						10	0.15

PEPPERS Location, year (variety), reference	Application Form	kg ai/ha	kg ai/hl	water, l/ha	no.	PHI days	Residues, mg/kg methamidophos
Spain, Cartagena, 1994, (Hariner F1), TMN266B (ER94ESP001001)	SL	1.0			3	7 15	0.06 0.05
Spain, San Pedro del Pinatar, 1994, (Atol) Sweet peppers, TMN266B (ER94ESP001002)	SL	1.0			3	7 15	0.14 0.24
Spain, Valencia, 1995, (Sweet Italian) Sweet peppers, TMN266B (ER95ESP001001)	SL	1.0			3	0 3 7 15	0.25 0.08 0.19 0.06
Spain, Valencia, 1995, (Sweet Italian) Sweet pepper, TMN266B (ER95ESP001002)	SL	1.0			3	0 3 7 15	1.1 0.24 0.49 0.05
Spain, Barcelona, 1997, (Largo Italiano) Sweet peppers, RA-2015/97 (704563)	SL	1.3 1.2	0.12 0.12	1050 1000	1 +2	-0 0 3 7 10	0.09 0.9 0.28 0.38 0.1
Spain, Barcelona, 1997, (PR Vuyo) Sweet peppers, RA-2015/97 (701475)	SL	1.3 1.2 1.2	0.12 0.08 0.12	1060 1540 1000	1 +1 +1	-0 0 3 7 10	0.23 0.8 0.15 0.18 0.25
Spain, Valencia, 1997, (Lamuyo) Sweet peppers, RA-2015/97 (704555)	SL	1.2	0.12	1000	3	-0 0 3 7 10	0.19 0.58 0.31 0.43 0.19
USA, California, 1987, (F-1 Hybrid Sunny) Sweet peppers, 95665 (456-MN007-87D)	EC	1.1	0.33	337	5	0 3 7 14	0.72 0.33 0.14 0.04
USA, California, 1987, (Jupiter) Sweet peppers, 95665 (458-MN011-87D)	EC	1.1	0.3	374	5	0 3 7 14	0.8 0.29 0.52 0.38
USA, Florida, 1987, (Gator Belle) Sweet peppers, 95665 (VBL-MN010-87D)	EC	1.1	0.3	374	5	0 3 7 14	0.62 0.26 0.21 0.22
USA, North Carolina, 1987, (Burpees Hybrid) Sweet peppers, 95665 751-MN008-87D) c=0.02	EC	1.1	0.4	281	5	0 3 7 14	0.57 0.35 0.31 0.03
USA, Texas, 1987, (Big Bertha) Sweet peppers, 95665 RTX-MN012-87D-A	EC	1.1	0.3	374	5	0 3 7 14	1.5 0.75 0.87 0.95
USA, Texas, 1987, (Big Bertha) Sweet peppers, 95665 RTX-MN012-87D-B)	EC	1.1	0.3	374	6	0 3	1.1 1.2
USA, Texas, 1987, (Grande Rio 66) Sweet peppers, 95665 (RTX-MN009-87D)	EC	1.1	0.6	187	5	0 3 7 14	0.35 0.36 0.22 0.07
Mexico, Sinaloa, 1996, (Jalapeno) Hot peppers, 108052 MEX-MN-P01-96H	SL	0.9	0.28	325	5	14	0.42
Mexico, Chihuahua, 1996, (Jalapeno) Hot peppers, 108052 MEX-MN-P02-96H	SL	0.9	0.3	300	5	14	0.1
Mexico, Vera Cruz, 1996, (Mitla) Hot peppers, 108052 MEX-MN-P03-96H	SL	1.1	0.3	366	5	14	0.43

c = result obtained from control sample.

Table 37. Methamidophos residues in soya beans resulting from supervised trials in Brazil.

SOYA BEANS	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Brazil, São Paulo, 1984, 0978-84-A	SL	0.48			1	15	<0.01
						21	≤0.01
Brazil, São Paulo, 1984, 0978-84-B	SL	0.96			1	21	<0.01
Brazil, São Paulo, 1988, 0644-88-A	SL	0.48	0.48	100	3	0	with pod 0.19
						5	<0.01
						8	<0.01
						16	<0.01
						23	≤0.01
Brazil, São Paulo, 1988, 0644-88-B	SL	0.96	0.96	100	3	23	<0.01
Brazil, Chapadão do Ceu, 2000, (Monsoy 2000), BRA-057-00-A	SL	0.48	0.48	100	3	23	≤0.04
Brazil, Chapadão do Ceu, 2000, (Monsoy 2000), BRA-057-00-B	SL	0.96	0.96	100	3	23	<0.04
Brazil, Iracemapolis SP, 2001, (IAC 19), BRA-034-01-A	SL	0.48	0.48	100	3	23	≤0.04
Brazil, Iracemapolis SP, 2001, (IAC 19), BRA-034-01-B	SL	0.96	0.96	100	3	23	<0.04

Table 38. Methamidophos residues in potatoes resulting from supervised trials in France, Germany, Greece, Italy and Spain.

POTATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
France, Moustoir Remungol, 1979, (Bintje), 6700-79	EC	0.6	0.1	600	5	45	<0.01
Germany, Höfchen, 1974, (Hela), 6704-74	EC	0.6	0.1	600	1	0	<0.01
		0.5	0.08		+1	14	≤0.01
		0.4	0.07		+1	22	<0.01
						28	<0.01
Germany, Höfchen, 1974, (Saskia), 6705-74	EC	0.6	0.1	600	1	0	<0.01
		0.5	0.08		+1	14	≤0.01
		0.4	0.07		+1	22	<0.01
						28	<0.01
Germany, Laacherhof, 1974, (Saskia), 6703-74	EC	0.6	0.1	600	1	0	<0.01
		0.5	0.08		+1	14	≤0.01
		0.4	0.07		+1	22	<0.01
						28	<0.01
Germany, Höfchen, 1977, (Grata), 6703-77	SL	0.72	0.12	600	2	5	<0.01
						7	<0.01
						14	≤0.01
						21	<0.01
Germany, Krs. Celle, 1977, (Frigga), 6736-77	SL	0.6	0.15	400	3	11	0.01
		0.48	0.12		+4	18	<0.01
						25	<0.01
Germany, Krs. Celle, 1977, (Frigga), 6734-77	SL	0.6	0.15	400	2	0	<0.01
		0.48	0.12		+3	7	<0.01
						14	≤0.01
						21	<0.01
						35	<0.01
Germany, Krs. Celle, 1977, (Saskia), 6735-77	SL	0.6	0.15	400	3		
		0.48	0.12		+3	7	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
						42	<0.01
Germany, Krs. Celle, 1977, (Frigga), 6733-77	SL	0.6	0.15	400	2	0	<0.01
		0.48	0.12		+1	7	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01

POTATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Germany, Laacherhof, 1977, (Saskia), 6702-77	SL	0.72	0.12	600	2	0	<0.01
						5	<0.01
						7	<0.01
						14	≤0.01
						21	<0.01
Germany, Osterath, 1977, (Grata), 6704-77	SL	0.72	0.12	600	2	0	0.02
						4	<0.01
						7	<0.01
						14	≤0.01
Germany, Stade, 1977, (Grata Z), 6721-77	SL	0.6	0.2	300	3	0	0.02
						7	0.03
						14	0.02
						21	0.02
						28	0.02
Germany, Stade, 1977, (Grata Z), 6726-77	SL	0.6 0.48	0.2 0.16	300	3 +2	0	0.01
						7	0.01
						14	0.01
						21	0.01
						28	0.01
Germany, Stade, 1977, (Grata Z), 6722-77	SL	0.6 0.48	0.2 0.16	300	3 +2	0	<0.01
						7	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
Germany, Stade, 1977, (Grata Z), 6723-77	SL	0.6 0.48	0.2 0.16	300	3 +3	0	<0.01
						7	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
Germany, Stade, 1977, (Grata Z), 6727-77	SL	0.6 0.48	0.2 0.16	300	3 +3	0	<0.01
						7	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
Germany, Stade, 1977, (Grata Z), 6728-77	SL	0.6 0.48	0.2 0.16	300	3 +4	0	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
Germany, Stade, 1977, (Grata Z), 6724-77	SL	0.6 0.48	0.2 0.16	300	3 +4	0	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
Germany, Uelzen, 1977, (Taiga), 6731-77	SL	0.6 0.48	0.15 0.12	400	3 +3	0	<0.01
						14	0.02
						28	<0.01
Germany, Uelzen, 1977, (Taiga), 6729-77	SL	0.6 0.48	0.15 0.12	400	2 +1	0	0.01
						7	<0.01
						14	≤0.01
						21	<0.01
						28	<0.01
Germany, Uelzen, 1977, (Taiga), 6732-77	SL	0.6 0.48	0.15 0.12	400	3 +4	0	<0.01
						14	≤0.01
Germany, Uelzen, 1977, (Taiga), 6730-77	SL	0.6 0.48	0.15 0.12	400	2 +3	0	<0.01
						7	≤0.01
						21	<0.01
						28	<0.01
Germany, Höfchen, 1987, (Grandifolia), 6804-87	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01

POTATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Germany, Klein-Niedersheim, 1987, (Ulla), 6806-87	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Germany, Laacherhof, 1987, (Grata), 6805-87	EC	0.6	0.09	700	7	0	0.01
						7	0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Germany, Worms-Heppenheim, 1987, (Grata), 6807-87	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Germany, Höfchen, 1988, (Grandifolia), 0395-88	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Germany, Klein-Niedersheim, 1988, (Culpa), 0397-88	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Germany, Laacherhof, 1988, (Granola), 0396-88	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Germany, Worms-Heppenheim, 1988, (Ulla), 0398-88	EC	0.6	0.09	700	7	0	<0.01
						7	<0.01
						10	<0.01
						14	≤0.01
						21	<0.01
Greece, Macedonia, 1996, (Hermes), EA950150 (EA950150-GR01)	SL	0.64	0.09	710	1	22	≤0.01
		0.66	0.09	730	+1		
		0.63	0.09	700	+1		
Greece, Macedonia, 1996, (Spunta), EA950150–GR02	SL	0.64	0.09	710	3	0	0.02
						3	0.03
						7	0.02
						15	0.01
						21	≤0.01
Italy, Lombardia, 1995, (Hermes), EA950150 (EA950150-IT01)	SL	0.5	0.07	700	1	21	≤0.01
		0.53	0.07	745	+1		
		0.51	0.07	711	+1		
Italy, Lombardia, 1995, (Mona Lisa), EA950150 (EA950151-IT01)	SL	0.49	0.07	680	1	0	<0.01
		0.47	0.07	664	+1	3	<0.01
		0.5	0.07	713	+1	7	<0.01
						14	<0.01
						21	≤0.01
Spain, Valencia, 1995, (Etzina), EA950150 (EA950150-SP02)	SL	0.6	0.086	700	3	21	≤0.01
Spain, Canet de Mar, 1985, (Mona Lisa), 6800-85	EC	0.5	0.08	625	1	15	≤0.01
Spain, Valencia, 1995, (Etzina), EA950150 (EA950150-SP01)	SL	0.6	0.086	700	3	21	≤0.01
Spain, Valencia, 1995, (Etzina), EA950151–SP01	SL	0.6	0.086	700	3	0	<0.01
						3	<0.01
						7	<0.01
						14	<0.01
						21	≤0.01

Table 39. Methamidophos residues in potatoes resulting from supervised trials in Canada and the USA.

POTATOES	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Canada, British Columbia, 1972, (Netted Gem), 35405	SL	1.1	0.12	729	8	14 29	≤0.01 <0.01
Canada, Ontario, 1972, (Norland), 35404	SL	1.1	0.12	935	8	15 30	≤0.01 <0.01
Canada, Ontario, 1972, (Superior), 35451	SL	1.12	0.12	935	8	15 30	≤0.01 <0.01
USA, Colorado, 1987, (Norgold Russet), 96716 (253-MN003-87D)	EC	1.1	4.0	28 air	4	7 14	<0.01 ≤0.01
USA, Idaho, 1987, (Russet Burbank), 96716 (453-MN004-87D)	EC	1.1	0.4	280	4	8 15	<0.01 ≤0.01
USA, Maine, 1987, (Russet Burbank), 96716 (152-MN001-87D)	EC	1.1	0.12	935	2	8 15	<0.01 ≤0.01
USA, North Dakota, 1987, (Norchip), 96716 (251-MN002-87D)	EC	1.1	2.42	46 air	4	7 14	<0.01 ≤0.01
USA, Washington, 1987, (Russet Burbank), 96716 (454-MN005-87D)	EC	1.1	0.27	412	4	7 14	<0.01 ≤0.01
USA, California, 1996, (Red Lasota), 108060 FCA-MN011-96H	SL	1.1		358	4	14	≤0.05
USA, Colorado, 1996, (Centennial), 108060 453-MN010-96H	SL	1.1	0.4	280	4	14	≤0.05
USA, Florida, 1996, (Atlantic), 108060 VBL-MN004-96H	SL	1.1		316	4	14	≤0.05
USA, Idaho, 1996, (Russet Burbank), 108060 451-MN012-96D	SL	1.1	0.35	318	4	7 14 21 28	<0.05 ≤0.05 <0.05 <0.05
USA, Idaho, 1996, (Russet Burbank), 108060 452-MN014-96H	SL	1.1		264	4	13	≤0.05
USA, Idaho, 1996, (Russet Burbank), 108060 452-MN015-96H	SL	1.1		280	4	14	≤0.05
USA, Idaho, 1996, (Russet Burbank), 108060 452-MN016-96H	SL	1.1		256	4	14	≤0.05
USA, Indiana, 1996, (Norkota Russet), 108060 HIN-MN005-96D	SL	1.1		270	4	7 14 21 28	<0.05 ≤0.05 <0.05 <0.05
USA, Kansas, 1996, (Kennebee), 108060 STF-MN006-96H	SL	1.1	0.48	234	4	14	≤0.05
USA, Maine, 1996, (Katahdin), 108060 854-MN002-96H	SL	1.1		333	4	14	≤0.05
USA, Minnesota, 1996, (Red Pontiac), 108060 851-MN008-96H	SL	1.1	0.43	262	4	14	≤0.05
USA, Nebraska, 1996, 108060 SNE-MN007-96H	SL	1.1	0.48	232	4	14	≤0.05
USA, New York, 1996, (Chippewa), 108060 854-MN001-96H	SL	1.1	0.4	280	4	14	≤0.05
USA, Oregon, 1996, (Russet Burbank), 108060 451-MN013-96H	SL	1.1	0.35	318	4	14	≤0.05
USA, Washington, 1996, (Norkota), 108060 454-MN017-96H	SL	1.1		288	4	14	≤0.05
USA, Washington, 1996, (Russet Burbank), 108060 454-MN018-96H	SL	1.1		274	4	14	≤0.05
USA, Wisconsin, 1996, (Russet Burbank), 108060 851-MN009-96H	SL	1.1	0.3	374	4	14	≤0.05
USA, Georgia, 1997, 108060 TGA-MN003-96H	SL	1.1		312	4	14	≤0.05

Table 40. Methamidophos residues in sugar beet resulting from supervised trials in France, Germany, Greece, Italy and Spain.

SUGAR BEET	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
France, Baigneaux, 1976, (Polybeta), 6714-76	SL	0.4	0.08	500	1	134	tops <0.01 roots <0.01
France, Blincourt, 1976, (Monohill/Monobel), 6715-76	SL	0.4	0.09	450	1	141	tops <0.01 roots <0.01
France, Capelle par Templeuve, 1976, (Gigamono), 6716-76	SL	0.5	0.1	500	1	117	tops <0.01 roots <0.01
France, Doudeville, 1984, (Monohill), 6700-84	SL	0.5	0.1	500	1	152	tops <0.02 roots <0.01
France, St Hilaire Le Petit, 1984, (Monosvalof), 6701-84	SL	0.5	0.125	400	1	105	tops 0.01 roots <0.02
France, Livry, 1995, (Roberta), EA950142 EA950142-FR01	600 SL	0.48	0.1	480	2	28	tops 6.1 roots ≤0.01
France, Moulins, 1995, (Gabriella), EA950142 EA950143-FR01	600 SL	0.49	0.1	490	2	0 7 14 21 28	tops 14 roots 0.01 tops 9.1 roots 0.01 tops 13 roots <0.01 tops 5.0 roots <0.01 tops 2.3 roots ≤0.01
Germany, Laacherhof, 1970, 228-70	SL	0.59	0.07	800	2	64	root <0.01 tops 0.02
Germany, Leverkusen, 1970, 230-70	SL	0.59	0.07	800	2	72	roots <0.01 tops <0.01
Germany, Paulinehof, 1970, 229-70	SL	0.59	0.07	800	2	72	roots <0.01 tops <0.01
Germany, Laacherhof, 1971, 104-71	SL	0.59	0.15	400	2	80 101	roots <0.01 roots <0.01
Germany, Laacherhof, 1971, 105-71	SL	0.59	0.15	400	2	80 101	tops 0.02 tops 0.02
Germany, Paulinehof, 1971, 102-71	SL	0.59	0.15	400	2	78 99	roots <0.01 roots <0.01
Germany, Paulinehof, 1971, 103-71	SL	0.59	0.15	400	2	78 99	tops 0.06 tops 0.05
Germany, Kirchherten, 1975, (Klein Wandsleben E), 6714-75	SL	0.48	0.048	1000	4	23 50 105	tops 1.4 tops 0.05 roots <0.01 tops <0.01 roots <0.01
Germany, Kirchherten, 1975, (Polybeta), 6713-75	SL	0.48	0.048	1000	4	23 50 89	tops 0.6 roots 0.01 tops 1.5 roots <0.01 tops <0.01 roots <0.01
Germany, Wevelinghoven, 1975, (Gem 65), 6715-75	SL	0.48	0.048	1000	4	28 55 118	tops 0.9 roots ≤0.01 tops 0.1 roots <0.01 tops <0.01 roots <0.01
Germany, Laacherhof, 1976, (Gem 65), 6711-76	SL	0.48	0.08	600	4	0 42 56 62 70	tops 5.2 tops 0.07 tops 0.05 tops 0.02 tops <0.01 roots <0.01

SUGAR BEET	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Germany, Laacherhof, 1976, (Kawemono), 6712-76	SL	0.48	0.08	600	4	0 42 56 62 70	tops 6.4 tops 0.09 tops 0.03 tops 0.03 tops 0.03 roots <0.01
Germany, Laacherhof, 1976, (Kawemono), 6713-76	SL	0.48	0.08	600	4	0 42 56 62 70	tops 6.7 tops 0.07 tops 0.04 tops 0.03 tops 0.02 roots <0.01
Greece, Akrini, 1995, (Rizor), EA950142 EA950142-GR01	SL	0.5	0.1	500	2	29	tops <0.01 roots <0.01
Greece, Riakion, 1996, (Bingo), EA950142 EA950143-GR02	600 SL	0.5	0.1	500	2	0 7 14 21 28	tops 5.6 roots <0.01 tops 5.2 roots <0.01 tops 1.3 roots <0.01 tops 0.39 roots <0.01 tops 0.28 roots <0.01
Italy, S Romualdo, 1989, (Monfort), 0383-89	SL	0.5	0.06	800	2	0 20 30 40	roots <0.005 roots <0.005 roots <0.005 roots <0.005
Spain, Tobarra, 1995, (Gabriella), EA950142 EA950142-SP01	SL	0.51	0.1	510	2	28	tops 2.7 roots <0.01
Spain, Roderno, 1996, (Orix), EA950142 EA950143-SP02	600 SL	0.5	0.1	500	2	0 7 14 21 28	tops 8.6 roots <0.01 tops 6.0 roots <0.01 tops 2.3 roots <0.01 tops 2.7 roots <0.01 tops 0.08 roots <0.01

Table 41. Methamidophos residues in fodder beet resulting from supervised trials in Germany.

FODDER BEET	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Germany, Höfchen, 1987, (Kyros), 6712-87	SL	0.48	0.12	400	2	0 14 28 42	tops 13 c=0.02 roots 0.01 tops 6.2 roots <0.01 tops 3.1 roots ≤0.01 tops 1.1 roots <0.01
Germany, Höfchen, 1987, (Kyros), 6715-87	SL	0.48	0.12	400	2	0 14 28 42	tops 14 c=0.02 roots <0.01 tops 4.7 roots <0.01 tops 2.1 roots ≤0.01 tops 0.53 roots <0.01

FODDER BEET Location, year (variety), reference	Application Form	kg ai/ha	kg ai/hl	water, l/ha	no.	PHI days	Residues, mg/kg methamidophos
Germany, Laacherhof, 1987, (Brigadier), 6713-87	SL	0.48	0.12	400	2	0 14 28 42	tops 16 c=0.13 roots <0.01 tops 7.2 roots <0.01 tops 2.8 roots ≤0.01 tops 1.1 roots <0.01
Germany, Laacherhof, 1987, (Brigadier), 6716-87	SL	0.48	0.12	400	2	0 14 28 42	tops 13.2 c=0.12 roots <0.01 tops 4.9 roots <0.01 tops 2.9 roots ≤0.01 tops 1.3 roots <0.01
Germany, Worms-Heppenheim, 1987, (Brigadier), 6714-87	SL	0.48	0.08	600	2	0 14 28 42	tops 7.4 roots 0.03 tops 1.8 roots <0.01 tops 0.49 roots ≤0.01 tops 0.05 roots <0.01
Germany, Worms-Heppenheim, 1987, (Brigadier), 6717-87	SL	0.48	0.08	600	2	0 14 28 42	tops 6.8 roots 0.13 tops 2.1 roots <0.01 tops 0.54 roots ≤0.01 tops 0.03 roots <0.01

c = result obtained from control sample.

Table 42. Methamidophos residues in maize resulting from supervised trials in Germany, Greece and Spain.

MAIZE Location, year (variety), reference	Application Form	kg ai/ha	kg ai/hl	water, l/ha	no.	PHI days	Residues, mg/kg methamidophos
Germany, Rohrbach, 1971, (Inrakorn), 0149-71	SL	0.9	0.23	400	2	51	grain <0.01
Germany, Monheim, 1973, (Inrafruh), 0160-73	EC	0.75	0.13	600	2	4 40 61	whole plant 1.9 whole plant 0.06 grain <0.01
Germany, Monheim, 1973, (Prior), 0159-73	EC	0.75	0.13	600	2	4 40 61	whole plant 3.9 whole plant 0.03 grain <0.01
Germany, Monheim, 1973, (Velox), 0158-73	EC	0.75	0.13	600	2	4 40 61	whole plant 1.7 whole plant 0.03 grain <0.01
Greece, Galatista, 1996, (PR 3165), EA950146 EA950146-GR03	SL	0.77	0.09	820	2	59	cob <0.01 grain <0.01
Greece, Paleochora, 1996, (Kostanza), EA950146 EA950146-GR01	SL	0.79 0.77	0.09	833 821	1 +1	60	cob <0.01 grain <0.01
Greece, Paleochora, 1996, (Ulis), EA950146 EA950147-GR04	SL	0.77 0.76	0.09	818 809	2	0 14 60	plant 60 cob 0.92 plant 6.0 c=0.01 cob 0.08 cob <0.01 grain <0.01
Greece, Pegonia, 1996, (X 8931), EA950146 EA950146-GR02	SL	0.8 0.77	0.09	852 819	1 +1	59	cob <0.01 grain <0.01

MAIZE	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Spain, Bel-Lloc, 1995, (Constanza), EA950146 EA950146-SP02	SL	0.75	0.09	800	2	55	cob <0.01 grain <0.01
Spain, Bel-Lloc, 1995, (Sicilia), EA950146 EA950146-SP01	SL	0.77	0.09	813	2	44	cob ≤0.01 grain ≤0.01
Spain, Valencia, 1995, (Laurus), EA950146 EA950147-SP01	SL	0.7 0.79	0.09	746 840	1 +1	0 14 60	plant 35 cob 0.2 plant 3.8 c0.04 cob 0.03 cob <0.01 grain <0.01
Spain, Valencia, 1995, (Nelson), EA950146 EA950147-SP02	SL	0.75 0.74	0.09	800 790	1 +1	0 14 60	plant 22 cob 0.12 plant 2.5 cob 0.02 cob <0.01 grain <0.01

c = result obtained from control sample.

Table 43. Methamidophos residues in cotton seed resulting from supervised trials in Brazil, India and the USA.

COTTON SEED	Application					PHI	Residues, mg/kg
Location, year (variety), reference	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	days	methamidophos
Brazil, São Paulo, 1988, 643/88 0643-88-A	SL	1.2	0.4	300	4	0 4 7 14 21	boll 0.26 0.02 <0.01 <0.01 ≤0.01
Brazil, São Paulo, 1988, 643/88 0643-88-B	SL	2.4	0.8	300	4	21	<0.01
India, Tamil Nadu, 1979, (Varalaxmi), 6725/86 (IND 6725-86-A)	SC	0.6	0.06	1000	3	14 21	<0.01 <0.01
India, Tamil Nadu, 1979, (Varalaxmi), 6725/86 (IND 6725-86-B)	SC	1.2	0.12	1000	3	14 21	<0.01 <0.01
USA, California, 1969, (Acala SJ 1), T-1787	SL	1.1	1.5	75 air	4	47	≤0.01 (2) trash 0.29, 0.9
USA, California, 1969, (Acala SJ 1), T-1789	SL	2.2	0.48	466	4	52 62 72	seed <0.01 meal <0.01 delinted seed <0.01 hulls <0.01 oil <0.01 trash 0.92, 1.1 seed <0.01 meal <0.01 delinted seed <0.01 hulls <0.01 oil <0.01 trash 0.5, 0.57 seed <0.01 meal <0.01 delinted seed <0.01 hulls <0.01 oil <0.01 trash 0.11, 0.2
USA, Mississippi, 1969, (Stoneville 213), T-1788	SL	1.1	2.4	47 air	4	32	<0.01 (2) trash 0.57, 0.77

COTTON SEED Location, year (variety), reference	Application Form	kg ai/ha	kg ai/hl	water, l/ha	no.	PHI days	Residues, mg/kg methamidophos
USA, Mississippi, 1969, (Stoneville 213), T-1790	SL	2.2	0.96	233	4	33	seed <0.01 (2) meal <0.01 (2) delinted seed <0.01 (2) hulls <0.01 (2) oil <0.01 (2) trash 2.7, 2.4
						44	seed <0.01 (2) meal <0.01 (2) delinted seed <0.01 (2) hulls <0.01 (2) oil <0.01 (2) trash 0.87, 0.8
						54	seed <0.01 (2) meal <0.01 (2) delinted seed <0.01 (2) hulls <0.01 (2) oil <0.01 (2) trash 0.64 (2)
USA, California, 1973, (Acala SJ 1), T-2527 (T-2527 A)	EC	1.1	0.31	365	7	18	seed 0.01 (2) lint 0.69 (2) trash 1.3, 1.0
USA, California, 1973, (Acala SJ 1), T-2527 (T-2527 B)	EC	1.1	0.31	365	6	32	seed <0.01 (2) lint 0.35, 0.48 meal <0.01 (2) hulls <0.01 (2) crude oil <0.01 (2) refined oil <0.01 (2) trash 0.29, 0.31
USA, California, 1973, (Acala SJ 1), T-2527 (T-2527 C)	EC	1.1	0.31	365	5	39	seed ≤0.01 (2) lint 0.19, 0.37 meal <0.01 (2) hulls <0.01 (2) crude oil <0.01 (2) refined oil <0.01 (2) trash 0.35, 0.26
USA, California, 1973, (Acala SJ 1), T-2527 (T-2527 A)	EC	2.2	0.61	365	7	18	0.03
USA, California, 1973, (Acala SJ 1), T-2527 (T-2527 B)	EC	2.2	0.61	365	6	26	0.02
USA, California, 1973, (Acala SJ 1), T-2527 (T-2527 C)	EC	2.2	0.61	365	5	39	0.01
USA, California, 1973, (Acala SJ 1), T-2528 (T-2528 A)	EC	1.1	2.4	47 air	7	7	0.02 (2)
USA, California, 1973, (Acala SJ 1), T-2528 (T-2528 B)	EC	1.1	2.4	47 air	6	18	<0.01 (2)
USA, California, 1973, (Acala SJ 1), T-2528 (T-2528 C)	EC	1.1	2.4	47 air	5	25	<0.01 (2)
USA, Florida, 1973, (Stoneville 213), T-2529 (T-2529 A)	EC	1.1	0.31	365	6	32	seed <0.01 (2) lint <0.01 (2)
USA, Florida, 1973, (Stoneville 213), T-2529 (T-2529 B)	EC	1.1	0.31	365	7	25	seed <0.01 (2) lint <0.01 (2)
USA, Florida, 1973, (Stoneville 213), T-2529 (T-2529 C)	EC	1.1	0.31	365	8	19	seed <0.01 (2) lint <0.01 (2)
USA, Florida, 1973, (Stoneville 213), T-2529 (T-2529 A)	EC	2.2	0.61	365	6	32	<0.01 (2)
USA, Florida, 1973, (Stoneville 213), T-2529 (T-2529 B)	EC	2.2	0.61	365	7	25	<0.01 (2)
USA, Florida, 1973, (Stoneville 213), T-2529 (T-2529 C)	EC	2.2	0.61	365	8	19	<0.01 (2)

COTTON SEED Location, year (variety), reference	Application Form	kg ai/ha	kg ai/hl	water, l/ha	no.	PHI days	Residues, mg/kg methamidophos
USA, Mississippi, 1973, (Stoneville 213), T-2530 (T-2530 B)	EC	1.1	0.31	365	6	17	seed <0.01, 0.01 lint 1.0, 1.0 meal <0.01 (2) hulls 0.28, 0.25 crude oil <0.01 (2) refined oil <0.01 (2) trash 1.2, 1.8
USA, Mississippi, 1973, (Stoneville 213), T-2530 (T-2530 C)	EC	1.1	0.31	365	5	33	seed <0.01 (2) lint 0.61, 0.79 meal <0.01 (2) hulls <0.01 (2) crude oil <0.01 (2) refined oil <0.01 (2) trash 0.45, 0.37
USA, Mississippi, 1973, (Stoneville 213), T-2530 (T-2530 A)	EC	1.1	0.31	365	7	6	seed 0.03 (2) lint 5.1, 3.5 trash 2.4, 2.6
USA, Mississippi, 1973, (Stoneville 213), T-2530 (T-2530 B)	EC	2.2	0.61	365	7	6	0.04, 0.05
USA, Mississippi, 1973, (Stoneville 213), T-2530 (T-2530 A)	EC	2.2	0.61	365	6	17	<0.01, 0.01
USA, Mississippi, 1973, (Stoneville 213), T-2530 (T-2530 C)	EC	2.2	0.61	365	5	33	<0.01 (2)
USA, Arizona, 1997, (Delta Pine 5461), 108317 458-MN012-97H	EC	1.1	0.8	141	4	48	≤0.01 trash 1.5
USA, Arkansas, 1997, (PM 1215 RR), 108317 354-MN003-97H	EC	1.1	1.04	108	4	49	≤0.01
USA, California, 1997, (Acala Maxxa), 108317 FCA-MN010-97H	EC	1.1	0.68	164	4	53	0.06 trash 7.7
USA, California, 1997, (Maxxa), 457-MN011	EC	1.1	0.66	170	4	49	0.16 trash 4.3
USA, Georgia, 1997, (DPL 5415), 108317 TGA-MN001-97H	EC	1.1 1.2 1.1	1.0 1.1 1.1	110 112 104	2 +1 +1	50	≤0.01
USA, Louisiana, 1997, (DPL 50), 108317 355-MN004-97H	EC	1.1	0.74	152	4	48	≤0.01
USA, Mississippi, 1997, (DPL 50), 108317 BMS-MN002-97D-A	EC	1.1	0.9	125	4	57 69	≤0.01 trash 0.85 <0.01 trash 0.07
USA, Mississippi, 1997, (DPL 50), 108317 BMS-MN002-97D-B	EC	1.1	0.9	125	4	42 42 52 52	<0.01 trash 1.8 ≤0.01 trash 0.10
USA, Oklahoma, 1997, (PM 183), 108317 456-MN007-97H	EC	1.1	0.8	140	4	52 52	0.05 trash 0.69
USA, Texas, 1997, (2156), 108317 456-MN009-97H	EC	1.1	0.9	125	4	54 54	0.06 trash 0.20
USA, Texas, 1997, (2156), 108317 456-MN006-97D	EC	1.1 1.2 1.1	0.9 1.0 0.9	123 123 126	2 +1 +1	42 52 61 70	0.08 0.09 0.07 0.06
USA, Texas, 1997, (Paymaster HS 2000), 108317 456-MN008-97H	EC	1.1	0.8	140	4	50 50	0.01 trash 0.20
USA, Texas, 1997, (Suregrow 125), 108317 459-MN005-97H	EC	1.1	0.82	137	4	50	≤0.01

FATE OF RESIDUES IN STORAGE AND PROCESSING

Processing studies were provided on apples (Spain), peaches (Italy, Spain), tomatoes (USA), potatoes (USA), soya beans (USA), sugar beet (USA) and cotton seed (USA), together with a range of studies

reporting the effects of washing, cooking or dehydration on residues in brassica vegetables, tomatoes, peppers and potatoes.

A processing experiment conducted in Spain (Heinemann and Ohs, 1996a) on apples, treated twice with methamidophos in the field with 1.2 kg ai/ha (0.08 kg ai/hl) and harvested 21 days after treatment, studied the residues in raw apples, washed apples, juice, apple sauce and wet and dry pomace (Table 44). Using simulated commercial practice, apple sauce was prepared by washing, dicing and heating the apples with 125 ml water/kg of fruit, at 98-100 °C for 15-20 minutes, before screening to separate the sauce and pomace. Sugar (100 g/l of sauce) was then added before pasteurisation (84-90 °C). Juice was prepared by mashing and pressing the washed, diced apples, with the raw juice being flash-heated to 90 °C for 30 seconds and cooled to 40-50°C before enzyme treatment (pectinase and amylase). The juice was then centrifuged, filtered and pasteurised (up to 89 °C for about 40 seconds) before being bottled. The wet pomace remaining after the juice extraction was dried to obtain the dry pomace (4-10% water content).

Diessler and Blaβ (1998), in two similar studies in Spain and Italy with peaches, investigated the effects of washing and processing (jam, preserve and juice) on the behaviour of methamidophos residues. Fruit picked 28 days after the last of two methamidophos treatments (see Table 44) were washed and processed into jam, juice and preserve. The jam and preserve were prepared by peeling the washed peaches, removing the stones and either adding gelling sugar and cooking at 100 °C for 3-4 minutes (jam) or adding sugar syrup and pasteurising (preserve). Juice was prepared by mashing the stoned peaches, adding 0.1% ascorbic acid and heating in a microwave oven to 65 °C for 10 minutes before pressing the mash to separate the juice. The raw juice was centrifuged, diluted to 50% with water, returned to a Brix level of about 12° by adding sugar and then pasteurised for 15-150 seconds at 82-90 °C before bottling.

Table 44. Methamidophos residues in raw and processed apples and peaches resulting from processing studies in Spain and Italy.

COMMODITY	Application				PHI days	Residues, mg/kg methamidophos	Processing factor
Location, year (variety), reference	Form	kg ai/ha	water, l/ha	no.			
APPLES							
Spain, Barcelona, 1990, (Suprema), RA-3102/98	SC	1.2	1500	2	21	0.14	
						washed 0.1, 0.12, 0.13	0.83
						juice 0.06, 0.1, 0.06	0.52
						sauce 0.11, 0.12, 0.11	0.81
						wet pomace 0.03 (3)	0.21
						dry pomace 0.12, 0.11 (2)	0.79
PEACHES							
Italy, Monte S Biagio, 1994, (local variety), RA2107/94 (406724)	SL	0.68	1200	2	28	0.11	
						washed 0.08	0.73
						juice 0.05	0.45
						jam 0.1	0.91
						preserve 0.09	0.82
Spain, Barcelona, 1994, (July lady), RA2107/94 (404039)	SL	0.6	1250	2	28	0.09	
						washed 0.05	0.55
						juice 0.02	0.22
						jam 0.03	0.33
						preserve 0.02	0.22

Fujie (1986) reported on five studies on tomato processing conducted in USA. In two of these studies (New Jersey and California), fruit were harvested 0-10 days after the last of 12 treatments with methamidophos at 1.1 kg ai/ha and were processed into juice, purée and canned fruit. In another study, in Iowa, tomatoes treated at 1.1 kg ai/ha were harvested on the day the last of four applications was made and were processed into juice and wet pomace. A similar study, in New Jersey, involved tomatoes from crops treated 3 times with 1.1 kg ai/ha and harvested 7 and 14 days after the last treatment. While processing details were not available, methamidophos residues in the various processed fractions are summarised in Table 45.

In the fifth study, tomatoes were treated with an exaggerated rate of methamidophos (3.4 kg ai/ha), harvested (green) one day after treatment and allowed to ripen for 3-7 days, then were processed into juice, canned (peeled) tomatoes and catsup (ketchup). The juice was prepared by blanching the raw tomatoes in boiling water for one minute before they were peeled and cored by hand and pressed through cheesecloth to separate the juice, which was then pasteurised at 82 °C for 18 minutes. Peeled, cored tomatoes were also heated on a steam bath for 40 minutes at 100 °C to simulate the sterilization procedure used commercially for canned tomatoes. Residues were also measured in dry pomace (consisting of the skins and cores, together with the seeds and fibre remaining after juice extraction) and in dry pulp, derived from unpeeled whole tomatoes after pressing to remove half the juice (designed to reflect commercial processing of cull tomatoes for animal feed). In both cases, the drying process was with hot air, and the final moisture content was between 5% and 8%.

In a more recent study by Lenz (1994a), methamidophos was applied six times to field-grown tomatoes at the exaggerated rate of 5.6 kg ai/ha. The fruit were harvested 7 days after the last application and processed in laboratory-scale experiments that simulated commercial practice. Tomatoes were washed to remove dirt and debris and then passed through a chlorine dip and rinsed with potable water. The fruit were then crushed through a 4mm screen, flash-heated to about 100 °C, before passing the crushed material through a fine screen (0.8 mm) to remove the skins and seeds. The resultant juice was canned, sealed and cooked for 50 minutes at 115 °C. Tomato purée and sauce were prepared by concentrating the juice using a single-pass, wiped-surface evaporator, with the paste (at 24-25% soluble solids) and purée (17% soluble solids) being canned and cooked at 88 °C for 5 minutes. The results are summarized in Table 45.

A cooking study in Germany (Möllhoff, 1978), also summarized in Table 45, compared residues found in raw tomatoes with those present in tomatoes cooked either in an open system or in a closed (reflux condenser) system.

Table 45. Methamidophos residues in raw and processed tomatoes resulting from processing studies in USA and Germany.

TOMATOES	Application				PHI	Residues, mg/kg	Processing
Location, year (variety), reference	Form	kg ai/ha	water, l/ha	no.	days	methamidophos	factor
USA, New Jersey, 1968,	EC	1.12	935	12	0	1.6, 1.1	
(Campbell 1327),						juice 0.66, 0.59	0.42
98332 (T-1168)						purée 0.70, 0.62	0.44
						canned 0.59, 0.56	0.37
					3	0.94, 0.65	
						juice 0.38, 0.2	0.40
						purée 0.49, 0.34	0.74
						canned 0.51, 0.45	0.54
					10	0.52, 0.32	
						juice 0.42, 0.43	0.81
						purée 0.42	0.81
						canned 0.42, 0.38	0.81
USA, California, 1968, (Ace),	EC	1.12	468	12	0	1.7	
98332 (T-1169)						juice 0.75	0.44
						purée 0.87	0.51
						canned 0.55	0.32
					3	0.77	
						juice 0.7	0.91
						purée 0.8	1.0
						canned 0.83	1.1
					10	0.71	
						juice 0.38	0.53
						purée 0.62	0.87
						canned 0.58	0.82

TOMATOES	Application				PHI	Residues, mg/kg	Processing
Location, year (variety), reference	Form	kg ai/ha	water, l/ha	no.	days	methamidophos	factor
USA, Iowa, 1982, (Better Boy), 98332 (T-5570)	EC	1.12	625	4	0	0.42 juice 0.33 wet pomace 0.52	 0.79 1.2
USA, New Jersey, 1982, (Campbell 1372), 98332 (T-5571	EC	1.12	468	3	7	0.53 juice 0.66 wet pomace 0.6 dry pomace 2.7	 1.2 1.1 5.1
					14	0.4 juice 0.35 wet pomace 0.32 dry pomace 1.5	 0.88 0.8 3.8
USA, 1974, 98332 (41107)	EC	3.36		1	1 +3-7 ripen ing	1.5 juice 1.4 canned fruit 1.1 catsup 1.0 dry pomace 3.1 dry pulp 3.8	 0.93 0.73 0.67 2.1 2.5
USA, 1990, 101236 (Lenz, 1994a) (FCA-MN002-90P)		5.56	374	6	7	2.17 juice 2.65 wet pomace 1.29 purée 6.23 paste 8.79 dry pomace <1.0	 1.2 0.60 2.9 4.1 0.37
Germany, Laacherhof, 1978, RA 857	EC	0.36	600	2	0	0.067 cooked (open) 0.067 0.055 cooked (closed) 0.048	 1.0 0.97

A study by Lenz (1994b) in California (Table 46) reported the effects of processing on methamidophos residues in potatoes. Potatoes, harvested 14 days after the last of 10 treatments with methamidophos at the exaggerated (5×) rate of 5.6 kg ai/ha, were processed in the laboratory in a manner that simulated commercial practice. The potatoes were brush-washed in water containing 3-5 ppm chlorine, hand-peeled, trimmed and washed before further processing. Chips were prepared by slicing the peeled potatoes (3mm thick), blanching them for 1 minute in water at 88 °C and frying them in vegetable oil at 177 °C for 2 minutes. Potato granules were prepared by dicing the peeled, rinsed potatoes and boiling them for 15-20 minutes, before mashing and drying to less than 5% moisture using a forced air dryer at 80-82 °C. As no residues were observed in the raw commodity prior to processing, the study cannot be used to estimate processing factors.

Table 46 also summarizes a study by Misra *et al.* (1990) on the effects of storage and cooking on methamidophos residues in potatoes in India. Potatoes were harvested 20 days after the last of 5 treatments with 0.38 kg ai/ha or 0.63 kg ai/ha and stored at room temperature for 20, 30 or 60 days before being analyzed. Samples of fresh and stored potatoes were also analyzed after peeling, washing and boiling.

Table 46. Methamidophos residues in raw and processed potatoes resulting from processing studies in USA and India.

POTATOES	Application				PHI	Residues, mg/kg	Processing
Location, year (variety), reference	Form	kg ai/ha	water, l/ha	no.	days	methamidophos	factor
USA, California, 1990, (Red La Soda), Lenz, 1994b, (101235)	SC	5.6	374	10	14	<0.01 granules <0.01 chips 0.02 wet peel <0.01 dry peel <0.01	 2

POTATOES	Application				PHI	Residues, mg/kg	Processing
Location, year (variety), reference	Form	kg ai/ha	water, l/ha	no.	days	methamidophos	factor
India, 1983, Misra *et al.,* 1990	SC	0.375	1250	6	20	1.1	
						cooked 0.2	0.18
						cooking water 0.45	0.41
						stored 30 days 0.09	0.08
						stored 30d, cooked <0.02	
						cooking water <0.02	
						stored 60 days <0.02	
						stored 60d, cooked <0.02	
						cooking water <0.02	
		0.625	1250	5	20	1.45	
						cooked 0.3	0.21
						cooking water 0.5	0.34
						stored 30 days 0.14	0.1
						stored 30d, cooked <0.02	
						cooking water <0.02	
						stored 60 days <0.02	
						stored 60d, cooked <0.02	
						cooking water <0.02	

A soya bean processing study was conducted in USA (Anonymous 1984b, report 86903 – see Table 47) in 1984. Beans harvested 15 days after the last of two applications of methamidophos (1.7 kg ai/ha) were threshed and hulled on a laboratory mill, with the meats being separated by screening on a flat, perforated screen (to recover about 50% of the added seed). The meats were then flaked to about 4-5 mm thick, and the oil was extracted by soaking in solvent for 30 minutes at 38 °C. The oil was recovered from the solvent by vacuum evaporation and further processed by de-acidification, to produce refined oil and soapstock, with the refined oil being further treated with steam to produce deodorised oil.

Table 47. Methamidophos residues in raw and processed soya beans resulting from processing studies in USA.

SOYA BEANS	Application				PHI	Residues, mg/kg	Processing
Location, year (variety), reference	Form	kg ai/ha	water, l/ha	no.	days	methamidophos	factor
USA, Indiana, 1984, (Pioneer 3901), 86903	SL	1.7	234	2	15	beans (no pod) 0.08	
						hulls 1.08	13.5
						flakes 0.06	0.75
						meal 0.13	1.6
						crude oil <0.01	<0.13
						refined oil <0.01	<0.13
						deodorized oil <0.01	<0.13
						soapstock <0.01	<0.13

One processing study conducted in the USA on sugar beet is summarized in Table 48, in which sugar beet roots were harvested 3 days after the last of six applications of methamidophos (1.1 kg ai/ha) and processed into juice, pulp, molasses and sugar (Anonymous 1977x). Processing involved slicing the beet before passing them through a diffuser (at 24-27 °C) to separate the juice from the wet pulp, after which the juice was treated with 2% milk of lime and carbonated and filtered twice at 27-30 °C, to produce thin juice and lime cake. Thick juice was then prepared from the thin juice by vacuum evaporation and further processed into molasses and sugar.

Table 48. Methamidophos residues in raw and processed sugar beet resulting from processing studies in the USA.

SUGAR BEET Location, year (variety), reference	Application				PHI days	Residues, mg/kg methamidophos	Processing factor
	Form	kg ai/ha	water, l/ha	no.			
USA, Colorado, 1977, (Mono Hy D2), 53030	EC	1.1	186	6	3	roots 0.05	
						diffusion juice <0.01	<0.2
						juice <0.01	<0.2
						thin juice <0.01	<0.2
						thick juice <0.01	<0.2
						lime cake <0.01	<0.2
						wet pulp <0.01	<0.2
						dry pulp <0.01	<0.2
						molasses <0.01	<0.2
						raw sugar <0.01	<0.2
						Moisture contents:	
						wet pulp 84%	
						dry pulp 49%	

A residue study conducted in the USA on cotton (Leslie, 1989) and summarized in Table 49, involved the analysis of cotton seed and processing fractions from seed which were harvested from a field trial in Texas, where five treatments of an exaggerated rate of 5.6 kg ai/ha had been applied up to 7 days before harvest. After using a cotton gin to separate seed cotton into cottonseed, lint and gin trash, a delinter was used to remove the lint and then the seed was hulled and screened to separate the hull and kernel fractions. The kernels were preheated to 74 °C and flaked to 2-3 mm thick before the oil was extracted with hexane, using a steam-jacketed extractor at 63 °C for 30 minutes (this extraction being repeated five more times). The spent meal was then aired for 4 hours to remove the remaining solvent and the crude oil was recovered from the hexane using a laboratory evaporator at 85 °C. Refinement of the crude oil was by reaction with sodium hydroxide (15 minutes at 20-24 °C then 12 minutes at 63-67 °C), followed by 60 minutes settling at 60-65 °C after which the refined oil was separated from the soapstock by decanting and filtered.

Table 49. Methamidophos residues in raw and processed cotton seed resulting from processing studies in the USA.

COTTON SEED Location, year (variety), reference	Application				PHI days	Residues, mg/kg methamidophos	Processing factor
	Form	kg ai/ha	water, l/ha	no.			
USA, Texas, 1989, (Stoneville 825), 99786 (Leslie, 1989)	EC	5.6	187	5	7	0.74	
						meal 0.43	0.58
						hulls 0.56	0.76
						soapstock <0.01	<0.014
						crude oil <0.01	<0.014
						refined oil <0.01	<0.014

The effect of dehydration on methamidophos residues in peppers has been reported by Thornton (1973). Thirty fresh peppers were treated with the equivalent of 0.6 kg ai/ha and left outdoors for 24 hours. The seeds were then removed, the peppers were chopped and dried in a forced-air oven for 24 hours at 60 °C, removing 93% by weight as water. The concentration of methamidophos increased in proportion, to about ten times the level in the raw peppers.

In a study conducted in Taiwan and reported by Tsai *et al.* (1997), broccoli samples from crops treated 10 days before harvest with methamidophos at rates of 0.4 kg ai/ha and 0.6 kg ai/ha were subjected to a range of washing or dipping treatments. Broccoli were either washed in running water (10 ml/minute) or dipped in water (1 litre/100 g broccoli) for 0.5, 1, 3 or 5 minutes before being analyzed. Reported reductions in residue concentrations in the washed broccoli ranged from 11% (0.5 minute wash) to 48% (5 minute wash), in the equivalent dipping experiments the reductions in concentration were 4-51%. In addition, raw broccoli samples were also dipped for 3 minutes in brine, at concentrations of 0.5%, 1%, 2% and 3%, before being analyzed. At the lowest brine concentration, methamidophos concentrations were reduced by 19-46%, while at the highest (5%) concentration, 36-

53% of residues were removed. This study also reported on a preliminary blanching study, where samples of field-treated broccoli were blanched in steam (90°C) for 1 minute or dipped in either water or 2% brine at 80 °C (5 minutes) or 95°C (3 minutes). In this preliminary study, residue reductions of 99-100% were reported for all treatments except the 5 minute dip in water at 80°C, where a 97.4% residue reduction was reported.

Savoy cabbages, treated twice in the field with a 0.06 kg ai/hl (0.36 kg ai/ha) methamidophos, were harvested 3 days after treatment and cooked for 20 minutes in either a closed or open vessel before being analyzed. Residue concentrations were reduced by about 20% (Mölhoff, 1978).

In a study conducted in Singapore, Chinese cabbages (*Brassica chinensis*) were treated in the field with weekly sprays of 0.1% or 0.2% methamidophos and harvested 1 and 3 days after the last treatment. The harvested Chinese cabbages were finely chopped and 50 g samples were either rinsed in a sieve for one minute or soaked in 200 ml cold water. Samples of raw, rinsed and soaked cabbage were also boiled for 2 minutes in 200 ml water (Ong *et al.,* 1988). Processing factors of 0.3-0.4 were estimated for the soaked raw cabbage and up to 0.1 for the boiled Chinese cabbage.

A study in India investigated the effect of washing and cooking on cabbage heads and cauliflower curds (Dikshit *et al.,* 1986). Field-treated crops, sprayed once with 0.33 kg ai/ha or 0.55 kg ai/1100 litres/ha methamidophos, were sampled at 0 and 7 days after treatment. Samples (100 g) of cabbage heads and cauliflower curds were washed for 1 minute in cold water or chopped and boiled for 20 minutes in a covered container (Table 50). In the washed cabbage, processing factors of 0.24-0.34 were calculated in the day 0 samples, while in the day 7 samples a lower residue reduction was observed (processing factors of 0.7-0.8). Boiling for 20 minutes significantly reduced the levels of methamidophos residues in all samples, with processing factors of about 0.1-0.2 for both cabbages and cauliflowers.

Table 50. Methamidophos residues in raw, washed and boiled cabbage and cauliflower resulting from processing studies in India.

COMMODITY Location, year (variety), reference	Application				PHI days	Residues, mg/kg methamidophos	Processing factor
	Form	kg ai/ha	water, l/ha	no.			
CABBAGES							
India, 1981, Dikshit *et al,.* 1986	EC	0.33	1100	1	0	4.1	
						washed 1.0	0.24
						boiled 0.54	0.13
					7	1.0	
						washed 0.76	0.76
						boiled 0.24	0.24
		0.55	1100	1	0	6.3	
						washed 2.1	0.33
						boiled 0.7	0.11
					7	2.1	
						washed 1.48	0.7
						boiled 0.38	0.18

COMMODITY Location, year (variety), reference	Application				PHI days	Residues, mg/kg methamidophos	Processing factor
	Form	kg ai/ha	water, l/ha	no.			
CAULIFLOWERS							
India, 1981, Dikshit *et al*,. 1986	EC	0.33	1100	1	0	curds 4.60	
						washed curds 1.35	0.29
						boiled curds 0.62	0.13
					7	curds 1.0	
						washed curds 0.8	0.8
						boiled curds 0.12	0.12
		0.55	1100		0	curds 7.85	
						washed curds 2.14	0.34
						boiled curds 1.0	0.13
					7	curds 2.45	
						washed curds 1.4	0.76
						boiled curds 0.32	0.17

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating cows

Lactating Holstein dairy cows (4-5 years old, 400-565 kg bw) were administered methamidophos and metribuzin, twice daily for 28 days, by bolus at dose rates of 0.006/0.3, 0.03/0.09 and 0.15/0.03 mg/kg bw/day, equivalent to nominal feeding rates of 0.2, 1.0 and 5.0 ppm for methamidophos (Anonymous, 1972b and 1972c). Samples of milk were collected after 27 and 28 days of dosing and the animals were slaughtered after 28 days dosing and samples of tissues collected for analysis using analytical method 31093 (Table 51). Milk production by individual animals during the course of the study ranged from 5.2 to 22 kg/day.

Residues in tissues were all <LOQ at the end of the dosing period. Low levels of methamidophos were detected in milk, but only from the highest dose group.

Table 51. Residues of methamidophos in tissues and milk of lactating dairy cows orally dosed with methamidophos and metribuzin for 28 consecutive days, (Anonymous,1972b and 1972c).

Sample	Days after start of feeding§	Methamidophos residue (mg/kg)		
		0.2 ppm	1.0 ppm	5.0 ppm
Back fat	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Renal fat	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Omental fat	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Loin steak	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Round steak	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Flank steak	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Liver	28	<0.07 (3) [1/]	<0.07 (3) [1/]	<0.07 (3) [1/]
Kidney	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Heart	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Brain	28	<0.01 (3)	<0.01 (3)	<0.01 (3)
Milk	27	<0.001 (3)	<0.001, <0.001, 0.002	0.008, 0.008, 0.004
	28	<0.001, <0.001, 0.001	<0.001, <0.001, 0.001	0.019, 0.021, 0.021

[1/] Estimated LOQ based on the rate of decomposition of residues in liver with frozen storage and the storage interval.

Methamidophos residues in liver are not stable during storage in the freezer. To supplement the above study and obtain information on residues in liver, calves (Hereford and Angus, 313-368 kg bw) were orally dosed by bolus with methamidophos and metribuzin, at 0.3/0.09 or 0.6/0.3 mg/kg bw for 30-32 days, equivalent to nominal feeding rates of 10 and 20 ppm methamidophos, respectively (Anonymous 1975f 43803). At the end of the dosing period the calves were slaughtered and samples of liver collected for analysis, which was conducted within 1-5 days of sample collection. Residues

of methamidophos in liver were <0.01 (3) for the 10 ppm dose group and <0.01, <0.01 and 0.03 mg/kg for the 20 ppm dose group.

Laying hens

Laying hens (1.359-1.899 kg bw) were fed methamidophos at 2, 6 and 20 ppm in the diet for 28 days (Ackerman *et al.,* 1975b 43787). The feed consumption prior to commencement of dosing was 72 to 101 g per day. Feed was prepared by adding an ethanolic solution of methamidophos to corn oil and adding this to the layer ration. Batches of feed were prepared weekly. Eggs were collected daily and chickens from each dose group were slaughtered on completion of the trial. Samples of muscle (composited light and dark), fat (composited subcutaneous and visceral), heart and gizzard (composite heart + gizzard), liver and kidney were collected at slaughter for analysis by GC-FPD (method 31093). Because of the low levels of residues detected in the tissues of birds exposed to the 20 ppm feeding level, analysis of tissues from the lower dose groups was not carried out (Table 52).

The highest residue levels were observed in eggs, which reached a plateau level by *ca.* 7 days of dosing. Of the tissue samples, methamidophos residues were highest in muscle, heart/gizzard and skin with only low levels detected in fat and liver.

Table 52. Residues of methamidophos in tissues and eggs of laying hens (composite samples) fed methamidophos at 2, 6 and 20 ppm in the diet for 28 consecutive days, (Ackerman *et al.,* 1975b).

Tissue	Residue (mg/kg) [1/]			
	2 ppm	6 ppm	20 ppm	20 ppm, mean of composite samples
Fat			0.002 (3)	0.002
Liver			0.002, 0.002, 0.004	0.003
Kidney			0.004 (3)	0.004
Skin			0.018, 0.018, 0.017	0.018
Hearts and gizzards			0.022, 0.019, 0.021	0.021
Muscle			0.046, 0.041, 0.033	0.040
Eggs				
3 days			0.098, 0.068, 0.106	0.091
7 days			0.086, 0.098, 0.112	0.099
14 days			0.094, 0.138, 0.134	0.122
28 days	0.006, 0.005, 0.005	0.017, 0.020, 0.024	0.103, 0.095, 0.111	0.103

[1/] Replicate analyses of composite tissue samples.

NATIONAL RESIDUE LIMITS

The Meeting was aware of the national MRLs shown in Table 53.

Table 53. National MRLs for methamidophos.

Country	MRL, mg/kg	Commodity
Argentina	5.0	hops (dry)
	2.0	clover forage
	1.0	sugar beet (tops or leaves)
	0.5	citrus, cucurbits, melon, peppers (sweet), tobacco, tomatoes
	0.2	leafy & other stem vegetables
	0.1	alfalfa forage, alfalfa seed, almonds, beans, beans (climbing French), cotton seed, garlic, grapes, pome fruit, potatoes, rape seed, soya beans, stone fruit, sunflowers
	0.05	cereals, rice, sugar beet

Country	MRL, mg/kg	Commodity
Australia	5.0	hops (dry)
	2.0	celery, pepper (sweet), tomatoes
	1.0	brassica vegetables, egg plants, lettuce (head), lettuce (leaf), peaches
	1.0 T	other leafy vegetables
	0.5	citrus fruit, cucumbers, lupins (dry)
	0.25	potatoes
	0.2	bananas (dwarf), bananas
	0.1	cotton seed, rape seed, soya beans (dry)
	0.05	sugar beet
	0.02 *	peanuts
	0.01 *	meat (mammalian), edible offal (mammalian), milks, tree tomato
Austria	2.0	hops
	1.0	cucumbers
	0.5	beans (with pod), cabbages (head), flowering brassicas, garden peas (with pod), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce (head)
	0.1	apricots, artichokes, cotton seed, grapes, tea
	0.05	peaches
	0.05	pome fruit
	0.01	all food of animal origin
Belgium	2.0	hops
	1.0	cucumbers
	0.5	Brussels sprouts, cabbages (head), flowering brassicas, legume vegetables (with pod), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce (head)
	0.1	apricots, artichokes, cotton seed
	0.1 *	tea
	0.05	peaches, pome fruit
	0.01 *	all food of animal origin, other plant commodities
Brazil	0.01	beans, soya beans
	0.1	cotton seed, peanust, potatoes, wheat
	0.3	tomatoes
Canada	1.0	broccoli, Brussels sprouts, lettuce, peppers
	0.5	cabbages, cauliflowers, celery, cucumbers, egg plants, tomatoes
	0.3	beans
Chile	2.0	lettuce (head), tomatoes
	1.0	peaches
	0.1	beet (sugar), potatoes, rape
	0.01 *	cattle fat, cattle meat, goat fat, goat meat, milk, sheep fat, sheep meat
Croatia	0.1	cereals, fruit, potatoes, sugar beet
	0.01	all food of animal origin,
Cyprus	2.0	cauliflowers, celery, tomatoes
	1.0	beet (leaves), cabbages, cucumbers, egg plants, lettuce, peaches, peppers (cayenne), peppers (sweet)
	0.5	citrus
	0.1	beet (roots), potatoes
	0.01	meat, milk

Country	MRL, mg/kg	Commodity
Denmark	2.0	hops
	1.0	cucumbers
	0.5	beans (with pod), cabbages (head), flowering brassicas, garden peas (with pod), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce
	0.1	apricots, artichokes, cotton seed
	0.05	nectarines, peaches, pome fruit
	0.01	grapes, leafy brassicas, strawberries
	0.02 *	egg products, eggs (without shell), milk, milk products
	0.01 *	beans (without pod), bulb vegetables, cane fruit (*Rubus* spp), cereals, fat, garden peas (without pod), herbs, kohlrabi, meat (preparations of), meat by-products, meat, mushrooms, nuts, other berries & small fruit, other fruiting vegetables, other leafy vegetables, other oil seeds, other stem vegetables, other stone fruit, potatoes, pulses, root & tuber vegetables, tea, tropical & subtropical fruits
European Community	2.0	hops (dry)
	1.0	cucumbers
	0.5	head brassicas, flowering brassicas, beans (with pods), peas (with pods), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce
	0.1	apricots, artichokes, cotton seed, tea
	0.05	nectarines, peaches, pome fruit
	0.01 *	cereals, other fruit, other vegetables, meat, edible offal, fat, dairy products, eggs
Finland	0.2	fruit, vegetables
France	2.0	hops
	1.0	egg plants
	0.5	apricots, cereals, peaches, tomatoes
	0.3	grapes, other stone fruit, pome fruit
	0.2	cabbages (head), cotton, lettuce
	0.1	cucumbers, tea
	0.01	citrus, other fruit, other oil seeds, other vegetables, potatoes
Germany	2.0	hops
	1.0	cucumbers
	0.5	cabbages (head), flowering brassicas, legume vegetables (with pods), spearmint, tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce
	0.1	apricots, artichokes, cotton seed, tea, tea-like products
	0.05	peaches, pome fruit
	0.01	eggs, gherkin, meat. meat (preparations of), milk, milk products, other plant commodities
India	0.1	cotton seed, safflower seed
Israel	2.0	alfalfa, celery, clover, vetch
	1.0	brassica vegetables, celeriac, cucumbers, peppers, squash, tomatoes
	0.5	melons
	0.1	cucumbers, cotton seed, garlic, onions (bulb), potatoes, peanuts
	0.05	beetroot, fodder beet, chickpeas, sugar beet
	0.01	fat, meat, milk
Italy	2.0	hops
	1.0	cucumbers
	0.5	cabbages (head), flowering brassicas, legume vegetables (with pods), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce
	0.15	sugar beet, tobacco
	0.1	apricots, artichokes, cotton seed, tea
	0.05	peaches, pome fruit
	0.01	cereals, other fruit, other oil seeds, other vegetables, potatoes, pulses
	0.1 *	eggs, egg products
	0.01 *	mammalian meat, mammalian fats, mammalian meat by-products, meat preparations, milk, milk products

Country	MRL, mg/kg	Commodity
Japan	5.0	hops
	2.0	tomatoes, peppers (sweet), other *Solanaceae*
	1.0	broccoli, Brussels sprouts, cabbages, cauliflowers, cucumbesr, egg plants, gherkins, lettuce, peaches
	0.5	melons
	0.25	potatoes
	0.1	cotton seed, other fruit, rape seed
	0.05	soya beans (dry), sugar beet
Korea	5.0	hops
	2.0	tomatoes, peppers (sweet)
	1.0	Korean cabbages, cauliflowers, cucumbers, celery, egg plants, green and red peppers, lettuce, Korean lettuce, peaches
	0.5	citrus, melons
	0.1	cotton seed, other fruit, other seeds
	0.05	potatoes, soya beans
Luxembourg	2.0	hops
	1.0	cucumbers, gherkins
	0.5	cabbages (head), tomatoes
	0.2	citrus, egg plants, lettuce (head)
	0.1	cotton seed, flowering brassicas
	0.01 *	cereals, eggs, egg products, fat,
	0.01 *	eggs, egg products, fat, meat, meat by-products, meat (preparations of), milk, milk products, other plant commodities, tea
Malaysia	1.0	cayenne peppers, cucumbers, egg plants, peppers (sweet), tomatoes
	0.5	citrus, watermelons
	0.25	peaches
	0.1	edible fat & oil, potatoes
Mexico	2.0	alfalfa
	1.0	broccoli, Brussels sprouts, cabbages, capsicums (peppers/chilli), cauliflowers, celery, cucumbers, egg plants, lettuce (head), melons, tomatoes
	0.5	potatoes
	0.1	cotton, soya beans
Netherlands	2.0	hops
	1.0	cucumbers
	0.5	beans (with pods), brassicas (head), flowering brassicas, peas (with pods), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce
	0.1	apricots, artichokes, cotton seed
	0.05	nectarines, peaches, pome fruit
	0.01 *	other food, tea
New Zealand	1.0	brassica vegetables
	0.5	citrus, leafy vegetables
	0.2	fruiting vegetables (except tomatoes)
	0.1	potatoes, tomatoes, other food
Poland	5.0	hops
	0.2	citrus, leafy & stem vegetables
	0.1	other vegetables, tea
	0.02	other fruit
	0.01	cereal grain, eggs (without shell), milk, milk products, potato
	0.01	meat, meat (preparations of)
Portugal	0.5	broccoli, cabbages (red, white, head, Savoy), flowering brassicas, peaches, tomatoes
	0.01 *	potatoes
South Africa	5.0	tobacco
	1.0	*Cruciferae*, mangoes, nectarines, peaches
	0.5	tomatoes
	0.2	citrus, potatoes

Country	MRL, mg/kg	Commodity
Spain	2.0	hops
	1.0	cucumbers, peppers (sweet)
	0.5	cabbages, tomatoes
	0.2	artichokes, citrus, egg plants, lettuce, peas (pods and/or immature seeds), pome fruit, stone fruit
	0.1	beans (pods and/or immature seeds), cotton seed, tea, tea plants (infusion)
	0.05	leeks, sugar beet
	0.01	beans (dry), berries (wild), bulb vegetables, cacao, cane fruit (*Rubus* spp), cereals, coffee, cola, flowering brassicas, forage crops & straw, fruit & vegetables (dried), grapes, herbs, kohlrabi, leafy brassicas, mushrooms, nuts, berries & small fruits, cucurbits (inedible peel), other leafy vegetables, other legume vegetables, other oil seeds, pulses, other *Solanaceae*, other stem vegetables, peas (dried), potatoes, root & tuber vegetables, spices, spinach & similar, strawberries, sugar cane, sweet corn, tobacco, tropical & subtropical fruits, watercress, witloof chicory
Sri Lanka	1.0	beans, beet, cabbages, cowpeas
	0.1	potatoes
Sweden	2.0	hops
	1.0	cucumbers
	0.5	beans (with pod), cabbagse (head), flowering brassicas, garden peas (with pod), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce
	0.1	apricots, artichokes, cotton seed
	0.05	nectarines, peaches, pome fruit
	0.1 *	tea
	0.01 *	cereals, eggs, egg products, meat, meat by-products, meat (preparations of), milk, milk products, other fruit, other oil seeds, other vegetables, potatoes, pulses
Switzerland	1.0	cucumbers
	0.5	Brussels sprouts, cabbages (head), tomatoes
	0.3	plums
	0.2	citrus, egg plants, lettuce (head)
	0.1	apricots, cotton seed, grapes, other vegetables, tea
	0.05	pome fruit, peaches
	0.01	cereals, other fruit, root and tuber vegetables
Taiwan	0.5	citrus, fruiting vegetables (tomatoes, egg plants, sweet peppers, etc.), leaf vegetables (cabbages, cauliflowers, Chinese cabbages, broccoli, lettuce, Brussels sprouts, mustard, Chinese mustard, Chinese kale, celery, water spinach, spinach, lettuce, garland chrysanthemum, leaf-beet, garlic, spring onions, Chinese leeks, etc.), melon vegetables (cucumbers, bitter melons, luffa, wax gourds, pumpkins, vegetable pears, etc.), peas and beans (snap beans, snow peas, vegetable soy beans, lablab, asparagus beans, kidney beans, etc.), pome (apples, pears, peaches, plums, Japanese apricots, cherries, jujubes, persimmons, etc.)
	0.2	drupes (mangoes, longans, litchis, loquats, etc.)
	0.1	rice, other cereals and crops (corn, sorghum, sweet potato, etc.), root vegetables (radishes, carrots, ginger, onions, potatoes, bamboo shoots, asparagus, co-ba, taro, etc.).
	0.03	beans (dry)
Uruguay	0.1	tomatoes
UK	2.0	hops
	1.0	cucumbers
	0.5	Brussels sprouts, cabbages (head), tomatoes
	0.2	egg plants, grapefruit, lemons, lettuce, limes (sour), mandarins, oranges, other citrus, pommelos
	0.1	cotton seed
	0.1 *	tea
	0.01 *	berries & small fruits, other brassica vegetables. bulb vegetables, cereals, eggs, herbs, meat (preparation of), milk, mushrooms, mustard (seed), nuts, oil seeds, other cucurbits, other leafy vegetables, root & tuber vegetables, tropical & subtropical fruit, pulses, other *Solanaceae*, stem vegetables, sweet corn

Country	MRL, mg/kg	Commodity
USA	1.0	broccoli, Brussels sprouts, cabbages, cauliflowers, cucumbers, egg plants, lettuce, peppers, tomatoes
	0.5	melons
	0.1 *	cotton seed, potatoes

* Indicates lower limit of analytical determination.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Monitoring data

Recent data on monitoring of methamidophos in national residue monitoring programmes in Australia, the UK and in the European Community were provided.

In the Australian Residue Survey of 2001-2002, pecan nuts (45 samples) and macadamia nuts (134 samples) were tested for residues using a multi-residue method. No methamidophos residues were detected in any of the samples tested. Reporting limits were 0.05 mg/kg for macadamia nuts and 0.1 mg/kg for pecans.

The results of residue monitoring conducted in the UK during 2001, as part of the coordinated EU residue monitoring programme, are summarized in Table 54.

Table 54. Results for methamidophos from the UK 2001 pesticide pesidue monitoring programme.

Commodity Year (months)	Samples analysed	Samples with detects	Samples <LOR [1/]	Samples with residues in range, mg/kg				
				≤0.01	>0.01 ≤0.02	>0.02 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.2
Grapes, 2001 (Jan-Sep)	54	0	54					
Star fruit, 2001 (Apr-Aug)	25	0	25					
Kiwifruit, 2001 (Jan-Sep)	47	0	47					
Mangoes, 2001 (May-Aug)	36	0	36					
Peaches, 2001 (Apr-Jun)	19	1	18			1		
Nectarines, 2001 (Apr-Jun)	17	1	16				1	
Cows' milk, 2001 (Jul-Sep)	51	0	51					
Tea, 2001 (Aug-Sep)	48	0	48					
Pizzas, 2001 (Apr-Aug)	24	0	24					

[1/] LOR is limit of reporting (0.05 mg/kg for tea and 0.01 mg/kg for the other commodities).

The EU co-ordinated residue monitoring programme for 2000, covering rice, cucumbers, head cabbage and peas, included methamidophos as one of the 20 pesticides analyzed in the programme and the results are summarized in Table 55.

Table 55. Results for methamidophos from the EU co-ordinated 2000 residue monitoring programme.

Commodity, year	samples analyzed	samples with detects	samples <LOR [1/]	Samples with residues in range, mg/kg		
				≤1.0	>1.0	Maximum residue (mg/kg)
Rice, 2000	869	0	869			
Cucumber, 2000	1176	24	1152	24		0.39
Cabbages, head, 2000	962	0	962			
Peas, 2000	730	0	730			

[1/] LOR is limit of reporting.

Monitoring data from the USA, extracted from the USDA Pesticides Data Program (PDP), for potatoes and tomatoes in the period 1996 to 1998, and from the US FDA Surveillance Monitoring

Program (SMP), for imported peppers, tomatoes, squash and strawberries for the period 1996 to 1998, are summarized in Table 56.

Table 56. Results for methamidophos from USA residue monitoring programmes (USDA, FDA).

Commodity year (months)	samples analyzed	samples with detects	samples <LOR [1/]	Samples with residues in range, mg/kg					
				≤0.01	>0.01 ≤0.02	>0.02 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.2	>0.2
Peppers, bell, FDA-I 1996-98 [2/]	598	142	456	8	7	33	28	39	27
Potatoes, PDP 1994-95 [3/]	1401	19	1391	15	1	3			
Peppers (non bell), FDA-I 1996-98 [2/]	717	242	475	19	13	54	52	58	46
Squash, FDA-I 1996-98 [2/]	388	11	377	6	1	1	3		
Strawberry, FDA-I 1996-98 [2/]	157	13	144	9	3				1
Tomato, PDP 1996-97 [3/]	884	282	602	82	63	75	36	19	7
Tomato, canned, PDP 1999 [3/]	310	58	252	52	5	1			

[1/] LOR is limit of reporting (0.01 mg/kg).

[2/] US FDA import surveillance residue monitoring programme.

[3/] USDA Pesticide Data Program.

Eight market basket surveys were conducted in the USA during 1984 and 1985, with 26-62 commodities being collected and analyzed in each quarterly survey. The edible portions of each commodity from each location per survey were combined and analyzed for residues of acephate and methamidophos (LOD 0.01-0.02 mg/kg). Detectable methamidophos residues were found in cantaloupe, celery, cucumbers and crisphead lettuce, at levels of 0.1 mg/kg or less, and in tomatoes and sweet peppers at levels of up to 0.26 mg/kg. No residues of methamidophos were detected in any commercially processed food, except canned snap beans, where a maximum of 0.01 mg/kg methamidophos was recorded.

APPRAISAL

Methamidophos was evaluated initially in 1976 for residues and toxicology and the latest evaluation for residues was in 1997. It was identified as a priority compound under the Periodic Review Programme of the 29th Session of the CCPR for review by the 2002 JMPR (ALINORM 97/24A). At the 31st Session of the CCPR, the Committee noted that an acute RfD would be established by the 2000 JMPR but this was finally established only at the 2002 Meeting. The present Meeting received information on methamidophos metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials, farm animal feeding studies, fate of residues in processing and national MRLs. Some information on GAP, national MRLs and residue trials was reported by the governments of Australia, Germany and The Netherlands.

The 2002 JMPR established an ADI and acute RfD for methamidophos of 0-0.004 mg/kg bw and 0.01 mg/kg bw respectively.

Methamidophos is a broad-spectrum organophosphorus insecticide with uses on many crops. It is also formed as a metabolite of the insecticide acephate.

Animal metabolism

The Meeting received reports on animal metabolism studies of methamidophos in lactating goats and laying hens.

Methamidophos is rapidly metabolised and is not identified as a component of the residue in ruminant (goat) kidney, liver, muscle or fat and only at low levels in milk. In laying hens, low levels of methamidophos are detected in liver and eggs.

In three lactating goat metabolism studies with [*S*-methyl-^{14}C]methamidophos daily doses were administered in one, two or three parts and the interval between the last dose and slaughter ranged from 3 h to 11 days. Generally, methamidophos accounted for <3% of the ^{14}C in milk, with the majority of radioactivity incorporated into lactose, proteins/amino acids and triglycerides. In tissues, most of the ^{14}C was incorporated into natural products: proteins, and amino acids in kidney, liver and muscle; and triglycerides in fat. Methamidophos was not detected in any tissues and only low levels of the metabolites DMPT and SMPAA were detected in kidney but not in other tissues or milk.

In laying hens administered [*S*-methyl-^{14}C]methamidophos, the parent compound was identified as a minor component of the ^{14}C residues in liver (<1%) and represented <7% of the ^{14}C residue in egg white and yolk. Natural products (lipids, proteins and amino acids) accounted for most of the ^{14}C with only trace amounts of the metabolites SMPAA and DMPT.

Plant metabolism

The Meeting received plant metabolism studies of methamidophos on tissue cultures of sweet potatoes and tobacco, glasshouse grown bean, cabbage, lettuce and tomato plants as well as outdoor grown lettuce, potato and tobacco plants.

Radioactivity in glasshouse grown cabbage and tomato plants was mostly associated with natural products, mainly lipids, pigments and amino acids, although trace amounts of the metabolite DMPT were detected. Most of the residue on exposed foliage of tomato and lettuce plants was methamidophos. Methamidophos is systemic.

Comprehensive studies of the nature of the residue followed the foliar application of [*S*-methyl-^{14}C]methamidophos to lettuce and potato plants. Most of the ^{14}C residue in lettuce leaves harvested 21 days after application was as methamidophos (about 66%). A minor metabolite, a conjugate of *S*-methyl phosphorothiolate, was detected at <2% of the ^{14}C, with the remainder associated with natural products, principally sugars and starch.

Radioactivity in potato tubers, harvested 14 days after the last of four foliar sprays with [*S*-methyl-^{14}C]methamidophos, was essentially accounted for by incorporation into starch and other natural products; methamidophos represented only 0.2% of the total radioactive residue.

In a comparison of the decline of labelled and unlabelled methamidophos residues in field and glasshouse grown tobacco plants, methamidophos was also the major fraction of ^{14}C in tobacco leaves (glasshouse). The decline in methamidophos residues was more rapid in the field (half-life 5 days) than in the glasshouse (half-life 15 days).

In both animals and plants, methamidophos undergoes hydrolysis of the ester and thioester moieties, liberating one-carbon fragments to the general metabolic pool for incorporation into natural products. The main identified residue component is methamidophos *per se*.

Environmental fate in soil

Information was provided on the soil adsorption of methamidophos and its fate during soil photolysis, aerobic and anaerobic soil degradation, the column leaching of aged residues and field dissipation.

Methamidophos degradation on soil surfaces by photolysis occurred with a half-life of about 60 h. The major product was SMPAA with small amounts of DMPT also observed.

The half-lives of methamidophos in soil under aerobic test conditions were estimated to be ≤6 days in the laboratory and ≤3 days in the field. Degradation occurred by hydrolysis of ester and amino groups to form SMPAA and DMPT. The principal mechanism of degradation appears to be microbial metabolism.

Methamidophos and DMPT are only very weakly adsorbed by soils and can be classified as mobile. The rate of degradation both in the laboratory and in the field is such that residues are not expected to persist at detectable levels for more than a few days. Methamidophos is not persistent.

Environmental fate in water-sediment systems

The Meeting received information on the sterile aqueous hydrolysis of methamidophos and its fate in water-sediment systems.

Methamidophos is stable to hydrolysis at low pH but is readily degraded at neutral and high pH (half-life 27 and 3.2 days at pH 7 and 9 respectively).

The half-life for the degradation of methamidophos in the water-sediment systems was estimated to be 4-6 days.

In summary, chemical hydrolysis is only expected to occur in waters having high pH values. Indirect photochemical transformation of methamidophos is expected to occur but is considered to be only a minor route of degradation. Biodegradation in the aquatic environments is expected to be rapid, so that methamidophos is not expected to persist in the environment.

Analytical methods

Samples in field trials were analysed for methamidophos by solvent extraction (ethyl acetate, acetone/water and, in the case of oily crops and animal commodities, acetonitrile/hexane), clean-up by solvent partition and/or silica gel or gel permeation chromatography, and determination by gas chromatography with a thermionic detector. A typical LOQ was 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of methamidophos in various commodities under freezer storage (-20°C). Residues were stable in or on the following commodities (storage period in parentheses): broccoli (9 months); lettuce (2 months); cabbages (8 months); cauliflowers (6 months); Brussels sprouts (5 months); celery, peppers, peanut forage (26 months); sorghum grain (6 months); sorghum forage (10 months); rape seed, potato tubers, granules and dry peel, tomato fruit, purée and dry pomace (24 months); bovine milk, meat and fat, eggs (3 months) and poultry liver and heart/gizzard (2 months).

Methamidophos residues were not stable in cattle liver, with only 26% of the residues remaining after 3 months frozen storage.

Residue definition

Methamidophos is the principal component of residues in crops. Significant residues of methamidophos and metabolites were not observed in animal commodities. The Meeting agreed that the residue should be defined as methamidophos.

The log P_{ow} of –0.8 and the animal metabolism and feeding studies suggest that methamidophos should not be described as fat-soluble.

Definition of methamidophos residue (for compliance with MRLs and for estimation of dietary intake): *methamidophos*.

The definition applies to plant and animal commodities. Methamidophos residues may arise from the use of methamidophos and/or acephate.

Supervised trials

Supervised trials were reported for the use of methamidophos on broccoli, cabbages, cauliflowers, cotton seed, fodder beet, maize, nectarines, peaches, peppers (including chilli peppers), potatoes, soya beans, sugar beet and tomatoes.

Trials data or relevant GAP were not reported for the following crops with current CXLs: alfalfa forage, green (2 mg/kg), head lettuce, (1 mg/kg), and tree tomato (0.01 mg/kg). The Meeting agreed to recommend withdrawal of the CXLs for these commodities.

In cases where maximum residue levels have been estimated for acephate, it is also necessary to ensure that the resulting methamidophos residues are covered by a maximum residue level estimate for methamidophos. Residues of methamidophos arising from the use of acephate, and derived from

the trials used to estimate the maximum residue, STMR and HR levels for acephate, are reported below.

Citrus fruits. Methamidophos residues arising from the use of acephate on mandarins in rank order (median underlined) were 0.02, 0.03, 0.04, 0.05, 0.06, 0.08, 0.08, 0.09, 0.09, 0.13, 0.14, 0.15, 0.25 and 0.26 mg/kg. The Meeting estimated a maximum residue level, STMR and HR for methamidophos in mandarins of 0.5, 0.085 and 0.26 mg/kg, all based on whole fruit as insufficient information was available to estimate residues in the edible portion.

Pome fruits. Methamidophos residues in apples and pears from the use of acephate (n=13) were <0.1, <0.1, 0.03, 0.03, 0.04, 0.04, 0.06, 0.06, 0.13, 0.14, 0.16, 0.22 and 0.28 mg/kg. The Meeting estimated a maximum residue level, STMR and HR for methamidophos in pome fruits of 0.5, 0.06 and 0.28 mg/kg.

Stone fruits. Methamidophos trials on nectarines were conducted in Italy (GAP for peaches 0.03-0.06 kg ai/hl, maximum 2 sprays, PHI 35 days) and Portugal (GAP for peaches 0.6 kg ai/ha, 0.06 kg ai/hl, maximum 1 spray, PHI 35 days). No trials matched GAP, as either the application rates were too low or samples were not collected after an appropriate interval.

Data reported from supervised trials on peaches in France (GAP 0.05 kg ai/hl, PHI 14-21 days), Italy, Portugal and Spain (GAP 0.05-0.08 kg ai/hl, PHI petal fall + 10 days). The trials in France and Spain did not match GAP in these countries. The Meeting decided to evaluate the trials in Spain according to the GAP of Italy and Portugal.

Four trials in Italy approximated Italian GAP, with methamidophos residues of 0.04, 0.06, 0.11 and 0.13 mg/kg, all on a pulp basis (calculated whole fruit residues 0.04, 0.05, 0.11 and 0.12 mg/kg) 28 days after application at 0.05-0.06 kg ai/hl. A further six trials in Spain matched GAP in Italy and/or Portugal and showed residues of 0.01, 0.02, 0.03, 0.04, 0.09 and 0.15 mg/kg.

The residues in peaches from trials with methamidophos according to GAP were 0.01, 0.02, 0.03, 0.04, 0.04, 0.05, 0.09, 0.11, 0.12 and 0.15 mg/kg. Residues of methamidophos in peaches arising from the use of acephate according to Greek GAP were 0.09, 0.1, 0.16, 0.22, 0.28 and 0.35 mg/kg and, as this use gave higher residues than the use of methamidophos, the Meeting used the acephate data to estimate a maximum residue level, STMR and HR.

The Meeting also considered that the residues of methamidophos on peaches and nectarines treated at the same rate would be similar and noted that GAP for the use of acephate in Greece was for stone fruit which includes both peaches and nectarines. The Meeting estimated a maximum residue level, STMR and HR for peaches and nectarines of 0.5, 0.19 and 0.35 mg/kg and recommended withdrawal of the draft MRL of 1 mg/kg for peaches.

Brassica vegetables. Trials were reported from Belgium (no GAP), Canada (GAP 0.53-1.1 kg ai/ha, PHI 7 days for cauliflower, 14 days for broccoli), Germany (GAP 0.36 kg ai/ha, PHI 21 days), and the UK (no GAP) on broccoli and cauliflowers.

No trials on broccoli matched GAP. Residues in eight trials on cauliflower from Germany approximating GAP in that country were <0.01 (5), 0.01 (2) and 0.04 mg/kg while the residue of methamidophos in one trial in Canada approximating Canadian GAP was 0.08 mg/kg.

The residues in trials according to GAP in Canada and Germany appeared to be from different populations and could not be combined for estimating a maximum residue level. Residues in cauliflowers complying with GAP in Germany in rank order, median underlined, were ≤0.01 (5), 0.01 (2) and 0.04 mg/kg.

Residues of methamidophos in broccoli and cauliflowers from the use of acephate (n=14) were <0.01(5), 0.01, 0.01, 0.03, 0.03, 0.08, 0.09, 0.1, 0.2 and 0.33 mg/kg, giving a higher maximum residue level than the trials with methamidophos. The Meeting used the acephate data to estimate a maximum residue level, STMR and HR for methamidophos in flower head brassicas of 0.5 mg/kg, 0.02 mg/kg and 0.33 mg/kg respectively. The estimated maximum residue level of 0.5 mg/kg is recommended to replace the existing CXL of 0.5 mg/kg for cauliflower.

Trials on head cabbages were reported from Canada (GAP 0.53-1.1 kg ai/ha, PHI 7 days) and Germany (GAP 0.36 kg ai/ha, PHI 14 days).

Residues in head cabbages in three trials in Canada approximating GAP in that country were 0.04, 0.60 and 0.62 mg/kg. Residues in eight trials on head cabbages in Germany, approximating German GAP, were <0.01 (2), 0.01, 0.03, 0.04, 0.07, 0.09 and 0.20 mg/kg.

The residues evaluated according to the GAP of Canada and Germany appeared to be from the same population and were combined to estimate a maximum residue level. The residues in rank order, median underlined (n=11) were <0.01 (2), 0.01, 0.03, 0.04, 0.04, 0.07, 0.09, 0.20, 0.60 and 0.62 mg/kg.

The Meeting estimated a maximum residue level, STMR and HR for methamidophos in head cabbages of 1 mg/kg, 0.04 mg/kg and 0.62 mg/kg respectively. The maximum residue level of 1 mg/kg is recommended to replace the existing CXL of 0.5 mg/kg.

Tomatoes. Trials on tomatoes with methamidophos were reported from Brazil (no GAP), France (no GAP), Germany (no GAP), Greece (GAP 0.6-0.9 kg ai/ha, PHI 21 days), Italy (no GAP), Mexico (GAP 0.6-0.9 kg ai/ha, PHI 7 days), Spain (no GAP), Turkey (no GAP) and the USA (GAP 0.84-1.1 kg ai/ha, maximum 5.6 kg ai/ha/season, PHI 7 days).

Methamidophos residues in 18 trials in the USA matching GAP, ± 30%, in rank order, median underlined, were 0.05, 0.08, 0.12, 0.14, 0.14, 0.16, 0.22, 0.24, 0.26, 0.31, 0.36, 0.36, 0.42, 0.56, 0.86, 1.3, 1.4 and 1.5 mg/kg.

The Meeting estimated a maximum residue level, STMR and HR for methamidophos in tomatoes of 2 mg/kg, 0.285 mg/kg and 1.5 mg/kg respectively. The maximum residue level is recommended to replace the draft MRL of 1 mg/kg for tomato.

Peppers. In Mexico, methamidophos is registered for use on peppers at 0.6-0.9 kg ai/ha with harvest permitted 14 days after the last application. In five trials in the USA matching those conditions, methamidophos residues on sweet peppers were 0.04, 0.07, 0.22, 0.38 and 0.95 mg/kg.

Methamidophos residues in peppers from the use of acephate (n=9) were 0.05, 0.22, 0.24, 0.25, 0.25, 0.29, 0.34, 0.35 and 1.6 mg/kg. As the acephate trials produced higher residues, the Meeting used the acephate data to estimate a maximum residue level, STMR and HR for methamidophos in peppers of 2, 0.25 and 1.6 mg/kg. The maximum residue level is recommended to replace the existing CXLs for chilli peppers of 2 mg/kg and sweet peppers of 1 mg/kg.

Common beans. Residues of methamidophos in beans, except broad beans and soya beans, arising from the use of acephate, in rank order median underlined (n=8), were 0.01, 0.04, 0.15, 0.15, 0.19, 0.34, 0.45 and 0.54 mg/kg.

The Meeting estimated a maximum residue level, STMR and HR for methamidophos in beans, except broad beans and soya beans, of 1, 0.17 and 0.54 mg/kg respectively.

Soya beans. Field trials were reported from Brazil (GAP 0.15-0.5 kg ai/ha, PHI 23 days). Residues in four trials approximating GAP were <0.01, <0.01, <0.04 and <0.04 mg/kg.

The Meeting decided that four trials were not sufficient to recommend a maximum residue level for such an important crop. However, the Meeting noted that, in an additional four trials conducted at twice the maximum application rate, residues were all below the LOQ (<0.01, <0.01, <0.04, <0.04 mg/kg). Those trials were considered as support for the trials complying with GAP.

Residues of methamidophos in soya beans, arising from the use of acephate, in rank order median underlined (n=7), were <0.01, <0.01, <0.01, ≤0.01, 0.02, 0.06 and 0.06 mg/kg. The Meeting noted that the residues from the use of acephate would lead to the higher estimates for a maximum residue level, STMR and HR for methamidophos in soya beans and estimated values of 0.1, 0.01 and 0.06 mg/kg respectively. The maximum residue level is recommended to replace the existing CXL of 0.05 mg/kg for soya beans (dry).

Potatoes. Field trials were reported from Canada (GAP 0.9-1.1 kg ai/ha, PHI 14 days), France (no GAP), Germany (GAP 0.5-0.6 kg ai/ha, PHI 14 days), Greece (GAP 0.045-0.09 kg ai/hl, PHI 21 days), Italy (GAP 0.57 kg ai/ha, PHI 21 days), Spain (no GAP) and the USA (GAP 0.84-1.1 kg ai/ha, maximum 4.5 kg ai/ha, PHI 14 days). The Meeting decided to evaluate the trials conducted in Spain against the GAP of Italy.

Twenty-nine trials in Germany matched GAP in that country, with residues in potatoes of <0.01 (25), 0.01, 0.01, 0.02 and 0.02 mg/kg. In two trials in Greece according to GAP, residues in potatoes were <0.01 mg/kg at 21 and 22 days after application and were also <0.01 mg/kg in two trials in Italy according to GAP, and in four trials in Spain approximating the GAP of Greece.

In 3 trials in Canada matching GAP, methamidophos residues in potatoes were all <0.01 mg/kg. In 23 trials in the USA matching GAP from that country the residues were <0.01 (5) and <0.05 (18) mg/kg.

The Meeting considered that, to estimate a maximum residue level, the trials could be considered to come from the same population. Residues in rank order, median underlined, were <u>≤0.01</u> (41), 0.01 (2), 0.02 (2) and <0.05 (18) mg/kg.

The Meeting estimated a maximum residue level, STMR and HR for methamidophos in potatoes of 0.05, 0.01 and 0.02 mg/kg respectively. The estimated maximum residue level of 0.05 mg/kg confirms the existing CXL.

Sugar beet and fodder beet. Field trials on sugar and fodder beet were reported from France (no GAP), Germany (GAP 0.36-0.48 kg ai/ha, PHI 28 days), Greece (no GAP), Italy (GAP 0.4-0.57 kg ai/ha, PHI 21 days) and Spain (no GAP). The Meeting evaluated the trials in France against the GAP of Germany.

Two trials in France and three in Germany matched GAP in Germany, with residues in sugar beet roots (only 4 results at GAP PHI) of <0.01 (3) and 0.01 mg/kg, and in tops of 0.9, 1.4, 1.5, 2.3 and 6.1 mg/kg. In six trials in Germany according to GAP, residues in fodder beet were all <0.01 mg/kg at 28 days after application and in tops 0.49, 0.54, 2.1, 2.8, 2.9 and 3.1 mg/kg. The Meeting agreed to combine the trials results for sugar and fodder beet to give a combined data set of <0.01 (9) and 0.01 mg/kg and estimated a maximum residue level, STMR and HR for methamidophos in sugar and fodder beet of 0.02, 0.01 and 0.01 mg/kg respectively. The maximum residue level of 0.02 mg/kg for sugar beet is recommended to replace the existing CXL of 0.05 mg/kg.

Residues in beet leaves or tops on a fresh weight basis from the combined trials approximating GAP and arranged in rank order, median underlined, were 0.49, 0.54, 0.9, 1.4, 1.5, <u>2.1</u>, 2.3, 2.8, 2.9, 3.1 and 6.1 mg/kg. Allowing for an average dry weight of 23%, the Meeting estimated a maximum residue level, an STMR and HR for methamidophos in sugar and fodder beet leaves or tops of 30, 9.1 and 26.5 mg/kg respectively on a dry weight basis. The estimate of a maximum residue level of 30 mg/kg for sugar beet leaves or tops is recommended to replace the existing CXL of 1 mg/kg.

Artichokes, globe. Methamidophos residues in globe artichokes from the use of acephate (n=4) were 0.02, 0.02, 0.04 and 0.08 mg/kg. The Meeting estimated a maximum residue level, STMR and HR for methamidophos in globe artichokes of 0.2, 0.03 and 0.08 mg/kg.

Maize. Field trials were reported from Germany (4 trials: no GAP), Greece (4 trials: GAP 0.6-0.8 kg ai/ha, PHI 21 days) and Spain (4 trials: GAP 0.05-0.08 kg ai/hl, PHI 35 days).

A single trial in Spain approximated GAP ± 25% for that country, with residues of <0.01 mg/kg in both the cob and grain. The Meeting agreed that the available data were insufficient to estimate a maximum residue level.

Cotton seed. Trials on cotton in Brazil (GAP 0.21-1.2 kg ai/ha, PHI 21 days), India (no GAP) and the USA (GAP 0.11-1.12 kg ai/ha, PHI 50 days) were reported to the Meeting.

In one trial in Brazil with 21 days PHI, methamidophos residues in cotton seed were below the LOQ (0.01 mg/kg).

In 15 US trials matching the GAP of the USA, residues in fuzzy seed, in rank order median underlined, were ≤0.01 (9), 0.01, 0.05, 0.06, 0.06, 0.09 and 0.16 mg/kg.

The Meeting decided that the Brazilian trial could be combined with the USA trials to estimate a maximum residue level, HR and STMR. Residues in cotton seed in rank order, median underlined, were ≤0.01 (10), 0.01, 0.05, 0.06, 0.06, 0.09 and 0.16 mg/kg.

Residues in cotton gin trash were 0.1, 0.2, 0.2, 0.35, 0.69, 0.85, 0.9, 1.5, 4.3 and 7.7 mg/kg.

The Meeting estimated a maximum residue level, STMR and HR for methamidophos in cotton seed of 0.2, 0.01 and 0.16 mg/kg respectively. The maximum residue level of 0.2 mg/kg for cotton seed is recommended to replace the existing CXL of 0.1 mg/kg.

Processing

Processing studies with methamidophos on apples, peaches, tomatoes, potatoes, soya beans, sugar beet and cotton seed were reported, together with a range of studies on the effects of washing, cooking or dehydration on residues in brassica vegetables, tomatoes, peppers and potatoes.

Two studies on peaches investigated residues in home-prepared jam as well as simulated commercially produced preserve and juice. With initial residue levels of 0.1 mg/kg, processing factors were estimated for washed peaches (0.64), jam (0.62), juice (0.33) and preserve (0.52).

The transfer of residues from field-treated raw tomatoes to juice, purée, paste and pomace, as well as the effect of canning on incurred residues, was investigated using simulated commercial practices. The Meeting estimated average processing factors for processed tomato commodities of 0.74 for juice, 0.69 for purée, 0.8 for wet pomace and 3.8 for dry pomace.

The Meeting derived processing factors of 13.5 for soya bean hulls, 1.6 for soya bean meal and 0.75 for soya bean flakes, based on a processing study in which soya beans treated with methamidophos in a field trial in the USA, containing initial residues of 0.08 mg/kg methamidophos, were processed using procedures that simulated commercial practice. No residues were detectable in any of the oil fractions or in the soapstock.

The Meeting estimated a maximum residue level, HR and STMR for methamidophos in soya beans to accommodate methamidophos residues arising from the use of acephate. As some acephate may be converted to methamidophos on processing, the relevant processing factors for the estimation of residues in crude soya bean oil (<0.5) and animal feed commodities (2.0 for soya bean meal and 4.5 for soya bean hulls) were based on the acephate processing study.

One processing study with sugar beet was reported in which sugar beet roots from a field trial in the USA, containing 0.05 mg/kg methamidophos, were processed into juice, pulp, molasses and sugar. The Meeting noted that no residues were detectable in any of the fractions analyzed.

Cotton seed from a residue trial in the USA in which fivefold rates of methamidophos were applied, was processed in a way that simulated commercial practice. Initial residues in the cotton seed were 0.74 mg/kg and no residues were detectable in the crude or refined oil, or the soapstock. Residues were found in the meal and hulls, and the Meeting estimated processing factors of 0.014 for cotton seed oil (crude), 0.58 for cotton meal and 0.76 for cotton hulls.

Farm animal dietary burden

The Meeting estimated the dietary burdens of methamidophos residues for livestock (Tables 57 and 58) using the diets in Appendix IX of the FAO Manual. The calculation from MRLs or HRs in feed provides the feed levels suitable for estimating animal commodity maximum residue levels, while the calculation from feed STMRs is suitable for the estimation of animal commodity STMRs.

Table 57. Farm animal estimated maximum dietary burden of methamidophos.

Commodity	MRL or HR	Group	% DM	MRL ÷ DM	% of diet			Residue contribution, mg/kg		
					Beef	Dairy	Poultry	Beef	Dairy	Poultry
Apple pomace, wet	0.28×1.35 = 0.378	AB	40	0.945						
Potato culls	0.02	VR	20	0.1						
Potato processed waste	0.02	AB	15	0.13						
Beet, fodder tops	6.1	AV	23	26.5	20	10		5.3	2.65	
Cotton seed	0.16	SO	88	0.18	25	25		0.045	0.045	
Cotton gin by-products	7.7	AM	90	8.56						
Cotton meal	0.16×0.58 = 0.093	-	89	0.104			20			0.0208
Cotton hulls	0.16×0.76 = 0.122	AM	90	0.135						
Soya bean seed	0.06	VD	89	0.067			20			0.0134
Soya bean meal	0.06×2.0 = 0.12	AL	92	0.13						
Soya bean hulls	0.06×4.5 = 0.27	AL	90	0.3						
TOTAL					45	35	40	5.3	2.7	0.0342

Table 58. Farm animal estimated STMR dietary burden of methamidophos.

Commodity	MRL or HR	Group	% DM	MRL ÷ DM	% of diet			Residue contribution, mg/kg		
					Beef	Dairy	Poultry	Beef	Dairy	Poultry
Apple pomace, wet	0.06×1.35 = 0.081	AB	40	0.2025						
Potato culls	0.01	VR	20	0.05						
Potato processed waste	0.01	AB	15	0.067						
Beet, fodder tops	2.1	AV	23	9.1	20	10		1.82	0.91	
Cotton seed	0.01	SO	88	0.011	25	25		0.00275	0.00275	
Cotton gin by-products	0.77	AM	90	0.86						
Cotton meal	0.01×0.58 = 0.0058	-	89	0.0065			20			0.0013
Cotton hulls	0.01×0.76=0.0076	AM	90	0.008						
Soya bean seed	0.01	VD	89	0.0122			20			0.0024
Soya bean meal	0.01×2 = 0.02	AL	92	0.022						
Soya bean hulls	0.01×4.5= 0.045	AL	90	0.05						
TOTAL					45	35	40	1.82	0.91	0.0037

The methamidophos dietary burdens for estimating animal commodity maximum residue levels and STMRs (residue levels in animal feeds expressed on dry weight) are beef cattle 5.3 and 1.8 ppm, dairy cattle 2.7 and 0.91 ppm and poultry 0.034 and 0.0037 ppm.

Farm animal feeding studies

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were dosed with methamidophos for 28 days at the equivalent of 0.2, 1.0 and 5.0 ppm in the diet. Residues in tissues were all <0.01 mg/kg. Owing to the interval between sample collection and analysis, liver residues were estimated to be <0.07 mg/kg, from the rate of decomposition in liver during frozen storage and the storage period. In a supplementary study, residues in liver after dosing for 30 days at 10 and 20 ppm in the diet were <0.01 mg/kg from the 10 ppm dose group and up to 0.03 mg/kg from the 20 ppm dose group. Residues in milk were a maximum of 0.021 mg/kg at the 5 ppm feeding level.

The Meeting also received information on the residue levels arising in tissues and eggs when laying hens were dosed with methamidophos for 28 days at the equivalent of 2, 6 and 20 ppm in the diet. Residues in composite samples of tissues or eggs were highest in the eggs. Transfer factors, based on average residues in tissues and eggs from the 20 ppm feeding level, were 0.0001, 0.00015, 0.0002, 0.0009, 0.0011, 0.002 and 0.006 for fat, liver, kidney, skin, heart/gizzard, muscle and eggs, respectively.

Maximum residue levels and STMRs in animal commodities

The maximum dietary burdens for beef and dairy cattle were estimated to be 5.3 and 2.7 ppm, respectively, so the levels of residues in tissues and milk can be estimated from the highest residues

observed in tissues and the mean residue level in milk at the 5 ppm feeding level (10 ppm for liver) and also by taking into account the results of the metabolism study on lactating goats. The estimated maximum residue levels expected in tissues were <0.01 mg/kg and the mean residue level in milk was 0.011 mg/kg.

The Meeting estimated maximum residue levels for meat (from mammals other than marine mammals) of 0.01* mg/kg; in edible offal (mammalian) of 0.01 (*) mg/kg; and in milks of 0.02 mg/kg. These are recommended to replace the existing CXLs of 0.01 (*) mg/kg for cattle fat, cattle meat, goat meat, goat fat, pig meat, pig fat, sheep meat, sheep fat, and milks.

The estimated STMRs dietary burdens for beef and dairy cattle were 1.8 and 0.91 ppm, respectively. The estimated STMRs were: meat (from mammals other than marine mammals) <0.01 mg/kg; fat (from mammals other than marine mammals) <0.01 mg/kg; edible offal (mammalian) <0.01 mg/kg; and milks <0.01 mg/kg (Table 59).

Table 59. Estimated maximum and STMR levels of methamidophos residues in cattle tissues and milk.

Dietary burden (ppm) [1] Feeding level [ppm] [2]		Methamidophos residues, mg/kg [3]								
		Milk	Fat		Muscle		Liver [4]		Kidney	
		Mean	high	mean	High	mean	high	mean	High	Mean
MRL, beef	(5.3) [5]		*(<0.01)* <0.01		*(<0.01)* <0.01		*(<0.01)* <0.01		*(<0.01)* <0.01	
MRL, dairy	(2.7) [5]	*(0.011)* 0.021								
STMR, beef	(1.8) [5]			*(<0.01)* <0.01		*(<0.01)* <0.01		*(<0.01)* <0.01		*(<0.01)* <0.01
STMR, dairy	(0.91) [5]	*(<0.01)* 0.021								

[1] Values in parentheses are the estimated dietary burdens.

[2] Values in square brackets are the actual feeding levels in the transfer study.

[3] Residue values in parentheses in italics are estimated from the dietary burden, the feeding levels in the feeding study and the residues found in the feeding study. "High" is the highest individual animal tissue residue in the relevant feeding group. "Mean" is mean animal tissue (or milk) residue in the relevant feeding group.

[4] The animal transfer feeding level for liver was 10 ppm for both estimation of the maximum residue level and the STMR.

The estimated maximum dietary burden for poultry is 0.034 ppm. The levels of residues in tissues and eggs are all expected to be <0.01 mg/kg when poultry are fed at this level.

The Meeting estimated maximum residue levels of 0.01 (*) mg/kg for poultry meat, poultry offal and eggs.

As no residues are expected at the maximum feeding level for poultry, the STMRs for poultry meat, edible offal and eggs are estimated to be zero.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Table 60 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: *methamidophos.*

The residue definition applies to plant and animal commodities.

Table 60. Summary of recommendations.

Commodity		MRL, mg/kg		STMR or	HR or
CCN	Name	New	Previous	STMR-P	HR-P
AL 1021	Alfalfa forage (green)	W	2		
VS 0620	Artichokes, globe	0.2 (Ac)	-	0.03	0.08
VB 0041	Cabbages, head	1	0.5	0.04	0.62
MF 0812	Cattle fat	W	0.01 *		

Commodity		MRL, mg/kg		STMR or STMR-P	HR or HR-P
CCN	Name	New	Previous		
MM 0812	Cattle meat	W	0.01 *		
VB 0404	Cauliflowers	W	0.5		
VP 0061	Beans, except broad bean and soya bean	1 (Ac)	-	0.17	0.54
SO 0691	Cotton seed	0.2	0.1	0.01	0.16
MO 0105	Edible offal (mammalian)	0.01 *		0.01	0.01
PE 0112	Eggs	0.01 * note		0	0.01
VB 0042	Flower head brassicas	0.5 (Ac)	-	0.02	0.33
AV 1051	Fodder beet	0.02		0.01	0.01
AM 1051	Fodder beet leaves or tops	30		9.1	26.5
MF 0814	Goat fat	W	0.01 *		
MM 0814	Goat meat	W	0.01 *		
VL 0482	Lettuce, head	W	1		
FC 0003	Mandarins (incl. mandarin-like hybrids)	0.5 (Ac)	-	0.085	0.26
MM 0095	Meat (from mammals other than marine mammals)	0.01 *		0.01 muscle 0.01 fat	0.01 muscle 0.01 fat
ML 0106	Milks	0.02	0.01 *	0.01	0.011
FS 0245	Nectarines	0.5 (Ac)		0.19	0.35
FS 0247	Peaches	0.5 (Ac)	1	0.19	0.35
VO 0444	Peppers, chilli	W	2		
VO 0445	Peppers, sweet	W	1		
VO 0051	Peppers	2 (Ac)		0.25	1.6
MF 0818	Pig fat	W	0.01 *		
MM 0818	Pig meat	W	0.01 *		
FP 0009	Pome fruit	0.5 (Ac)	0.5	0.06	0.24
VR 0587	Potatoes	0.05	0.05	0.01	0.02
PM 0110	Poultry meat	0.01 * note		0 muscle 0 fat	0.01 muscle 0.01 fat
PO 0111	Poultry, edible offal	0.01 * note		0	0.01
MF 0822	Sheep fat	W	0.01 *		
MM 0822	Sheep meat	W	0.01 *		
VD 0541	Soya beans (dry)	0.1 (Ac)	0.05	0.01	0.06
VR 0596	Sugar beet	0.02	0.03	0.01	0.01
AV 0596	Sugar beet leaves or tops	30	1	9.1	26.5
VO 0448	Tomatoes	2	1	0.285	1.5
FT 0312	Tree tomatoes	W	0.01	-	-
JF 0226	Apple juice			0.06	
OC 0691	Cotton seed oil, crude			0.00014	
OC 0541	Soya bean oil, crude			0.005	
JF 0448	Tomato juice			0.211	
	Tomato paste			0.239	
	Tomatoes, peeled			0.285	

* At or about the LOQ.

(Ac) = residues arising from use of acephate.

Note: animal commodity, no residues expected from consumption of feed commodities with methamidophos residues, as evaluated by JMPR.

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of methamidophos has resulted in recommendations for maximum residue levels and STMRs for raw and processed commodities. Consumption data were available for 17 food commodities and were used in the dietary intake calculation. The results are shown in Table 61.

The International Estimated Daily Intakes for the 5 GEMS/Food regional diets, based on estimated STMRs were in the range 2-20% of the maximum ADI of 0.004 mg/kg bw (Table 61). The Meeting concluded that the long-term intake of residues of methamidophos, arising from uses of methamidophos and acephate that have been considered by the JMPR, is unlikely to present a public health concern.

Table 61. International Estimated Daily Intakes (IEDIs) of methamidophos for the 5 GEMS/Food regional diets (ADI = 0-0.004 mg/kg bw/day).

Code	Commodity	STMR or STMR-P mg/kg	Diets: g/person/day. Intake = daily intake: µg/person									
			Mid-East		Far-East		African		Latin American		European	
			diet	intake	diet	intake	diet	intake	diet	intake	diet	intake
FP 0226	Apple (note 1)	0.06	7.5	0.5	4.7	0.3	0.3	0.0	5.5	0.3	40.0	2.4
JF 0226	Apple juice	0.06	4.5	0.3	0	0.0	0	0.0	0.3	0.0	3.8	0.2
VS 0620	Artichoke globe	0.03	2.3	0.1	0.0	0.0	0.0	0.0	0.0	0.0	5.5	0.2
VP 0061	Beans except broad bean & soya bean (green pods & immature seeds)	0.17	3.9	0.7	0.9	0.2	0.0	0.0	4.4	0.7	13.2	2.2
VB 0400	Broccoli (note 2)	0.02	0.5	0.0	1.0	0.0	0.0	0.0	1.1	0.0	2.7	0.1
VB 0401	Broccoli, Chinese (note 2)	0.02	-	-	-	-	-	-	-	-	-	-
-d	Cabbages (head & leafy brassicas, kohlrabi)	0.04	5.0	0.2	9.7	0.4	0.0	0.0	10.5	0.4	26.8	1.1
VB 0404	Cauliflower (note 2)	0.02	1.3	0.0	1.5	0.0	0	0.0	0.3	0.0	13	0.3
SO 0691	Cotton seed	0.01	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
OC 0691	Cotton seed oil, crude	0.00014	3.8	0.0	0.5	0.0	0.5	0.0	0.5	0.0	0.0	0.0
MO 0105	Edible offal (mammalian)	0.01	4.2	0.0	1.4	0.0	2.8	0.0	6.1	0.1	12.4	0.1
PE 0112	Eggs	0	14.6	0.0	13.1	0.0	3.7	0.0	11.9	0.0	37.6	0.0
FC 0003	Mandarins (incl. Mandarin-like hybrids)	0.085	8.8	0.7	0.2	0.0	0.0	0.0	6.3	0.5	6.0	0.5
MM 0095	Meat from mammals other than marine mammals: 20% as fat	0.01	7.4	0.1	6.6	0.1	4.8	0.0	9.4	0.1	31.1	0.3
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	0.01	29.6	0.3	26.2	0.3	19.0	0.2	37.6	0.4	124.4	1.2
ML 0106	Milks	0.01	116.9	1.2	32.1	0.3	41.8	0.4	160.1	1.6	289.3	2.9
-d	Peaches & nectarines	0.19	2.5	0.5	0.5	0.1	0.0	0.0	0.8	0.2	12.5	2.4
FP 0230	Pear (note 1)	0.06	3.3	0.2	2.8	0.2	0.0	0.0	1.0	0.1	11.3	0.7
VO 0051	Peppers	0.25	3.4	0.9	2.1	0.5	5.4	1.4	2.4	0.6	10.4	2.6
VR 0589	Potato	0.01	59.0	0.6	19.2	0.2	20.6	0.2	40.8	0.4	240.8	2.4
PM 0110	Poultry meat: 10% as fat	0	3.1	0.0	1.3	0.0	0.6	0.0	2.5	0.0	5.3	0.0
PM 0110	Poultry meat: 90% as muscle	0	27.9	0.0	11.9	0.0	5.0	0.0	22.8	0.0	47.7	0.0
PO 0111	Poultry, edible offal of	0	0.1	0.0	0.1	0.0	0.1	0.0	0.4	0.0	0.4	0.0
FP 0231	Quince (note 1)	0.06	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0
VD 0541	Soya bean (dry)	0.01	4.5	0.0	2.0	0.0	0.5	0.0	0.0	0.0	0.0	0.0
OC 0541	Soya bean oil, crude	0.005	1.3	0.0	1.7	0.0	3.0	0.0	14.5	0.1	4.3	0.0
VR 0596	Sugar beet	0.01	0.5	0.0	0.0	0.0	0.0	0.0	0.3	0.0	2.0	0.0
VO 0448	Tomato (fresh)	0.285	44.1	12.6	5.7	1.6	14.6	4.2	25.5	7.3	34.9	9.9
JF 0448	Tomato juice	0.314	0.3	0.1	0.0	0.0	0.0	0.0	0.0	0.0	2.0	0.6
-d	Tomato paste	1.425	5.8	8.3	0.2	0.3	0.3	0.4	0.0	0.0	4.0	5.7
-d	Tomatoes peeled	0.285	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	4.0	1.1
Total intake (µg/person)=			27.1		4.5		6.9		12.8		37.0	
Bodyweight per region (kg bw) =			60		55		60		60		60	
ADI (µg/person)=			240		220		240		240		240	
%ADI=			11.3		2.0		2.9		5.3		15.4	
Rounded %ADI=			10		2		3		5		20	

Note 1. Group maximum residue level proposed for pome fruit.
Note 2. Group maximum residue level proposed for flower head brassicas.

Short-term intake

The international estimated short-term intake (IESTI) for methamidophos was calculated for the food commodities (and their processed fractions) for which HRs, arising from the use of methamidophos and/or acephate, were estimated and for which consumption data were available. The results are shown in Tables 62 and 63.

The IESTI varied from 0-150% of the acute RfD (0.01 mg/kg bw) for the general population and from 0-410% of the acute RfD for children 6 years and below. The estimated short-term intakes from cabbages, sweet peppers and tomatoes were 120% to 150% of the acute RfD for the general population. For children 6 years and below, estimated short-term intakes were between 110% and 410% of the acute RfD from broccoli, cauliflowers, apples, sweet peppers, cabbages and tomatoes. The information provided to the Meeting precluded a conclusion that the acute dietary intake for these commodities would be below the acute RfD.

The Meeting concluded that the short-term intake of residues of methamidophos, arising from the uses of methamidophos and acephate, that have been considered by the JMPR is unlikely to present a public health concern, with the exception of apples, broccoli, cabbages (head), cauliflowers, sweet peppers and tomatoes.

Table 62. Assessment of risks to the general population from the short-term dietary intake of residues of methamidophos (acute RfD = 0.01 mg/kg bw, i.e. 10 µg/kg bw).

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight			Variability factor	Case	IESTI µg/kg bw/day	% acute RfD, rounded
				Country	Body wt (kg)	Large portion, g/person	Unit wt, g	Country	Unit wt, edible portion, g				
FP 0226	Apple (note 1)	-	0.24	USA	65.0	1348	110	FRA	100	3	2a	5.72	60
JF 0226	Apple juice	-	0.24	-	-	-	-	-	-	-	3	-	-
VS 0620	Artichoke globe	-	0.08	FRA	62.3	534	230	FRA	99	3	2a	0.94	9
VP 0061	Beans except broad bean & soya bean (green pods & immature seeds)	-	0.54	FRA	62.3	312	-	-	-	-	1	2.70	30
VB 0400	Broccoli (note 2)	-	0.33	USA	65.0	376	608	USA	474	3	2b	5.73	60
VB 0041	Cabbages, head	-	0.62	SAF	55.7	362	771	UNK	540	3	2b	12.09	120
VB 0404	Cauliflower (head) (note 2)	-	0.33	UNK	70.1	579	1733	UNK	780	3	2b	8.18	80
PE 0840	Chicken eggs	-	0.01	FRA	62.3	219	-	-	-	-	1	0.04	0
SO 0691	Cotton seed	0.01	-	USA	65.0	3	-	-	-	-	3	0.00	0
OC 0691	Cotton seed oil, crude	0.00014	-	-	-	-	-	-	-	-	3	-	-
MO 0105	Edible offal (mammalian)	-	0.01	FRA	62.3	277	-	-	-	-	1	0.04	0
FC 0206	Mandarin	-	0.26	JPN	52.6	409	70	JPN	70	3	2a	2.71	30
MM 0095	Meat from mammals other than marine mammals: 20% as fat	-	0.01	AUS	67.0	104	-	-	-	-	1	0.02	0
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	-	0.01	AUS	67.0	417	-	-	-	-	1	0.06	1
ML 0106	Milks	0.01	-	USA	65.0	2466	-	-	-	-	3	0.38	4
FS 0245	Nectarine	-	0.35	USA	65.0	590	110	FRA	99	3	2a	4.24	40
FS 0247	Peach	-	0.35	SAF	55.7	685	110	FRA	99	3	2a	5.55	60
FP 0230	Pear (note 1)	-	0.24	USA	65.0	693	100	FRA	89	3	2a	3.22	30
VO 0444	Peppers, chili (note 3)	-	1.6	USA	65.0	90	45	USA	43	3	2a	4.35	40
VO 0445	Peppers, sweet (incl. pimento) (note 3)	-	1.6	FRA	62.3	207	172	UNK	160	3	2a	13.54	140

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight			Variability factor	Case	IESTI µg/kg bw/day	% acute RfD, rounded
				Country	Body wt (kg)	Large portion, g/person	Unit wt, g	Country	Unit wt, edible portion, g				
VR 0589	Potato	-	0.02	NLD	63.0	687	216	UNK	216	3	2a	0.36	4
PM 0110	Poultry meat: 10% as fat	-	0.01	AUS	67.0	43	-	-	-	-	1	0.01	0
PM 0110	Poultry meat: 90% as muscle	-	0.01	AUS	67.0	388	-	-	-	-	1	0.06	1
PO 0111	Poultry, edible offal of	-	0.01	USA	65.0	248	-	-	-	-	1	0.04	0
FP 0231	Quince (note 1)	-	0.24	AUS	67.0	175	92	USA	56	3	2a	1.03	10
VD 0541	Soya bean (dry)	0.01	-	JPN	52.6	159	-	-	-	-	3	0.03	0
OC 0541	Soya bean oil, crude	0.005	-	-	-	-	-	-	-	-	3	-	-
VR 0596	Sugar beet	-	0.01	-	-	-	-	-	-	-	-	-	-
VO 0448	Tomato (fresh, juice, paste, peeled)	-	1.5	USA	65.0	391	123	USA	123	3	2a	14.69	150

Note 1. Group maximum residue level proposed for pome fruit.
Note 2. Group maximum residue level proposed for flower head brassicas.
Note 3. Maximum residue level proposed for peppers includes both peppers, sweet, and peppers, chilli.

Table 63. Assessment of risks to children up to 6 years from the short-term dietary intake of residues of methamidophos (acute RfD = 0.01 mg/kg bw, i.e. 10 µg/kg bw).

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight			Variability factor	Case	IESTI µg/kg bw/day	% acute RfD, rounded
				Country	Body wt (kg)	Large portion, g/person	Unit wt, g	Country	Unit wt, edible portion, g				
FP 0226	Apple (note 1)	-	0.24	USA	15.0	679	110	FRA	100	3	2a	14.06	140
JF 0226	Apple juice	-	0.24	-	-	-	-	-	-	-	3	-	-
VS 0620	Artichoke globe	-	0.08	FRA	17.8	89	230	FRA	99	3	2b	1.20	10
VP 0061	Beans except broad bean & soya bean (green pods & immature seeds)	-	0.54	FRA	17.8	203	-	-	-	-	1	6.15	60
VB 0400	Broccoli (note 2)	-	0.33	USA	15.0	164	608	USA	474	3	2b	10.84	110
VB 0041	Cabbages, head	-	0.62	SAF	14.2	220	771	UNK	540	3	2b	28.83	290
VB 0404	Cauliflower (head) (note 2)	-	0.33	NLD	17.0	209	1733	UNK	780	3	2b	12.19	120
PE 0840	Chicken eggs	-	0.01	FRA	17.8	134	-	-	-	-	1	0.08	1
SO 0691	Cotton seed	0.01	-	USA	15.0	1	-	-	-	-	3	0.00	0
OC 0691	Cotton seed oil, crude	0.00014	-	-	-	-	-	-	-	-	3	-	-
MO 0105	Edible offal (mammalian)	-	0.01	FRA	17.8	203	-	-	-	-	1	0.11	1
FC 0206	Mandarin	-	0.26	JPN	15.9	353	70	JPN	70	3	2a	8.07	80
MM 0095	Meat from mammals other than marine mammals: 20% as fat	-	0.01	AUS	19.0	52	-	-	-	-	1	0.03	0
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	-	0.01	AUS	19.0	208	-	-	-	-	1	0.11	1
ML 0106	Milks	0.01	-	USA	15.0	1286	-	-	-	-	3	0.86	9
FS 0245	Nectarine	-	0.35	AUS	19.0	302	110	FRA	99	3	2a	9.21	90
FS 0247	Peach	-	0.35	AUS	19.0	315	110	FRA	99	3	2a	9.46	90
FP 0230	Pear (note 1)	-	0.24	UNK	14.5	279	100	FRA	89	3	2a	7.56	80
VO 0444	Peppers, chili (note 3)	-	1.6	AUS	19.0	31	45	USA	43	3	2b	7.71	80

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight			Variability factor	Case	IESTI µg/kg bw/day	% acute RfD, rounded
				Country	Body wt (kg)	Large portion, g/person	Unit wt, g	Country	Unit wt, edible portion, g				
VO 0445	Peppers, sweet (incl. pim(i)ento) (note 3)	-	1.6	AUS	19.0	60	172	UNK	160	3	2b	15.17	150
VR 0589	Potato	-	0.02	SAF	14.2	300	216	UNK	216	3	2a	1.03	10
PM 0110	Poultry meat: 10% as fat	-	0.01	AUS	19.0	22	-	-	-	-	1	0.01	0
PM 0110	Poultry meat: 90% as muscle	-	0.01	AUS	19.0	201	-	-	-	-	1	0.11	1
PO 0111	Poultry, edible offal of	-	0.01	USA	15.0	37	-	-	-	-	1	0.02	0
FP 0231	Quince (note 1)	-	0.24	NLD	17.0	1	92	USA	56	3	2b	0.04	0
VD 0541	Soya bean (dry)	0.01	-	JPN	15.9	88	-	-	-	-	3	0.06	1
OC 0541	Soya bean oil, crude	0.005	-	-	-	-	-	-	-	-	3	-	-
VR 0596	Sugar beet	-	0.01	-	-	-	-	-	-	-	-	-	-
VO 0448	Tomato (fresh, juice, paste, peeled)	-	1.5	USA	15.0	159	123	USA	123	3	2a	40.50	410

Note 1. Group maximum residue level proposed for pome fruit.
Note 2. Group maximum residue level proposed for flower head brassicas.
Note 3. Maximum residue level proposed for peppers includes both peppers, sweet, and peppers, chilli.

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