OXAMYL (126)

First draft prepared by Ursula Banasiak, Federal Biological Research Centre for Agriculture and Forestry (BBA), Kleinmachnow, Germany

EXPLANATION

Oxamyl was listed by the 1997 CCPR (29th Session, ALINORM 97/24A) for periodic re-evaluation for residues by the 2002 JMPR.

IDENTITY

ISO common name: oxamyl

Chemical name
IUPAC: *N,N*-dimethyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide
CAS: methyl 2-(dimethylamino)-*N*-[[(methylamino)carbonyl]oxy]-2-oxoethanimidothioate

CAS Registry No: 23135-22-0

CIPAC No: 342

Synonyms: DPX-D1410, oxamil

Structural formula:
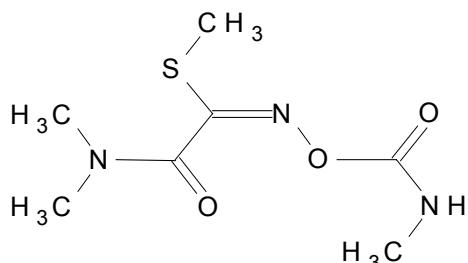
Molecular formula: $C_7H_{13}N_3O_3S$
Molecular weight: 219.3

Physical and chemical properties

<u>Pure active ingredient</u>

Appearance: White crystalline solid (Tuffy, 2000a)
Vapour pressure (at 25°C): 5.12×10^{-5} Pa (Barefoot, 1989a)
Henry's law constant (at 25°C): 3.96×10^{-8} Pa m^3 x mol^{-1} (Barefoot, 1989b)
Melting point: 99.8°C (Silveira, 1988)
Octanol/water partition coefficient (at 25°C): 0.36 (Melander, 1988)
Solubility in water (at 25°C):   28.2 g/100 g (Hoffmann, 1988)
Solubility in organic solvents (at 20°C):

acetone:	>250 g/kg
dichloromethane:	>250 g/kg
methanol:	>250 g/kg
n-heptane:	10.5 mg/l
ethyl acetate:	4.13×10^4 mg/l

o-xylene:	3.14×10^3 mg/l (Hansen, 2000)
Relative density:	1.313 g/cm^3 (1313 kg/m^3) (Tuffy, 2000b)
Hydrolysis (half-life at 25°C):	pH 5: <31 days, pH 7: 7.9 days, pH 9: 0.12 days (McNally and Wheeler, 1988a)
Photolysis (half-life at 25°C):	7 days (McNally and Wheeler, 1988b)

Technical material

Appearance:	no information.
Density:	no information.
Purity:	42% (w/w)
Melting range:	no information.
Thermal stability:	no information.
Stability:	no information.
Flammability:	oxamyl technical (42%) is flammable (flash point 57.4°C). The 42% material is dissolved in cyclohexanone, a flammable liquid (flash point 44°C) and water (Bates, 2000).
Auto-flammability:	303°C (±5°C) auto-ignition temperature of oxamyl technical 42% (Bates, 2000)

Formulations

| ai Content	Formulation type	Principal crop uses
240 g/l	SL	fruit, vegetables, potatoes, cotton
100 g/kg	GR	fruit, vegetables, potatoes, cotton
420 g/l	SL	cotton, peanuts, soya bean, potatoes, tobacco
50 g/kg	GR	fruit, vegetables, potatoes, cotton
100 g/l	SL	fruit, vegetables, potatoes, cotton
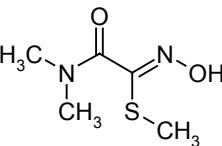
METABOLISM AND ENVIRONMENTAL FATE

Chemical names, abbreviated names and code numbers of oxamyl degradation products

The metabolism studies were conducted using
- methyl 2-(dimethylamino)-*N*-[[(methylamino)carbonyl]oxy]-2-oxo-[1-^{14}C]ethanimidothioate (referred to as [^{14}C]oxamyl) and
- methyl 2-(dimethylamino)-*N*-hydroxy-2-oxo-[1-^{14}C]ethanimidothioate (referred to as [^{14}C]oxamyl oxime).

As well as oxamyl, the compounds shown below were found in one or more studies involving the metabolism and/or environmental fate of oxamyl. The compounds are arranged in ascending order of IN- code number.

IN-A2213	**IUPAC name**	(*Z*)-2-hydroxyimino-*N,N*-dimethyl-2-(methyltyhio)acetamide
	CAS name:	methyl 2-(dimethylamino)-*N*-hydroxy-2-oxoethanimidothioate
	Abbreviated name:	oxamyl oxime

CAS registry number:	66344-33-0 (*Z*-isomer)	**Molecular weight:**	162.21
Molecular formula:	C$_5$H$_{10}$N$_2$O$_2$S	**Found in:**	Plants, rumen fluid, rat urine,

topsoil, saturated zone subsoil,
water/sediment, water (hydrolysis)

IN-A2912 **IUPAC name:** 2-hydroxyimino-*N,N*-dimethyl-2-(methylsulfonyl)acetamide
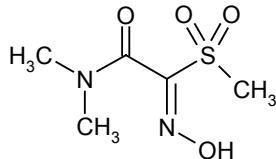
CAS registry number:	not available	**Molecular weight:**	194.21
Molecular formula:	$C_5H_{10}N_2O_4S$	**Found in:**	Looked for but not found in any metabolism studies

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IN-D1409 **IUPAC name** *N*-methyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide

CAS name: methyl 2-(methylamino)-*N*-[[(methylamino)carbonyl]oxy]-2-oxoethanimidothioate

Abbreviated name: *N*-demethyl-oxamyl
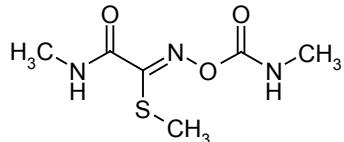
CAS registry number:	50917-40-3	**Molecular weight:**	205.24
Molecular formula:	$C_6H_{11}N_3O_3S$	**Found in:**	Rumen fluid, rat liver microsomes

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IN-D2708 **IUPAC name** dimethyloxamic acid

CAS name: dimethylamino(oxo)acetic acid

Abbreviated name: DMOA
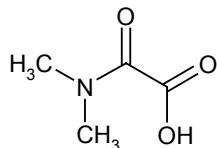
CAS registry number:	32833-96-8	**Molecular weight:**	117.105
Molecular formula:	$C_4H_7NO_3$	**Found in:**	Plants, rumen fluid, rat liver microsomes, topsoil, saturated zone subsoil, water/sediment

IN-E2816   **IUPAC name:**   2-(hydroxyimino)-*N,N*-dimethyl-2-(methylsulfinyl)acetamide
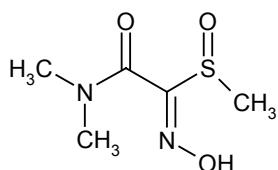
**CAS registry number:**   not available   **Molecular weight:**   178.21

Molecular formula: $C_5H_{10}N_2O_3S$ **Found in:**   Poultry excreta (tentative)

IN-F3905   **IUPAC name**   (*E*)-2-hydroxyimino-*N,N*-dimethyl-2-(methyltyhio)acetamide

CAS name: methyl (*E*) 2-(dimethylamino)-*N*-hydroxy-2-oxoethanimidothioate

Abbreviated name: oxime isomer
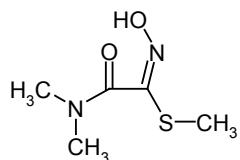
CAS registry number: 66344-32-9   **Molecular weight:**   162.21
(E-isomer)

Molecular formula: $C_5H_{10}N_2O_2S$ **Found in:**   Poultry excreta, topsoil

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IN-KP532   **Chemical name:**   methylamino(oxo)acetic acid
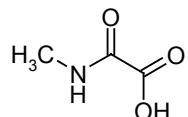
CAS registry number: 29262-58-6   **Molecular weight:**   103.08

Molecular formula: $C_3H_5NO_3$ **Found in:**   Goat rumen fluid, rat urine

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IN-KV998   **CAS name:**   *N*-methylethanediamide

CAS registry number: 22509-04-2   **Molecular weight:**   102.09
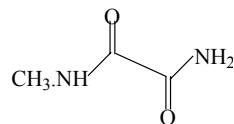
Molecular formula: $C_3H_6N_2O_2$ **Found in:**   Goat

IN-L2953   **IUPAC name:**   2-hydroxyamino-*N*-methyl-2-(methylthio)acetamide

CAS name: methyl N-hydroxy-2-(methylamino)-2-oxoethanimidothioate

Abbreviated name: N-demethyl oxime, NDMO
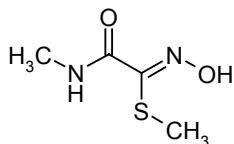
CAS registry number: 66157-67-3   **Molecular weight:**   148.18

Molecular formula: $C_4H_8N_2O_2S$ **Found in:**   Plants, rumen fluid, rat liver microsomes, rat

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IN-M2583   **CAS name:**   *S*-Methyl (dimethylamino)oxoethanethioate
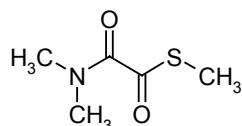
CAS registry number: Not available   **Molecular weight:**   147.20

Molecular formula: $C_5H_9NO_2S$ **Found in:**   Saturated zone subsoil

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IN-N0079   **IUPAC name:**   *N,N*-dimethyl-2-nitriloacetamide

CAS name: dimethylcarbonocyanidic amide

Abbreviated name: DMCF
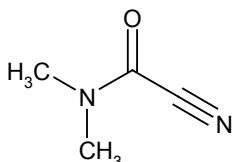
CAS registry number: 16703-51-8   **Molecular weight:**   98.10

Molecular formula: $C_4H_6N_2O$ **Found in:**   Plants, rumen fluid, rat, rat liver microsomes, saturated zone subsoil, water/sediment

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IN-T2921   **IUPAC name:**   *N,N,*-dimethyloxamide

CAS name: *N,N*-dimethylethanediamide

Abbreviated name: DMEA (also DMO)
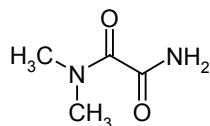
CAS registry number: 600-39-5   **Molecular weight:**   116.12

Molecular formula: $C_4H_8N_2O_2$ **Found in:**   Rumen fluid, plants, saturated zone subsoil, water/sediment

IN-U1966   **CAS name:**   *N,N*-dimethyl-2-[[[(methylamino)carboxyl]oxy]imino]-2-(methylsulfonyl)acetamide
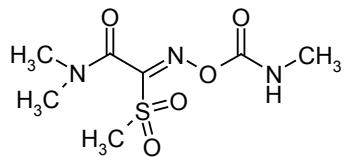
CAS registry number: not available   **Molecular weight:**   251.26

Molecular formula: $C_7H_{13}N_3O_5S$ **Found in:**   Looked for but not found in any metabolism studies

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IN-U1967   **CAS name:**   *N,N*-dimethyl-2-[[[(methylamino)carboxyl]oxy]imino]-2-(methylsulfinyl)acetamide
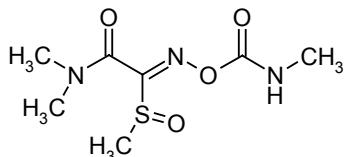
CAS registry number: **Molecular weight:**   235.26

Molecular formula: $C_7H_{13}N_3O_4S$ **Found in:**   Looked for but not found in any metabolism studies

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IN-00699   **IUPAC name:**   oxamide

           CAS name: ethanediamide
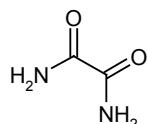
CAS registry number: 471-46-5   **Molecular weight:**   88.07

Molecular formula: $C_2H_4N_2O_2$ **Found in:**   Goat urine

IN-18474   **IUPAC name:**   oxamic acid

           CAS name: aminooxoacetic acid

   Abbreviated name:
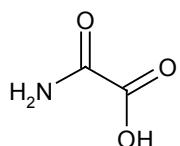
CAS registry number: 471-47-6   **Molecular weight:**   89.05

Molecular formula: $C_2H_3NO_3$ **Found in:**   Poultry excreta

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No code no.

    Chemical name: thiocyanate ion (shown as sodium salt)

$$Na^+ \quad ^-S\text{---}\!\!\equiv\!\!N$$
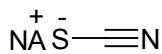
CAS registry number: 540-72-7   **Molecular weight:** 97.18

    Molecular formula: NaSCN   **Found in:** Goats, poultry

No code no.   **IUPAC name:** oxalic acid

         CAS name: ethanedioic acid
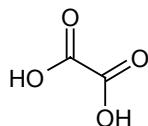
CAS registry number: 144-62-7   **Molecular weight:** 90.04

    Molecular formula: $C_2H_2O_4$ **Found in:** Goat, poultry excreta

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Animal metabolism

The metabolism of oxamyl has been studied in laboratory animals (mice and rats), and goats and poultry.

Studies on mice and rats were evaluated by the WHO Core Assessment Group at the present Meeting. They showed that the two major metabolic pathways were to 2-hydroxyimino-*N,N*-dimethyl-2-(methylthio)acetamide (oxamyl oxime, IN-A2213) and to dimethylamino(oxo)acetic acid (DMOA, IN-D2y08).

Goats *(in vivo)*, cow *(in vitro)*. Oxamyl is rapidly metabolized in the goat and its metabolic products are mainly excreted in the urine. Results from rumen fluid metabolism studies with radiolabelled oxamyl and selected plant metabolites (oxamyl oxime glucoside and IN-N0079) substantiated the metabolic pathway seen in the whole animal studies.

Belasco and Harvey (1980) collected rumen fluid from a rumen-fistulated Holstein cow which they incubated separately anaerobically at approximately 38°C with nutrients and ^{14}C-labelled oxamyl, IN-N0079 and oxamyl oxime glucoside. Samples were collected 1, 6 and/or 24 hours after incubation. Oxamyl oxime and/or IN-N0079 were the main metabolites in all three experiments, with minor amounts of IN-D2708, IN-KP532, IN-T2921, IN-L2953 and/or IN-D1409 in the oxamyl and IN-N0079 incubations. These results indicate that any oxamyl or its plant metabolites consumed by a ruminant would be metabolized before absorption into body tissues.

In the earlier study (Barefoot, 1990) a non-pregnant lactating goat (body weight approximately 60 kg) was dosed orally using a balling gun with a capsule containing 49 mg [^{14}C]oxamyl for five consecutive days after each morning milking, equivalent to a mean daily dose of 33 ppm in the diet based on actual average feed consumption of 1.5 kg/day. Milk, urine and faeces were sampled daily and selected tissues were taken within a day of the last dose. The overall recovery of the administered dose (including from the stomach and gastrointestinal tract) was 78%. The majority of the radioactivity was excreted in the urine (60% of the dose), with smaller amounts in the faeces (6.8%), with low levels in the tissues (5.3% *in toto*), milk (2.5% *in toto*) and gastrointestinal tract (1.1%), and negligible amounts in the rumen (0.1%) (Tables 1 and 2). Radioactivity in the milk reached a plateau of approximately 0.7 mg oxamyl equivalents/kg after 3 days (Table 3).

Approximately 17 different radiolabelled components (representing 78% of the residues in the milk) were extracted with methanol/methylene chloride. Milk proteins accounted for 6-10%, and after further extraction and analysis it was found that [^{14}C]lactose accounted for 6%. No oxamyl, oxamyl oxime, IN-N0079, or IN-L2953 was detected (Table 4). These results were consistent with the extensive metabolism of oxamyl and its ultimate incorporation into milk natural products.

Residues in liver and muscle were 2.9% (4.0 mg oxamyl equivalents/kg) and 2.2% (0.59 mg oxamyl equivalents/kg) of the administered dose respectively. Extraction with chloroform and/or methanol yielded multiple components from both liver and muscle. Unextractable residues were subjected to proteolytic digestion and acid hydrolysis. No oxamyl, oxamyl oxime, IN-L2953 or IN-N0079 was detected in any of the liver or muscle fractions (Table 5).

Radiolabelled components in the urine were separated into 6 fractions by HPLC and determined by TLC. Oxamyl, oxamyl oxime, IN-N0079, IN-D2708 and IN-L2953 were not detected and the main metabolites were water-soluble and polar (Table 6).

Table 1. Recovery of radioactivity from a lactating goat after five consecutive daily doses of [^{14}C]oxamyl at a level of 45 mg/day (Barefoot, 1990).

Sample	% of administered radioactivity
Urine	60.5
Faeces	6.8
Milk	2.5
Cage washings	1.4
Cage debris	0.4
Rumen contents	0.1
Gastro-intestinal tract contents	1.1
Tissues	5.3
Total	78.2

Table 2. Radioactivity in tissues of a lactating goat given five consecutive daily doses of [^{14}C]oxamyl at a level of 45 mg/day (Barefoot, 1990).

Sample	Concentration expressed as		
	% of administered ^{14}C	oxamyl equivalents, mg/kg	SD
Liver	2.9	4.09	0.14
Kidney	0.2	2.04	0.03
Muscle	2.2	0.59	0.04
Fat	<0.1	0.06	0.01
Rumen contents	0.1	0.76	0.01
Gastro-intestinal tract contents	1.1	0.33	0.02
Total	6.5		

SD: standard deviation, mg/kg

Table 3. Concentration of radioactivity in the milk, faeces and urine of a lactating goat after five consecutive daily doses of [^{14}C]oxamyl at a level of 45 mg/day (Barefoot, 1990).

Time, h	[^{14}C]oxamyl equivalents, mg/kg					
	Milk		Urine		Faeces	
	Mean	SD	Mean	SD	Mean	SD
0- 24	0.39	0.00	8.00	0.03	1.73	0.03
24- 48	0.64	0.00	11.09	0.25	3.59	0.20
48- 72	0.71	0.01	19.30	0.20	3.59	0.13
72- 96	0.69	0.01	12.58	0.12	2.40	0.20
96 – 120	0.77	0.01	19.70	0.61	3.62	0.13

Table 4. Composition of ^{14}C-residues in goat milk, percentages of total radioactivity recovered in various fractions. Total milk residues 0.64 mg/kg oxamyl equivalents (Barefoot, 1990).

Fraction	Fraction %	Compound	% of TRR
Fat[1]			<0.1
Protein[2]			6.0
Filtrate[3]	94		
Condensate			0.9
Solids[4]	14.5	lactose	6.2
		S1[5]	8.3
Methanol extract[6]	78.6		
Fraction 1	30.1		
		M1	1.0
		M2	5.1
		M3	4.8
		M4	2.8
		M5	14.8
		M6	1.6
Fraction 2	38.6		
		M7	2.7
		M8	3.7
		M9	32.2
Fraction 3[7]	10.1		10.1
Total			100.2

[1] Determined by hexane extraction of ultrafiltered milk.
[2] Determined by size exclusion analysis of whole milk.
[3] Prepared by ultrafiltration of milk which excludes molecules >10,000 daltons, assumes complete recovery of all compounds with molecular weight <10,000.
[4] Solids remaining after methanol/methylene chloride extraction from evaporation of water from ultrafiltered milk. Identified by HPLC and β-galactosidase hydrolysis.
[5] Radioactivity irreversibly absorbed by the carbohydrate column.
[6] Methanol/methylene chloride extraction after evaporation of water from ultrafiltered milk.
[7] At least 8 components, none accounting for more than 2.1 % of the radioactivity in milk.

Table 5. Distribution of radioactivity in liver and muscle of a goat. Total liver/muscle residues 4/0.59 mg/kg oxamyl equivalents (Barefoot, 1990).

Fraction	Liver			Muscle		
	% of ^{14}C in fraction	Compound (unknown)	% of [14]C	% of ^{14}C in fraction	Compound (unknown)	% of [14]C
Chloroform	1.7		1.7			
Methanol/water	42.3					
Fraction 1	4.1	(L1-L8)[1]	4.1			
Fraction 2	6.4	L9	0.9			
		L10	3.8			
		L11	1.1			
		L12	0.4			
Fraction 3	2.5	L13	2.5			
Fraction 4	20.8	L14	20.8[2]			
Fraction 5	8.5	L15	8.5[2]			
Combined extract				49.5	MU1	32.3
					MU2	10.7
					MU3	6.4
Protease digest	42.4	LP1	12.1	40.1	MP1	16.2
		LP2	3.7		MP2	12.0
		LP3	4.0		MP3	1.6
		LP4	7.6		MP4	3.2
		LP5	1.0		MP5	7.1
		LP6	3.6			
		LP7	2.3			
		LP8	8.1			
Solids[3]			4.2 %			0.5 %

Fraction	Liver			Muscle		
	% of ^{14}C in fraction	Compound (unknown)	% of [14]C	% of ^{14}C in fraction	Compound (unknown)	% of [14]C
Total			90.4			89.5

[1] At least 8 components, none exceeding 1.4 % of the radioactivity.
[2] Radioactivity remained at the origin of a silica TLC plate developed with n-propanol/water/acetic acid (70:10:1).
[3] Remaining after protease digestion.

Table 6. Composition of ^{14}C-residues in goat urine (Barefoot, 1990).

Compound (unknown)	TLC R_f	Fraction no.						% of ^{14}C in urine	% of total dose
		1	2	3	4	5	6		
U1	0.0	1.9						1.9	1.1
U2	0.09	2.7						2.7	1.6
U3	0.23	7.4						7.4	4.5
U4	0.63	2.8						2.8	1.7
U5	0.70	5.5						5.5	3.1
U6	0.85	1.1						1.1	0.7
U7	0.37		1.1					1.1	0.7
U8	0.0					1.0	0.7	1.7	1.0
U9	0.1					1.7	1.2	2.9	1.8
U10	0.23						0.8	0.8	0.5
U11	0.3			15.9	1.4	4.0	2.7	24.0	14.5
U12	0.35			5.6	2.2	2.5	2.0	12.3	7.4
U13	0.41			14.0	4.7			18.7	11.3
U14	0.48			8.1		3.9	1.7	13.7	8.3
U15	0.72			2.6		0.4	0.3	3.3	2.0
Fraction %		21.4	1.1	46.2	8.3	13.5	9.4		
Total								99.9	60.2

In a second study (Li, 1994) that confirmed the earlier results and provided additional details of the metabolites, a non-pregnant lactating goat (body weight 37 kg) was dosed orally daily with a capsule containing 59.3 mg [14C]oxamyl (using a balling gun) for five consecutive days, equivalent to a mean daily dose of 31 ppm in the diet based on actual average feed consumption of 1.9 kg/day. Milk, urine and faeces were sampled daily and selected tissues were taken within a day of the last dose. Overall recovery of the administered dose (including from the stomach and gastrointestinal tract) was 80%, most of which (45.3%) was excreted in the urine (and cage wash), with smaller amounts in the faeces (7.2%) and 1.9% exhaled as volatile metabolites. Radioactivity in the edible tissues was 1.9% of the dose in liver, 0.2% in kidney, 3.4% in muscle, and 1.2% in fat. Residues in milk increased from 1.7% (1.12 mg oxamyl equivalents/kg) on day 1 to 4.6% (2.93 mg/kg) on day 5.

Milk samples (24 h, 72 h and 120 h, am and pm combined) were extracted and analysed as shown in Figure 1. Analysis of the chloroform extract indicated that no residues of oxamyl, oxamyl oxime, IN-N0079, or IN-L2953 were present. The methanol/water extract contained the majority of the radioactivity. Radiolabelled thiocyanate was the main residue identified in the methanol/water extract and ^{14}C-lactose was a component of the solubilised residues following protease digestion of the milk solids. In addition to thiocyanate, many different, mostly polar, components were characterized in the methanol/water extract (Table 7).

No oxamyl, oxamyl oxime, oxamyl sulfone (IN-U1966), oxamyl sulfoxide (IN-U1967), IN-L2953 or IN-N0079 was detected in the organosoluble or aqueous fractions of the edible tissues. Radiolabelled thiocyanate was detected in all the tissues (expressed as oxamyl equivalents and percentage of total sample radioactivity) 0.24 mg/kg (2.8%) in liver, 0.43 mg/kg (9.1%) in kidney, 0.14 mg/kg (12.4%) in muscle and 0.19 mg/kg (31%) in fat (Table 8). Other extractable residues in liver included IN-KP532 (0.06 mg oxamyl equivalents/kg, 0.8%) and IN-KV998 (0.07 mg oxamyl equivalents/kg; 0.8%). Most of the radioactivity in the tissues was not solvent-extractable, but was

released by protease enzyme hydrolysis. The numerous radioactive components released by the protease digestion were highly polar. Oxalic acid (1.41 mg oxamyl equivalents; 16.9%) was found in the liver digest.

The main constituents in the urine were radiolabelled thiocyanate (2.54%), IN-00699 (oxamide, 10%), IN-KP532 (13%), and IN-KV998 (5.44%). No oxamyl, oxamyl oxime or IN-U1966 was detected. The concentrations of thiocyanate in the milk, tissues and urine is shown in Table 8.

Figure 1. Flowchart of fractionation and analysis of goat milk (Li, 1994).
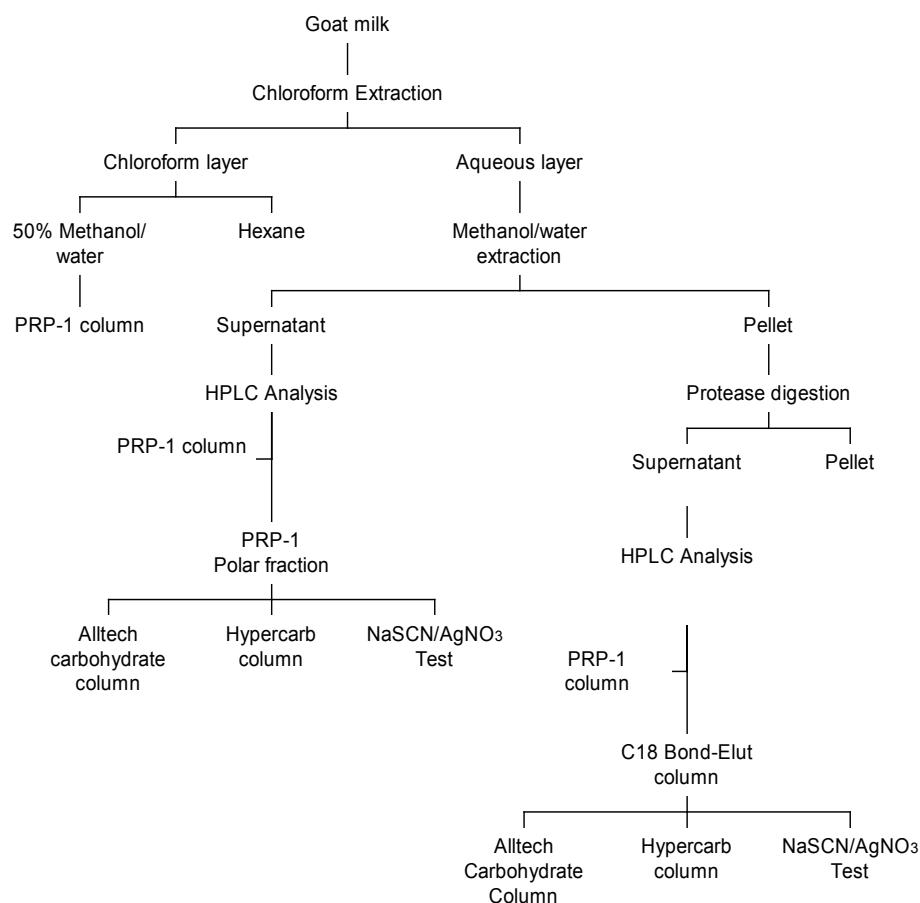
Table 7. Composition of the residue in methanol/water extracts and unextractable pellet of 24 h, 72 h, and 120 h milk (Li, 1994).

Fraction	Compound (RT, min.)[2]	24 h (1.45 mg/kg)[1]		72 h (2.87 mg/kg)[1]		120 h (4.12 mg/kg)[1]	
		% of ^{14}C	mg/kg[3]	% of ^{14}C	mg/kg[3]	% of ^{14}C	mg/kg[3]
Methanol/water		67.1	0.97	67.6	1.94	72.5	2.99
Polar fraction	Ma (3-9)	58.9		55.2		58.0	
Fraction 1	Thiocyanate (14-17)	23.9	0.35 (0.09)	22.5	0.65 (0.17)	36.1	1.49 (0.39)
Fraction 2	M2 (18-21)	7.30	0.11	7.26	0.21	3.63	0.15
Fraction 3	M3 (22-24)	1.11	0.02	2.23	0.06	1.13	0.05
Fraction 4	M4 (25-27)	2.58	0.04	1.93	0.06		
Fraction 5	M5 (28-30)	8.04	0.12	5.56	0.16	4.93	0.20
Fraction 6	M6 (31-33)	3.46	0.05	4.35	0.12	2.78	0.11
Fraction 7	M7 (34-35)	2.81	0.04	2.92	0.08	1.84	0.08
Fraction 8	M8 (36-39)	0.52	0.01	0.63	0.02	0.46	0.02
Fraction 9	M9 (41-45)	0.55	0.01	0.71	0.02	0.48	0.02
Fraction 10	M10(46-50)	1.38	0.02	0.63	0.02	0.36	0.01

Fraction	Compound (RT, min.)[2]	24 h (1.45 mg/kg)[1]		72 h (2.87 mg/kg)[1]		120 h (4.12 mg/kg)[1]	
		% of [14]C	mg/kg[3]	% of [14]C	mg/kg[3]	% of [14]C	mg/kg[3]
Fraction B	Mb (9.5.10.5)					0.57	0.02
Fraction C	Mc (12-13.5)	0.38	0.01	0.29	0.01	0.34	0.01
Fraction D	Md (14.5-16)	0.38	0.01			0.79	0.03
Fraction E	Me (17-19)	1.24	0.02	0.99	0.03	1.32	0.05
Pellet[4]		30.7	0.45	30.5	0.87	25.3	1.04
Supernatant[5]		29.4		27.6		23.3	
Polar fraction[6]	MP24, MP72, MP120	27.3		24.82		21.1	
Fraction 1	Thiocyanate	11.5	0.17 (0.04)	9.37	0.27 (0.07)	12.4	0.51 (0.13)
Fraction 2	MP2	0.48	0.01	0.48	0.01	0.29	0.01
Fraction 3	MP3	3.62	0.05	3.87	0.11	2.48	0.10
Fraction 4	MP4	1.85	0.03	2.48	0.07	1.21	0.05
Fraction 5	MP5	2.66	0.04	2.65	0.08	1.47	0.06
Fraction 6	MP6	1.43	0.02	0.74	0.02	0.65	0.03
Fraction 7	MP7	0.65	0.01	1.33	0.04	0.70	0.03
Fraction 8	MP8	2.04	0.03	1.51	0.04	0.54	0.02
Fraction 9	MP9	1.87	0.03	0.92	0.03	0.64	0.03
Lactose		4.49	0.07	5.50	0.16	3.96	0.16

[1] Total radioactive residue (TRR, oxamyl equivalent)
[2] retention time, min.
[3] % of [14]C x TRR, mg/kg oxamyl equivalent (mg/kg metabolite in parenthesis)
[4] Pellet was the remaining solid after extraction.
[4] Supernatant contained released radioactivity from protease digestion of milk pellet.
[6] Polar fraction was the aqueous fraction passed through C18 Bond-Elut® column.

Table 8. Concentration of [^{14}C]thiocyanate in milk, tissues and urine of a goat (Li, 1994).

Sample	HPLC analysis		AgNO₃ Precipitation test	
	%	mg/kg[1]	%	mg/kg[1]
24 h milk extract	23.9	0.35 (0.09)	22.8	0.33 (0.087)
24 h milk pellet	11.5	0.17 (0.04)	18.1	0.26 (0.07)
Total (24 h)	35.4	0.52 (0.13)		
72 h milk extract	22.5	0.65 (0.17)	22.0	0.63 (0.17)
72 h milk extract	9.37	0.27 (0.07)	16.6	0.48 (0.13)
Total (72 h)	31.9	0.92 (0.24)		
120 h milk extract	36.1	1.49 (0.39)	33.4	1.37 (0.36)
120 h milk pellet	12.4	0.51 (0.13)	16.6	0.68 (0.18)
Total (120 h)	48.5	2.00 (0.52)		
Kidney extract	9.12	0.43 (0.11)	11.2	0.53 (0.14)
Muscle extract	12.4	0.14 (0.04)	13.0	0.15 (0.04)
Fat extract	31.0	0.19 (0.05)	35.0	0.20 (0.056)
Liver extract	2.83	0.24 (0.06)	4.05	0.34 (0.09)
Urine 72 h	2.54	0.13 (0.03)	2.32	0.12 (0.03)

[1] As oxamyl equivalent (mg/kg metabolite in parenthesis).

The predominant metabolic pathway for oxamyl in goats is similar to that for rats. *In vitro* rumen fluid studies demonstrated that oxamyl (and/or its principal plant metabolites) would be extensively metabolized before absorption, and demonstrated the metabolic conversion of oxamyl to thiocyanate. In goat rumen fluid (as in rat) oxamyl was hydrolysed to oxamyl oxime and further to IN-N0079 (oxamyl could also form IN-N0079 directly through a rearrangement). Metabolites resulting from the N-demethylation of oxamyl and/or oxamyl oxime (e.g. IN-L2953 and IN-KP532) were also found in rumen incubations. Similar demethylated metabolites (IN-KP532, IN-00699 and IN-KV998) were found in whole animal studies together with natural products (presumably) resulting from incorporation of one and two carbon units. In ruminant whole-animal studies the detoxification

of IN-N0079 as thiocyanate (through cyanide displacement) played a major role in oxamyl metabolism. The main radioactivity in lactating goats was from thiocyanate and from natural products such as lactose.

<u>Hens</u>. In two studies laying hens were orally administered [^{14}C]oxamyl in capsules once a day for three consecutive days at levels equivalent to 1.2 and 22 ppm in the diet in the first study and 36.3 and 42.5 ppm in the second.

Cresswell and Hopkins (1990) dosed hens (5 per group) with [^{14}C]oxamyl at a level of 0.12 or 2.4 mg/capsule/day (equivalent to 1.2 and 22 ppm in the diet respectively) for three consecutive days. Excreta and eggs were collected daily and pooled by treatment group, and tissues within 24 hours of the last dose. Total radioactive recoveries were 67.2 and 74.5% of the administered dose respectively, 60.1 and 66.4% from the excreta. At the low dose 0.5% was recovered from muscle, fat and liver and 0.1% from eggs. Radioactive residues as oxamyl equivalents from the low-dose group were 0.02 mg/kg in the liver, 0.01 mg/kg in kidney, and <0.01 mg/kg in fat, muscle and eggs, and from the high dose group 0.59 mg/kg (0.4% of the dose) in the liver, 0.37 mg/kg (0.1%) in kidney, 0.03 mg/kg (<0.1%) in fat, 0.12 mg/kg (0.2%) in muscle and 0.11 mg/kg (0.2%) in eggs (Table 9).

Ethyl acetate extracts of the liver and excreta from the low dose group contained about 10% and 5% of the TRR respectively, and acid hydrolysis released an additional 30% and 40% respectively. No oxamyl, oxamyl oxime, IN-N0079 or IN-L2953 was detected in the ethyl acetate extracts or the acid hydrolysates.

Extraction and base hydrolysis of the excreta from the high dose group resulted in the release of one component with retention time (by reverse-phase HPLC) similar to IN-D2708 and two minor compounds that co-eluted with IN-L2953 and oxamyl oxime, but these were not confirmed by a second chromatographic method.

Radioactive residues were readily extractable from the high dose group samples; >85% from eggs, 82% from muscle and 67% from liver. No oxamyl or metabolites structurally related to oxamyl were found in the eggs. Extractable residues in liver and muscle were extremely polar; no oxamyl and little, if any, oxamyl oxime was found. Protease and acid treatment of the liver further demonstrated that the radioactivity was incorporated into natural products.

Table 9. Distribution of radioactivity from laying hens dosed with [^{14}C]oxamyl at levels equivalent to 1 ppm and 20 ppm in the diet (Cresswell and Hopkins, 1990).

Sample	Time, h	Dose level 1 ppm			Dose level 20 ppm		
		% of dose	mean conc., mg/kg	SD, mg/kg	% of dose	mean conc., mg/kg	SD, mg/kg
Excreta	00-24	19.9	0.46	0.04	21.3	8.40	0.34
	24-48	19.6	0.38	0.02	24.1	10.83	0.18
	48-72	20.6	0.45	0.03	21.0	9.05	0.33
Total		60.1			66.4		
Cage wash	00-24	2.0			2.2		
	24-48	1.8			2.0		
	48-72	2.6			2.9		
Total		6.4			7.1		
Eggs	00-24	<0.1	<0.01	<0.01	<0.1	0.11	<0.01
	24-48	N.D.	<0.01	<0.01	0.1	0.11	0.01
	48-72	0.1	<0.01	<0.01	0.1	0.12	0.01
Total		0.1			0.2		
Liver	72	0.3	0.02	<0.01	0.4	0.59	<0.01
Kidney	72	<0.1	0.01	<0.01	0.1	0.37	0.03
Gut	72	0.1			0.1		
Muscle	72	0.2	<0.01	<0.01	0.2	0.12	<0.01
Total		67.2			74.5		

The second study (Scott *et al*., 1994) confirmed the earlier results and identified a major metabolite, thiocyanate, also seen in the goat study. Two groups of white leghorn hens (5 hens in group 1 and 10 in group 2) were orally dosed daily for three consecutive days with [^{14}C]oxamyl at approximately 3.6 mg/day (equivalent to 36.3 ppm in the diet for group 1 and 42.5 mg/kg for group 2). A second group was included in the study because hepatic haemorrhages was found in two of the hens in group 1. No signs of toxicity or toxic effects were found in the group 2 hens.

Excreta and eggs were collected daily and liver, kidney, breast and thigh muscle, and fat 20-23 hours after the last dose. Volatile gases (1.9% of the dose) were only analysed from group 1. The total recovery of the administered dose was 76.2% for group 1 and 79.0% for group 2, most of which was in the excreta (67.4% group 1 and 71.4% group 2). The TRR as oxamyl equivalents in the tissues of group 2 hens was 2.01 mg/kg (0.7% of dose) in the liver, 1.72 mg/kg (0.2%) in kidney, 0.06 mg/kg (0.1%) in fat, 0.44 mg/kg (0.9%) in breast and 0.68 mg/kg (1.3%) in thigh muscle. Residues increased from 0.10 mg/kg (0.02%) and 0.32 mg/kg (0.06%) on day 1 to 1.06 mg/kg (0.13%) and 1.16 mg/kg (0.30%) on day 3 in egg yolks and whites respectively.

Tissues were extracted sequentially with hexane, methylene chloride, ethyl acetate and methanol/water. HPLC analyses indicated that no oxamyl, oxamyl oxime, oxamyl sulfone (IN-U1966), oxamyl sulfoxide (IN-U1967), IN-L2953 or IN-N0079 was present in any tissue or excreta sample. The main metabolite in all the tissue, egg and excreta samples was thiocyanate, representing 13.6% of the TRR in liver, 4.1% in breast and 10.3% in thigh muscle, 26% in egg whites (48-72-h sample), and 33.3% in egg yolks (48-72 h sample) (Table 10). No other residue was identified in tissues or eggs, but oxalic and oxamic acids, urea, the anti-isomer of oxamyl oxime (IN-F3905) and IN-E2816 were tentatively identified in the excreta.

Table 10. Concentrations of thiocyanate in tissues, eggs and excreta of laying hens (Scott *et al*., 1994).

Sample	% of TRR[1]	mg/kg[2]	
Excreta			
0 – 24 h	6.2		
24 – 48 h	2.9		
48 – 72 h	3.5		
Liver	13.6	0.273	(0.072)
Egg yolk			
0-24 h	33.5	0.035	(0.009)
24-48 h	17.6	0.086	(0.023)
48-72 h	33.3	0.353	(0.093)
Egg white			
0-24 h	46.5	0.149	(0.039)
24 – 48 h	36.3	0.330	(0.087)
48-72 h	26.0	0.301	(0.080)
Breast muscle (light meat)	4.1	0.018	(0.005)
Thigh muscle (dark meat)	10.3	0.070	(0.019)

[1] Percentage of total administered dose
[2] Oxamyl equivalents (mg/kg thiocyanate in parenthesis).

The predominant metabolic pathways for oxamyl in poultry are similar to those in goats and rats. The main metabolite in laying hens was thiocyanate. No intact oxamyl was present in excreta, tissues or eggs. The proposed metabolic pathways for oxamyl in laying hens involves the formation of oxamyl oxime by hydrolysis of the carbamate moiety. Oxamyl oxime or oxamyl itself is then converted to IN-N0079, then to cyanide which is rapidly detoxified by conversion to thiocyanate.

Metabolic pathways in animals

Oxamyl was rapidly and extensively metabolized in livestock (goats and poultry) and laboratory animals (rats). The initial metabolic pathways in goats, hens and rats involve hydrolysis to oxamyl

oxime (IN-A2213), or rearrangement to IN-N0079 (IN-N0079 could also be formed directly), which is then converted to IN-D2708 and ultimately incorporated into natural products. Minor metabolites IN-L2953, IN-KP532 and IN-D1409 from demethylation reactions were also observed. In the livestock the detoxification of IN-N0079 as thiocyanate (through cyanide displacement) is a major route. Most of the terminal radioactivity in lactating goats and laying hens was in thiocyanate and natural products such as lactose. In rats, conversion of IN-N0079 to thiocynate was not observed, but conjugates of the principal metabolites were found. Figure 2 shows the proposed metabolic pathways.

Figure 2. Proposed predominant metabolic pathways of oxamyl in animals.
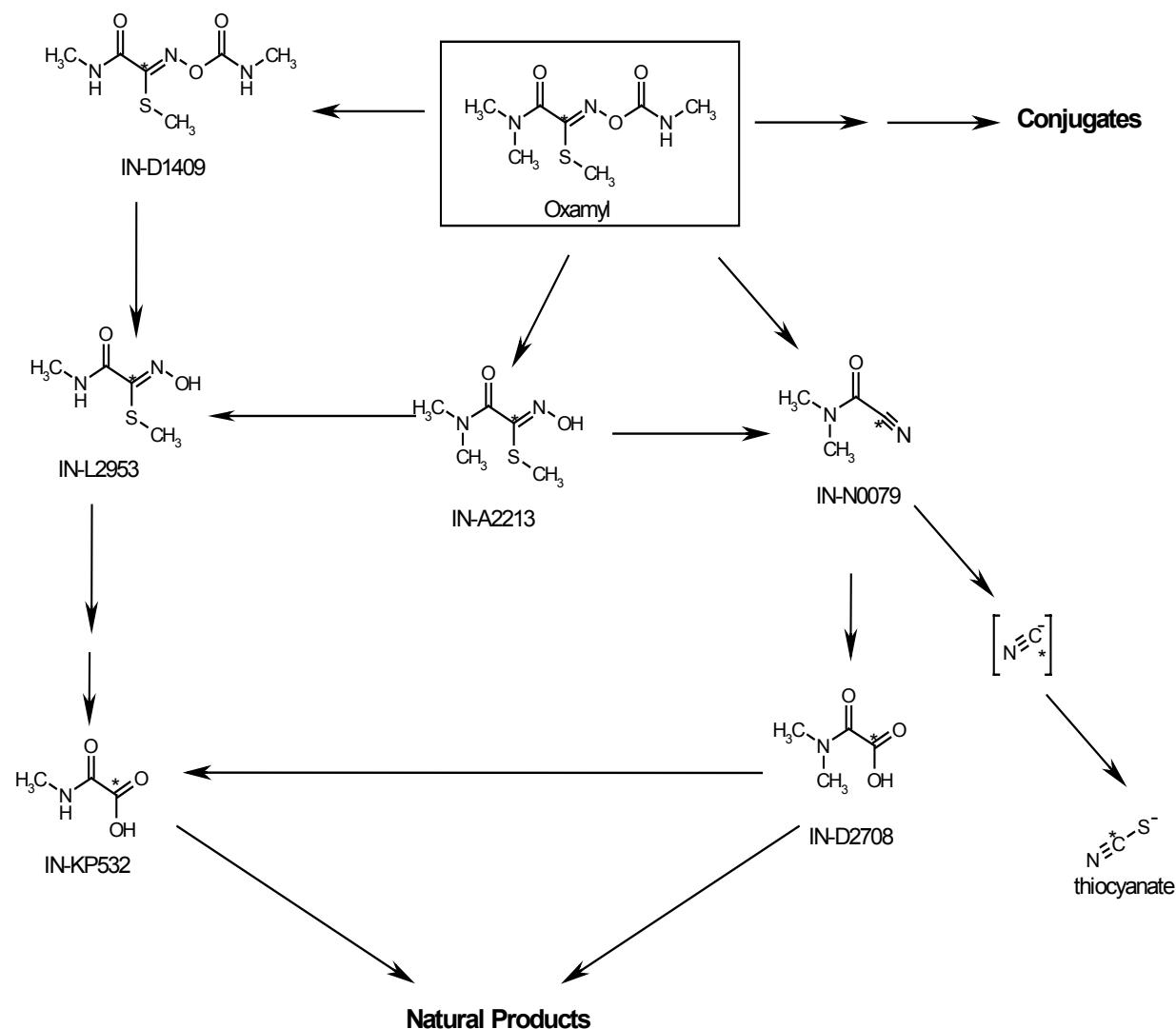
Plant metabolism

In studies on potatoes, peanuts, tobacco, apples and oranges (Brown *et al*., 2001, Harvey *et al*., 1978) plants were treated with [^{14}C]oxamyl applied either direct to the foliage, or to the fruit or soil.

<u>Oranges</u>. Harvey *et al*. (1978c) brushed immature oranges (Hamlin variety) in the field on the tree with a solution of [^{14}C]oxamyl equivalent to 1.4 g ai/l in 0.2% Tween 20 and harvested the fruit at maturity 6 weeks later. The TRR in the oranges was equivalent to 2.5 mg/kg, 82% in the peel (4.7 mg/kg oxamyl equivalents), of which 96% was extractable with methanol. The juice contained 18% (0.8 mg oxamyl equivalents/kg), with small amounts of oxamyl, oxamyl oxime, IN-N0079, and a large polar fraction. Glucose conjugates of oxamyl oxime and IN-L2953 were major components of

the polar fractions from both peel and juice. The ^{14}C residues on a whole fruit basis consisted of oxamyl (9% of the TRR), oxamyl oxime (6%), IN-N0079 (20%), oxamyl oxime glucoside (35%), IN-L2953 glucoside (22%) and unidentified polar compounds.

Apples. Harvey *et al*. (1978) also brushed apples (Jonathan variety) on the tree in the field with a solution of [^{14}C]oxamyl equivalent to 1.2 g ai/l and harvested the fruit at maturity 47 days later. The apples were washed with water and divided into peel and interior fractions which were extracted separately with methanol. The radioactivity was evenly distributed and 98% was extractable with methanol. Most of the metabolites (77%) were organosoluble (16% oxamyl, 42% oxamyl oxime and 17% IN-N0079) and the remainder was a water-soluble polar fraction containing polysaccharide conjugates of oxamyl oxime and IN-L2953. The monosaccharide conjugates were not found.

Tomatoes. Harvey *et al*. (1978) used a micropipette to spot 0.37 mg ai/fruit in 300 µl of a solution containing 0.2% Tween 20, evenly over the entire surfaces of green tomatoes (Bonny Best variety) attached to the vine grown in a greenhouse. Tomatoes were sampled 7, 11, 14 and 21 days after treatment, washed with distilled water to remove surface residues, macerated with methanol and filtered. Radioactive residues were determined in the dried pulp, the aqueous wash and the methanol extract before and after ethyl acetate partitioning. The concentration of total radioactivity in the tomatoes remained essentially constant throughout but the surface residues decreased. Oxamyl constituted 81.2% of the extractable residue in the tissues 7 days after treatment, and after 14 days the main component was oxamyl (36% of the applied radioactivity on the surface and 23% internal). The main metabolite 14 days after treatment was oxamyl oxime (13%; 12% internal): others included IN-N0079 (4%), the glucose conjugate of oxamyl oxime (5%) and polar metabolites or natural products (19%). Oxamyl constituted 60.4% of the ethyl acetate fraction after 21 days.

Potatoes. The metabolism of [^{14}C]oxamyl in tubers (Kennebec variety) was investigated in two field studies in Delaware, USA (Harvey *et al*., 1978). In the first four seed potatoes were planted in a furrow 10-cm wide which had been sprayed earlier the same day at a rate of 3.36 kg ai/ha. In the second plants were treated with 5 foliar applications at approximately 10-day intervals until 14 days before harvest.

All tubers were rinsed in water, divided into peel and interior fraction (flour), and lyophilized. The freeze-dried flour from tubers of the plants sprayed foliarly was sequentially extracted with water, methanol, ethyl acetate and tetrahydrofuran (THF). Separate portions of flour were incubated with anhydrous methanolic HCl, β-glucosidase and 2% sulfuric acid. The sulfuric acid extract was purified by silica gel chromatography, then silylated and analysed by GC-MS. The flour contained approximately 90% of the TRR in the potato: exhaustive extraction with aqueous and organic solvents released little radioactivity, mild acid hydrolysis released 39% of the TRR as oxamyl oxime and IN-L2953 while β-glucosidase treatment indicated glucose conjugates of oxamyl oxime and IN-L2953. Strong acid hydrolysis followed by LC clean-up, silylation and GC-MS analysis indicated that at least 35% of the radioactivity had been incorporated into starch.

In a recent greenhouse study by Brown *et al*. (2001) 25 l pots were planted with seed potatoes (Red Pontiac variety) and the soil was treated with a single application of a solution of [^{14}C]oxamyl containing inert ingredients to simulate a "Vydate" 10 l formulation at a rate of 8 kg ai/ha. Mature tubers and foliage were collected 127 days later, and the tubers rinsed with water, divided into peel and peeled potatoes. The peeled potatoes, peel and foliage were each extracted with methanol and aqueous methanol. The concentrated foliage extract was hydrolysed with β-glucosidase and acid to further elucidate the metabolic pathway in the plant. 81% of the TRR was in the peeled potatoes. The TRRs in the peel, peeled potatoes and treated foliage were 1.02, 0.78 and 1.25 mg/kg oxamyl equivalents respectively. The main residue in the peel and peeled potatoes (68% and 70.8% of the TRR respectively) was IN-D2708. No oxamyl or oxamyl oxime was detected in either. Identification was confirmed by co-elution with ^{14}C-IN-D2708 and by co-TLC of the methylated compounds. In the foliage 78.3% of the TRR (1.18 mg oxamyl equivalents/kg) was extracted and minor residues

included oxamyl oxime (5.9% of the TRR, 0.09 mg oxamyl equivalents/kg), oxamyl (1.1% of the TRR, 0.02 mg/kg) and IN-D2708 (1.9% of the TRR, 0.03 mg oxamyl equivalents/kg). The main foliar metabolite, 45.7% of the TRR, 0.69 mg oxamyl equivalents/kg, was water-soluble and resistant to enzyme and acid hydrolysis. It was eluted just before oxamyl oxime and behaved similarly to the glucose conjugate of oxamyl oxime found in field-grown potato plants treated foliarly.

Peanuts. In the first of two field studies in North Carolina, USA (Harvey *et al*., 1978) the leaves of the plants were treated twice with [^{14}C]oxamyl at 2.24 kg ai/ha applied three weeks after plant emergence and 4 weeks later. Just before the second application immature plants were taken for analysis, and at maturity plants were harvested and the peanuts separated from the hay. The green hay and the immature plants were extracted separately with ethyl acetate and then with methanol for TLC analysis. The methanol extract from the hay contained the most radioactivity and was purified by column chromatography. A portion of the polar column effluent was per-acetylated and analysed by LC-MS. The remaining portion of the polar metabolite fraction was incubated with β-glucosidase, purified by Porasil chromatography and analysed by LC, GC and MS. Peanuts were analysed for total radioactivity and extracted with methanol, then purified as above. Peanut residues were further extracted with hexane and Soxhlet extracted with methanol, then incubated with a mixture of cellulase enzymes.

In the second study a single in-furrow pre-plant treatment at 1.68 kg ai/ha was followed by two foliar applications at 1.12 kg ai/ha each 24 and 76 days after planting. One plant was sampled 40 days after the last application and the rest after 70 days. The 40-day plant was extracted and the extract purified using the procedure described for the polar metabolite fraction above. Mature peanuts were shelled, macerated with hexane, ethyl acetate and methanol, then analysed by LSC. The polar extracts of the kernels were refluxed with 1N HCl then extracted with ethyl acetate.

In both trials oxamyl was almost completely metabolized with less than 0.5% of the TRR remaining as oxamyl or oxamyl oxime. Further metabolism in the plants was through carbohydrate conjugation and *N*-demethylation (either before or after conjugation). The main metabolites were the glucose conjugates of oxamyl oxime and IN-L2953, which were further conjugated with additional carbohydrate residues.

Tobacco. Oxamyl metabolism was investigated in three laboratory experiments (Harvey *et al*., 1978).

In the first the foliage of two plants (Xanthi variety) was treated with an aqueous solution of [^{14}C]oxamyl in 0.2% Tween 20 at a rate of 10 mg/plant. The plants were placed in a plant-growth chamber (one in a glass bell jar to collect volatiles), and harvested 7 or 15 days after treatment. The foliage was washed with water to remove surface residues, the ^{14}C in the top and root tissues were was counted, and the leaves extracted and analysed by gel permeation and adsorption chromatography, and acid and alkaline hydrolysis. In the 15-day plant approximately 38% of the applied radioactivity was in the leaves. The main component was oxamyl, 21.1% of the applied radioactivity, and the metabolites were oxamyl oxime (1.9% of the applied), a glucose conjugate of oxamyl oxime (14.1%) and IN-D2708 (1.1%).

In the second study one 3-week-old plant was planted in soil containing [^{14}C]oxamyl incorporated at a rate of 6 mg/kg. After 3 weeks the plant was harvested. The aerial portion was extracted with methanol and the extract analysed by TLC. Approximately 10% of the applied radioactivity was in the tissues, mainly associated with oxamyl (2% of applied), oxamyl oxime (1.4% of applied) and a polar fraction (4.6% of applied).

In the third study potted seedlings were sprayed to run-off with aqueous solutions of oxamyl at concentrations of 0.48 g/l and 2.4 g/, and sampled 0, 3, 7, 14, 21 and 27 days after treatment. The tops and roots were extracted, concentrated and assayed for acetylcholinesterase inhibitory activity with a half-life of 19 days.

In a further study young seedlings were transplanted into a field plot in North Carolina, USA, previously treated with [^{14}C]oxamyl at 6.72 kg ai/ha (Harvey *et al*., 1978). Plants were thinned to a normal commercial distance about one month later and two were left to maturity. The immature seedlings contained 65-95% of the TRR as extractable residues (29-41 mg oxamyl equivalents/kg). The TRR in the mature plants was 0.5 mg oxamyl equivalents/kg, of which about 80% was extractable. Oxamyl and oxamyl oxime accounted for approximately 10% and 1% of the extractable residues in the seedlings respectively, and the polar remainder included glucose conjugates of oxamyl oxime and IN-L2953.

Metabolic pathways in plants

The metabolic pathways of oxamyl are similar in potatoes, peanuts, tobacco, apples, oranges and tomatoes. It is extensively metabolized in plants and the radiolabel was ultimately found in natural plant products. The metabolic routes include hydrolysis of the methylcarbamoyl group to yield the non-insecticidal oxamyl oxime (IN-A2213) which is conjugated with glucose. Oxamyl oxime is demethylated before or after glucose conjugation to give IN-L2953 and/or its glucose conjugate. Conjugation of the glucosides of oxamyl oxime and IN-L2953 with additional sugar residues was also observed. Oxamyl oxime (or oxamyl) is also metabolized to IN-N0079, which is further metabolized to IN-D2708 and the label ultimately incorporated into natural plant products. Figure 3 shows the proposed metabolic pathways.

Figure 3. Proposed metabolic pathways of oxamyl in plants.
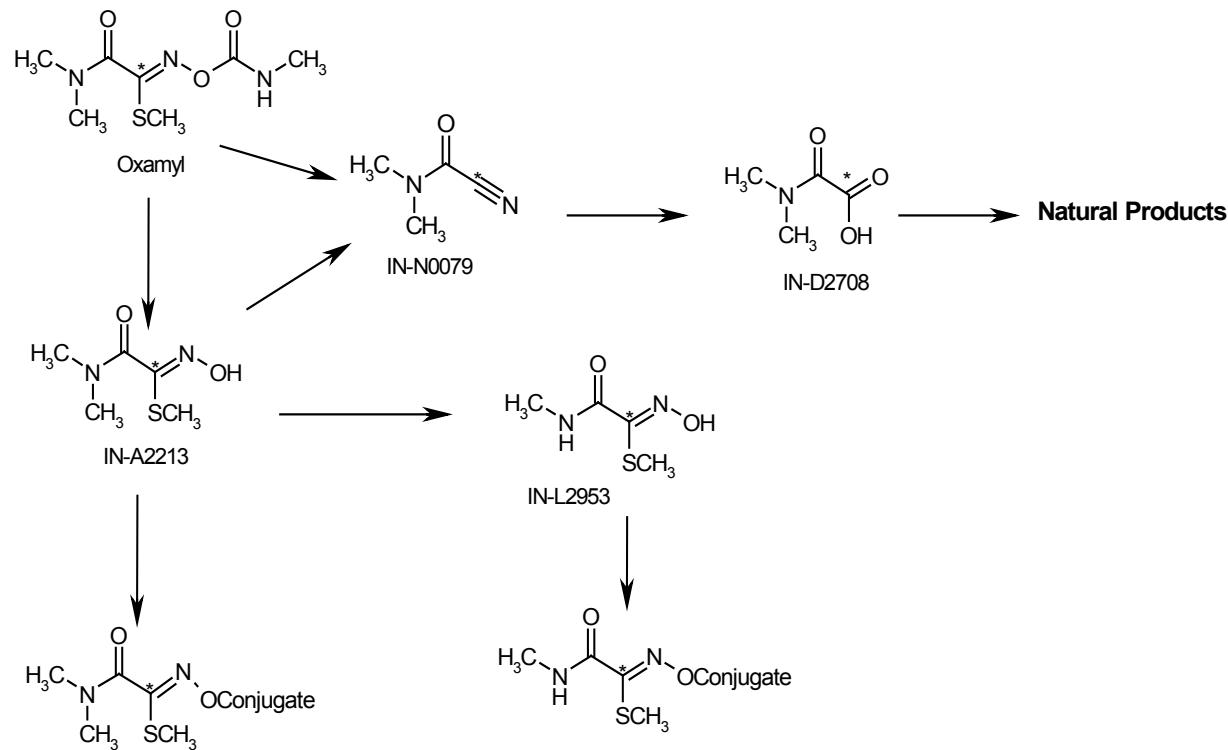
Environmental fate in soil

Aerobic degradation

The aerobic degradation of [^{14}C]oxamyl was determined in a moist, acidic silt loam soil (pH 4.7) contained in a flow-through glass chamber connected to caustic alkali traps (Harvey and Han, 1978). A full description of the incubation apparatus is given by Harvey and Pease (1973). The soil was

treated at a rate equivalent to 4.48 kg ai/ha, and after 42 days extracted with water and the extract analysed by radio-TLC. Total radioactivity was determined in the volatile traps after 21 and 42 days of incubation and $^{14}CO_2$ by precipitation with barium chloride. Substantial mineralization was observed after 42 days with 51% of the applied dose recovered as $^{14}CO_2$. Polar materials (11%), oxamyl (4%), and the oxime hydrolysis product, oxamyl oxime (<1%), were found in the soil extract. The soil was further extracted with hot caustic alkali, which removed most of the residues remaining after the initial water extraction. These residues were distributed in the organic matter primarily between the fulvic acid and α-humus fractions.

In a similar trial without volatile traps (Hawkins *et al.*, 1989) another acidic silt loam soil (pH 4.6, 3.7% organic matter) was treated with [^{14}C]oxamyl at 16 mg/kg and incubated for 14 days in a flow-through chamber in darkness at 25°C at a moisture content of 75% of 33 kPa. Samples were collected at treatment and after 4, 8, 10, 12 and 14 days. After methanol and methanol/water extraction, the extracts were analysed by radio-TLC. The main products were $^{14}CO_2$ and the acid IN-D2708 (10.3% of applied ^{14}C at 14 days).

In another study a sandy clay loam soil (pH 7.7, 1.5% organic matter) was treated with [^{14}C]oxamyl at 9.5 mg/kg and incubated in the dark at 25°C at a moisture content of 70-75% of 33 kPa for 51 days under aerobic conditions (Spare, 1991). Humidified air was passed through the test systems for at least one hour each working day. Caustic volatile trapping solutions were replaced at each sampling. After extraction with methanol/water and methanol, the extracts were analysed by radio-TLC and radio-HPLC. Oxamyl oxime was the initial degradation product, reaching a maximum of 24.3% of the applied radioactivity on day 10. The acid IN-D2708 was subsequently formed and reached a peak of 20.3% on day 21. The mineralization product $^{14}CO_2$ was the predominant degradation product and reached 45.3% of the applied radioactivity by day 51. Unextractable residues rose steadily throughout incubation to 24.2% in parallel with the evolution of $^{14}CO_2$.

In a recent study using a loam soil (pH 7.0, 2.4% organic matter) Smyser and Mattson (2000) applied [^{14}C]oxamyl at a concentration of 2 mg/kg and incubated the soil with a moisture content of 40% of maximum water holding capacity (0 bar or ~1 kPa) for 59 days in a flow-through system in the dark at 20°C. Caustic and ethylene glycol traps were used to collect $^{14}CO_2$ and volatile organic components. At various intervals soil samples were extracted with acetonitrile/water and analysed by LSC and radio-HPLC. Caustic trap contents were analysed by LSC before and after the addition of Na_2CO_3 and $BaCl_2$ to precipitate $^{14}CO_2$. Solvent-extracted soil was analysed by combustion and LSC. Unextractable residues were characterized by fractionation with strong acid and base solutions. The hydrolysis product oxamyl oxime was the initial degradation product and reached a maximum of 15% of the applied radioactivity on day 1, and the acid IN-D2708 reached a peak of 29.6% on day 14. Oxamyl oxime and IN-D2708 were the only degradation products in the soil extracts above 1% of the applied radioactivity. The predominant degradation product was $^{14}CO_2$ resulting from mineralization of oxamyl *via* oxamyl oxime and IN-D2708. By day 59 approximately 100% of the applied radioactivity was recovered as $^{14}CO_2$. Unextractable residues reached a plateau at approximately 20% by day 31. After further treatment of the residue from day 31 with strong acid and base, the radioactivity was found to be primarily associated with the fulvic and humic acid fractions in equal proportions.

In a further study Mattson and Smyser (2000) applied [^{14}C]oxamyl at a concentration of 2 mg/kg to a neutral silt loam (pH 7.0, 0.4% organic mater), a slightly alkaline silt loam (pH 7.8, organic matter 2.1%), and an acidic silty clay loam (pH 4.8, 4.4% organic matter). The soils were incubated in a flow-through system in the dark at 20°C with a soil moisture content of 40% to 50% of maximum water holding capacity (0 bar, ~1 kPa) for up to 179 days. Caustic and ethylene glycol traps were used to collect $^{14}CO_2$ and organic volatiles. The neutral silt loam was also incubated at 10°C to evaluate the effect of temperature on degradation. At various intervals soil samples were extracted with acetonitrile/water for analysis by LSC and radio-HPLC. Solvent-extracted samples were analysed by combustion and LSC. Caustic trap contents were analysed by LSC before and after the

addition of Na_2CO_3 and $BaCl_2$ to precipitate $^{14}CO_2$. The initial degradation product in each soil was the oxime IN-A2213, whose maximum levels were 7.6% (day 60) of applied radioactivity in the acidic soil, 51.0% (day 7) in the neutral soil, and 24.9% (day 2) in the slightly alkaline soil. The acid IN-D2708 was also found in the neutral and slightly alkaline soils, peaking at 25.7% (day 11) in the former and 34.7% (day 10) in the latter. In the acidic soil IN-D2708 was not found, probably owing to the slow formation of the oxime precursor IN-A2213, which was observed only at low levels. Substantial mineralization occurred in all three soils. At the end of the incubation, 73.1% of the applied radioactivity had been recovered as $^{14}CO_2$ from the neutral soil (day 60), 76.1% (day 31) from the slightly alkaline soil, and 25.6% (day 120) from the acidic soil. Unextractable residues ranged from 20.7% to 24.1% in the three soils at the end of the incubations. In the neutral soil at 10°C, the degradation pattern was the same as at 20°C. Peak levels of oxamyl oxime were 33.8% (day 32), IN-D2708 39.5% (day 90), $^{14}CO_2$ 81.0% (day 179) and unextractable residues 17.7% (day 179).

The route of oxamyl degradation in aerobic (oxidizing) aquifers was assessed by incubating two representative water-saturated oxidizing subsoils with [^{14}C]oxamyl (Dean, 2000). The soils were collected from within a shallow water table aquifer beneath agricultural fields in The Netherlands at a depth of approximately two to three metres. The "Roswinkel" subsoil was a sand with pH (0.01 M $CaCl_2$) of 5.9 and organic matter content of 0.2%. The "Eeserveen" subsoil was a more acidic sand with a pH of 4.5 and organic matter content of <0.1%. Both had a nitrate:ammonim nitrogen ratio of >3.5 indicating oxidizing conditions. Ferrous iron levels in ground water taken from the subsoil collection bore holes were low, <0.8 mg/l, also indicative of oxidizing conditions. The samples were equilibrated for one week in the laboratory and treated with [^{14}C]oxamyl at a concentration of 0.5 mg/kg, equivalent to approximately 1.6 to 1.9 mg/l in the soil water, then incubated in the dark in a humidified air flow-through system at 10°C for 120 days. At various intervals samples were exhaustively extracted with methanol/water and methanol and analysed by LSC and radio-HPLC. Caustic traps were used to capture $^{14}CO_2$ and ethyl digol (2-(2-ethoxy)ethoxyethanol) traps for any organic volatiles. The pH and reduction/oxidation potential of the test systems were monitored throughout the incubations. In "Roswinkel" subsoil, the average pH was 6.1 and the average redox potential +321 mV with little variation. Oxamyl oxime was observed at levels up to 68.8% (day 120). $^{14}CO_2$ was the only other product at levels above 5% (7.4%, day 120). Unextractable radioactivity reached a maximum of 4.4% on day 60. In Eeserveen subsoil the average pH was 4.6 and the average redox potential +400 mV. Little conversion of oxamyl was observed under these acidic conditions. Levels of oxamyl oxime reached only 1.0% by day 120 and $^{14}CO_2$ was not detected.

To assess the role of microbes in the degradation of oxamyl Dulka and Julis (1978) incubated samples of sterile and fresh silt loam soils (pH 6.4, 2.75% organic matter) treated with [^{14}C]oxamyl at concentrations of 4 mg/kg and 20 mg/kg in biometer or Erlenmeyer flasks aerobically in the dark at 25°C and 70% of normal water-holding capacity. The biometer flasks were equipped with caustic volatile traps at various intervals up to a maximum 51 days soil samples were ultrasonically extracted with methanol and analysed by LSC, radio-TLC, radio-HPLC, and GC-MS. In additional experiments the effect of representative soil microbes on degradation after treatment at the lower rate was investigated by inoculating sterilized soil. The differences in mineralization between the sterilized and the fresh soil were substantial. After 51 days only 4-8% of the applied radioactivity was recovered as $^{14}CO_2$ from sterile and 48%-63% from fresh soil. The main degradation products in fresh soil at both rates were oxamyl oxime and IN-D2708 at maximum levels of 2%-4% of the applied radioactivity. Extracts of the autoclave-sterilized control soil and inoculated soils contained oxamyl oxime and IN-D2708, as well as varying quantities of the nitrile IN-N0079 and the amide IN-T2921. However the authors suggest that the presence of IN-N0079 and IN-T2921 in sterilized and inoculated soil but not in fresh may have been an artefact and that these compounds must be products of the interaction of oxamyl with soil chemicals produced during the harsh sterilization procedure. Of the six organisms tested only the soil fungus *Sclerotinia sclerotium* was observed to decompose [^{14}C]oxamyl at a similar rate to fresh soil organisms.

In three field trials in the eastern USA (Harvey and Han, 1978) stainless steel cylinders (10.2 cm i.d. x 38.1 cm long) containing a silt loam in Newark, Delaware; a loamy sand in Clayton, North Carolina; and a fine sand in Bradenton, Florida (soil pH and organic matter not given) were driven into undisturbed soil. Using a method described by Harvey and Pease (1973) the top 2.5 cm layer of soil was temporarily removed from the columns, and the exposed surface was treated with [^{14}C]oxamyl at a rate equivalent to 6.72 kg ai/ha. The removed soil was replaced and the cylinders exposed to normal weather conditions for three to five months. Cylinders at the North Carolina and Florida sites were fitted with leachate collectors. At intervals cylinders were removed from the ground and soil sections were extracted with water and analysed by radio-TLC. The hydrolysis product oxamyl oxime was observed at each site. Maximum levels ranged from 0.3% to 13.3% of applied radioactivity at the first sampling after one week and decreased thereafter. A polar fraction ranged from 10.3% to 67.0% at the first sampling and had decreased by the second sampling at one month. $^{14}CO_2$ estimated by difference represented 79.1% to 93.3% by the third and last sampling at three months (Delaware and Florida) or five months (North Carolina). Unextractable residues ranged from 6.4% to 14.1%. Sodium hydroxide removed 76% of the residues that were unextractable with water from the five-month sample of loamy sand soil. Most of the residues were distributed in the hymatomelanic acid and α-humus fractions. Only minor amounts of radioactivity (<6%) were found in the leachate collectors, and were mainly from oxamyl oxime.

The impact of oxamyl on soil fungal and bacterial populations and microbial respiration was studied in fine sand, silt loam, and sandy loam soils (Peeples, undated). After treatment with oxamyl at 10 mg/kg, soils were incubated at 27°C and 65% of field capacity moisture for nine weeks. Samples were placed in flasks stoppered with foam plugs, and controls were incubated in the same manner. 1, 2, 4, and 8 weeks after treatment, the population of fungi and bacteria was determined in treated and control samples using dilution plates. Another set of samples was incubated under the same conditions in sealed flasks containing a caustic solution to trap CO_2. The evolution of CO_2 was measured titrimetrically after 1, 2, 4, 8, and 9 weeks. At the eighth week a glucose nutrient solution was added. Oxamyl had no effect on the fungal and bacterial populations. There was an initial rise in both populations compared to the control in the fine sand and silt loam soils, but this difference disappeared within two weeks. Average fungal populations were 3.8×10^4 in untreated and 4.0×10^4 in treated samples, and average bacterial 3.7×10^6 and 4.5×10^6 respectively, for the three soils during 8 weeks. There were no differences in the pattern or magnitude of carbon mineralization between the untreated and treated soils. The average amount of CO_2 produced in eight weeks was 980 mg/kg in untreated and 930 mg/kg in treated soil. After the nutrient medium was added the average cumulative CO_2 at nine weeks rose to 3350 mg/kg in untreated and 3300 mg/kg in treated soils.

The effect of oxamyl on nitrifying soil bacteria was assessed in two soils (Han, undated). 100-g samples of air-dried silt loam soil (4.2% organic matter, adjusted to pH 6.7 with CaCO$_3$) and loamy sand soil (0.7% organic matter, adjusted to pH 6.7 with CaCO$_3$) were inoculated with 1 g fresh dried garden soil and incubated at 30°C and 50% total water-holding capacity. After a two-week equilibration 200 mg/kg nitrogen as ammonium sulfate was added together with oxamyl at 0.5 mg/kg or 5 mg/kg. Ammonium sulfate was added to a second series of untreated samples and a third series was not treated with ammonium sulfate or oxamyl to allow for normal nitrification. Over eight weeks every three to seven days, samples were taken for determination of nitrate. No differences were found in the levels between control and oxamyl-treated samples in the loamy sand soil at any time. By day 53, nitrate levels ranged from 92 mg/kg to 94 mg/kg in both control and treated samples. In the silt loam soil, average nitrate levels were lower on days 1, 4, 11, and 15 at the 5 mg/kg level than in the control, but these differences were no longer apparent by day 22. By day 28 levels were 119 to 120 mg/kg in control and treated samples. The results indicate that oxamyl had no long-lasting effects on soil nitrification.

Degradation pathway in aerobic soil

Pathways were consistent throughout a range of soils despite various extractants (water, methanol/water, acetonitrile/water). Base-catalysed hydrolysis of the methylcarbamoyl moiety to form the corresponding oxime IN-A2213 is the initial step followed by formation of the acid IN-D2708 in a linear degradation pathway. Hydrolysis can occur as a result of abiotic and/or biotic action with more oxamyl oxime formed under alkaline conditions, as found in laboratory studies of hydrolysis (Harvey and Han, 1978; McNally and Wheeler, 1988b). IN-A2213 is readily mineralized to carbon dioxide, the predominant degradation product. Mineralization is also demonstrated by incorporation of residues into the soil organic matter fractions. In aerobic (oxidizing) saturated zone subsoils, the degradation path is the same as in aerobic topsoil, with the formation of oxamyl oxime being dependent upon pH. Figure 4 shows the proposed pathway in aerobic soils.

Figure 4. Proposed pathway for the degradation of oxamyl in aerobic topsoil and aerobic (oxidizing) saturated subsoils.

oxamyl

IN-A2213 (oxime)

IN-D2708 (acid)

CO_2, unextractable residues
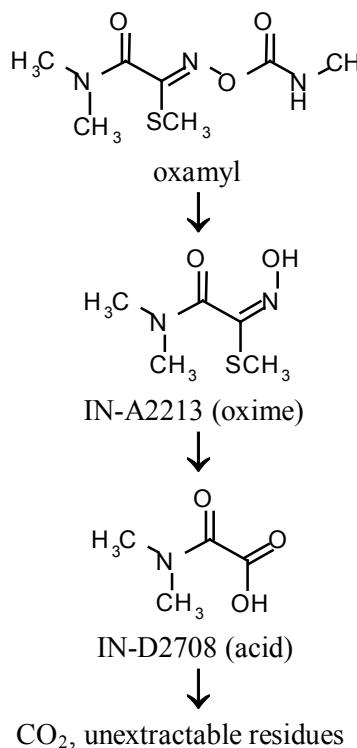
Anaerobic degradation

In a study by Harvey and Han (1978) an acidic silt loam soil (pH 4.7) treated with [^{14}C]oxamyl at a concentration of 6 mg/kg and the moisture adjusted to 87% of field capacity was contained in a flow-through glass chamber connected to caustic alkali traps. A full description of the apparatus is given in Harvey and Pease (1973). Nitrogen was passed through to induce anaerobic conditions. After 42 days' incubation, the soil was extracted with water and the extracts analysed by radio-TLC. Total radioactivity was determined in the traps and $^{14}CO_2$ by precipitation with barium chloride. Oxamyl oxime accounted for 41% of the applied radioactivity, oxamyl for 8%, a polar fraction for 42%, $^{14}CO_2$ for 3%, and unextracted residue for 6%. When compared to the aerobic experiment with the same soil, it appears that oxamyl hydrolysis was not inhibited by the anaerobic conditions but mineralization to CO_2 was slower.

In a study by Hawkins *et al.* (1989) another acidic silt loam soil (pH 4.6, 3.7% organic matter) in a flow-through chamber with traps was treated with [^{14}C]oxamyl at 20 mg/kg, aerobically

incubated in darkness at 25°C at a moisture content of 75% of 33 kPa for 20 days, then flooded with distilled water and the flow-through gas changed to nitrogen before anaerobic incubation for a further 60 days. Samples were collected on days 0 and 20 of the aerobic phase and on days 30 and 60 of the anaerobic phase. Trapping solutions were connected to the incubation systems to collect organic volatiles and $^{14}CO_2$. The soil was extracted with methanol and methanol/water, and the water phase with ethyl acetate. Analysis was by radio-TLC. The maximum levels of oxamyl oxime were 1.3% to 1.5% of the applied radioactivity after 30 days of anaerobic incubation, when the acid IN-D2708 was the predominant product reaching 78.8% to 86.0%. $^{14}CO_2$ evolution was 10.3% after 20 days of aerobic incubation and 13.8% after a further 60 days of anaerobic incubation indicating that mineralization was slower under anaerobic conditions. Unextractable residues were 8.1% to 8.7% after 20 days aerobic and 9.1% to 14.1% after a further 60 days anaerobic incubation.

Spare (1991) applied [^{14}C]oxamyl at a concentration of 9.5 mg/kg to a sandy clay loam soil (pH 7.7, organic matter 1.5%) in a flow-through apparatus. Aerobic incubation proceeded in the dark at 25°C and 70-75% of field capacity soil moisture for 11 days, after which samples were flooded with deionised water, purged with nitrogen, then incubated another 60 days anaerobically. Traps were connected to the incubation system to collect organic volatiles and $^{14}CO_2$. At intervals samples were extracted with methanol/water, and methanol and analysed by radio-TLC and radio-HPLC. Oxamyl oxime and the acid IN-D2708 were the primary degradation products, the former peaking at 69.5% of the applied radioactivity on day 20 of the anaerobic incubation and the latter at 23.1% on day 32. $^{14}CO_2$ and unextractable residues rose steadily reaching 12.0% and 18.4% respectively by the end of the anaerobic incubation. In a supplementary study using the same soil and conditions oxamyl oxime, IN-D2708, and $^{14}CO_2$ were again observed as the main degradation products (Spare, 1992).

The route of degradation in anaerobic (reducing) aquifers was assessed in the laboratory with two representative water-saturated reducing subsoils (Hooghalen loamy sand with strongly acidic pH (0.01 M CaCl$_2$) of 4.1 and an organic matter content of 8.4%, and Emmer Compascuum acidic sand with a pH of 4.4 and an organic matter content of 0.2%) from within a shallow water table aquifer beneath agricultural fields in The Netherlands (Dean, 2000). Both subsoils had a nitrate:ammonim nitrogen ratio of <0.2 indicating reducing conditions. Samples were collected from approximately 2-3 m below the surface and the resulting bore holes and the containers were flooded in the field with argon gas to maintain anaerobic conditions during collection and transport. Ferrous iron levels in ground water taken from the bore holes were approximately 5 mg/l, also indicative of reducing conditions. The samples were equilibrated in the laboratory for approximately two weeks, treated with [^{14}C]oxamyl at a concentration of 0.5 mg/kg, equivalent to approximately 0.4 to 2.4 mg/l in the soil water, and incubated in the dark in a humidified nitrogen flow-through system at 10°C for 60 days. At various intervals samples were exhaustively extracted with methanol/water and methanol for analysis by LSC and radio-HPLC. Caustic traps were attached to the flow-through system to capture $^{14}CO_2$ and ethyl digol traps to capture any organic volatiles. The pH and reduction/oxidation potential of the test systems were monitored throughout. In Hooghalen subsoil the average pH was 4.5 and the average redox potential +193 mV, indicating a moderately reducing environment, and in Emmer Compascuum average pH was 4.8 and the redox potential remained below +200 mV for the first 46 days (mean +164 mV), then rose to approximately +300 mV indicating a change to more oxidizing conditions. In both subsoils oxamyl was very rapidly converted to the nitrile IN-N0079 with maxima of 67.6% at day 0.5 and 71.2% at day 0 of the applied radioactivity. Levels of the acid IN-D2708 rose during incubation reaching 33.0% to 47.9% by day 60. Various other products included the amide IN-T2921 (maxima in the two subsoils of 14.4% on day 1 to 19.5% on day 4), but these were generally transient. $^{14}CO_2$ and other volatile residues were only minor products (<3%). Unextractable residues reached 11.0% to 32.2% by day 60. Oxamyl oxime was not found in these acidic subsoils, which is consistent with the hydrolysis study of McNally and Wheeler (1988b). Rapid reduction of oxamyl to the nitrile IN-N0079 in the presence of ferrous iron is consistent with the work of Bromilow *et al.* (1986) and Strathmann and Stone (2001).

Degradation pathways in anaerobic soil

In anaerobic topsoils the degradation of oxamyl is as in aerobic topsoils: hydrolysis oxime IN-A2213 followed by formation of the acid IN-D2708, ultimately leading to mineralization to carbon dioxide and incorporation into the soil organic matter. Degradation is not affected by anaerobic conditions but mineralization is slower. In anaerobic (reducing) subsoils where ferrous iron is present at sufficient levels oxamyl is readily converted to the nitrile IN-N0079 by Fe[II]-catalysed reduction. Depending on the pH and Fe[II] level in the system, oxamyl degradation may proceed in parallel *via* reduction to the nitrile and hydrolysis to the oxime. Figure 5 shows the proposed pathways.

Figure 5. Proposed pathways for the degradation of oxamyl in anaerobic topsoils and anaerobic (reducing) saturated subsoils.
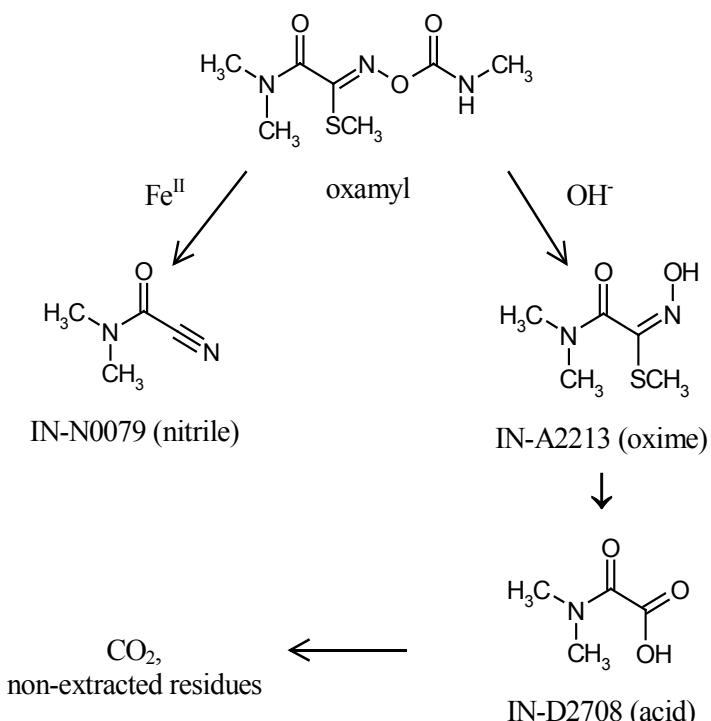
Minor and/or transient products not included.

Degradation half-life in soil under aerobic and anaerobic conditions

In this section the term half-life refers to a first-order or pseudo first-order kinetic process calculated as $ln(2)/k$, where "ln" is the natural log and "k" is the first-order rate constant with units of days^{-1}. The term DT-50 refers to the time, determined graphically or using a non-first-order method, for the analyte level to reach half of the original level, and does not imply any reaction order.

Laboratory studies

Harvey and Han (1978) treated a loamy sand (pH 6.8) and fine sand (pH 6.4) with 6 mg/kg [^{14}C]oxamyl and aerobically incubated samples for 28 days at 25°C and 30% of field capacity moisture. Volatile traps were not used. At intervals soil samples were extracted with water and analysed by LSC and radio-TLC. The half-life of oxamyl was 11 days in the loamy sand and 15 days in the fine sand.

In another study silt loam soil (pH 6.4, 2.75% organic matter) was treated at a concentration of 4 mg/kg and 20 mg/kg with [^{14}C]oxamyl, which was rapidly degraded under aerobic conditions at

25°C and 70% of normal water holding capacity (Dulka and Julis, 1978). The data were recalculated recently by DuPont from the original report using linear regression analysis (natural log of the % of applied radioactivity remaining as oxamyl *v.* time). The oxamyl half-lives were 11 days ($r^2 = 0.973$) at 4 mg/kg and 18 days ($r^2 = 0.979$) at 20 mg/kg.

In the aerobic section of the degradation study by Hawkins *et al.* (1989) [^{14}C]oxamyl had a half-life of 27 days in an acidic (pH 4.6) silt loam soil incubated for 12 days at 25°C and a water content of 75% at 33 kPa.

In the aerobic part of the degradation study by Spare (1991) [^{14}C]oxamyl had a half-life of 11 days in sandy clay loam (pH 7.7) incubated aerobically at 25°C and 70-75% of 33 kPa soil moisture. Using the data from the original study, half-lives of oxamyl, oxamyl oxime, and IN-D2708 were recalculated recently by DuPont using non-linear first-order regression, and were 11.5 days for oxamyl, 6.4 days for oxamyl oxime, and 5.0 days for IN-D2708 with an $r^2 = 0.987$ for the simultaneous fit of the regression model to the data.

[^{14}C]oxamyl was degraded with a half-life of 8 days in a loam soil (pH 7.0, 2.4% organic matter) incubated at 20°C and 40% of maximum water holding capacity (0 bar, ~1 kPa) (Smyser and Mattson, 2000). The half-lives of oxamyl oxime and IN-D2708 were 2 days and 8 days respectively.

The aerobic degradation rate was studied by Mattson and Smyser (2000) in three soils incubated at 20°C and 40-50% of 0-bar (~1 kPa) moisture (one soil was also incubated at 10°C). In a neutral (pH 7.0) silt loam incubated at 20°C the half-life of oxamyl was 3.0 days, and of oxamyl oxime and IN-D2708 5.9 and 3.6 days respectively, and at 10°C half-lives were 16.4 days for oxamyl, 21.5 days for oxamyl oxime, and 65.9 days for IN-D2708. In a slightly basic (pH 7.8) silt loam soil at 20°C, the oxamyl half-life was 4.1 days and the half-lives of oxamyl oxime and IN-D2708 were 1.7 and 3.4 days respectively. In a strongly acid (pH 4.8) silty clay loam at 20°C, the oxamyl half-life was 112 days, anomalous compared to all other results including those from other strongly acidic soils (Hawkins *et al.*, 1989). The oxamyl oxime half-life in this soil was 17.5 days; IN-D2708 was not observed. The microbial carbon content was <0.5% of the total soil organic carbon indicating poor microbial viability, possibly owing to stress during the period between collection in the field and the start of the laboratory study. Although this acidic soil is not typical of production agriculture soils it further demonstrates the importance of both pH and microbial activity on oxamyl degradation noted by Dulka and Julis (1978).

Smelt *et al.* (1979) evaluated the effect of soil moisture on the degradation rate of oxamyl in a humic loamy sand (pH 5.4, 4.5% organic matter), a clay loam (pH 7.1, 4.5% organic matter), a loamy sand (pH 7.4, 1.1% organic matter), and a peaty sand (pH 5.2, 9.0% organic matter). Samples were moistened to water contents of 0.03 kg/kg to 0.22 kg/kg in the humic loamy sand and 0.1 kg/kg to 0.36 kg/kg in the clay loam, and 0.12 kg/kg and 0.30 kg/kg in the loamy sand and peaty sand respectively, and incubated in the dark at 15°C for up to 140 days in loosely covered jars. At intervals samples were moistened with distilled water and extracted with acetone. The extracts were treated with chloroform and ethyl acetate/water, then analysed by GLC after conversion of oxamyl to oxamyl oxime. In soils with moisture above the wilt point half-lives were generally correlated with pH, with longer half-lives in the two acidic soils (34 days to 68 days) than the two basic soils (12 days to 20 days). There was a clear increase in conversion rate with increasing moisture in the two soils in which the water content was varied. Conversion rates in soil near field capacity were 1.6 (clay loam) to 1.8 (humic loamy sand) times those in soil near the wilting point. Changes were more pronounced at lower moisture levels. In the samples incubated well below the wilt point, the oxamyl conversion rate continued to decrease in the clay loam, but markedly increased in the humic sandy loam. It was suggested that this unexpected behaviour might be the result of a surface-catalysed reaction.

Dean (2000) incubated [^{14}C]oxamyl (0.5 mg/kg) in aerobic (oxidizing) saturated zone subsoils; the degradation rate was dependent on pH. In a mildly acidic (pH 6.1) sand subsoil with

0.2% organic matter, oxamyl was degraded by hydrolysis to oxamyl oxime with a half-life of 37 days, and in a strongly acidic (pH 4.6) sand with <0.1% organic matter it was essentially stable during the 120-day incubation.

Harvey and Han (1978) measured anaerobic degradation. They treated a silt loam soil (pH 4.9) with [^{14}C]oxamyl at 6 mg/kg and o 87% of field capacity. The soil containers were emptied five times and filled with nitrogen, sealed and incubated in the dark at room temperature. Samples were extracted with water after 0, 7, 14 and 28 days and analysed by LSC and radio-TLC. The half-life was 6 days.

In the anaerobic degradation study by Hawkins *et al.* (1989) [^{14}C]oxamyl applied at 20 mg/kg was degraded rapidly in a silt loam soil (pH 4.6, 3.7% organic mater) at 25°C. At the beginning of the trial 61% of the applied radioactivity was present as oxamyl but it was not detectable 30 days later. Assuming first-order degradation, the half-life would be <5 days.

In the anaerobic study by Spare (1991) the half-life of [^{14}C]oxamyl applied at 9.5 mg/kg was 6 days in a sandy clay loam soil (pH 7.7, organic matter 1.5%). Half-lives of oxamyl oxime and the acid IN-D2708 were recalculated recently by DuPont from the original results using linear least squares regression. The maximum percentage of the applied radioactivity was used as the starting point for each regression. The half-life of oxamyl oxime was 24 days ($r^2 = 0.968$, four data points used) and of IN-D2708 20 days ($r^2 = 0.741$, three data points).

In the study by Dean (2000) on anaerobic (reducing) saturated zone subsoils [^{14}C]oxamyl was degraded very rapidly as the result of an Fe^{II}-catalysed reduction which is well-documented in the literature and shown to be generally independent of pH (Bromilow, 1986; Strathmann and Stone, 2001). In Dean's study a strongly acidic (average incubation pH 4.5) loamy sand subsoil (8.4% organic matter) and a strongly acidic (average incubation pH 4.8) sand subsoil (<0.2% organic matter) were incubated with 0.5 mg/kg [^{14}C]oxamyl at 10°C. Oxamyl levels decreased to <1% within 1 day in both saturated subsoils, and the first order DT-90 values were estimated to be <6 hours. The half-life of the nitrile IN-N0079, the major degradation product, was 32 days in the loamy sand and 28 days in the sand.

Field studies

In a study by Harvey and Han (1978) a water-soluble liquid formulation of oxamyl (240 g/l) was applied to the surface of a prepared bed of a bare silt loam soil (pH 6.0, 1.5% organic matter) on 6 June 1973 in northern Delaware, USA at a rate equivalent to 6.33 kg/ha. Irrigation and rainfall at the site totalled 2.5 cm within 24 hours, 21.6 cm by 30 and 35.8 cm by 60 days. At intervals up to 60 days soil cores (1.9 cm i.d.) were taken to a depth of 30.5 or 76.2 cm, and divided into 10.2 cm or 15.2 cm depth segments which were homogenised. Samples were analysed using the method of Holt and Pease (1976) which converts oxamyl to oxamyl oxime before quantification, so that the results represented the combined residues of oxamyl and any oxamyl oxime. The half-life of the combined residue was 8 days. Residues were not observed (LOQ 0.04 mg/kg) in the deepest core segment.

A water-soluble formulation of oxamyl (240 g/l), was applied at field sites in Florida, California and Washington, USA, at a rate of 20.2 kg ai/ha (Lin and Eble, 1990; Lin, 1991). In Florida cucumbers (*Cucumis sativus* L.) were planted three days before pre-emergence treatment, in California cotton (*Gossypium hirsutum* L.) was planted one day after treatment, and in Washington oxamyl was applied to the leaves of established cherry trees (*Prunus cerasus* L.). The 0-30 cm depth soils were a sand (pH 5.8, 1.9% organic matter) in Florida, sandy loam (pH 7.9, 1.0% organic matter) in California, and clay loam (pH 7.1, 0.4% organic matter) in Washington. At the Florida site there was approximately 17 cm of rainfall during the first two months with a total of 142 cm rain and irrigation during the 14-month study period, at the California site there was approximately 20 cm of rain and irrigation during the first two months (total 74 cm during 18-month study), and at the Washington site there was only 2 cm of rain and irrigation during the first two months (total 21 cm

during 13-month study). Average air temperatures based on monthly figures were 23.3°C in Florida, 17.8°C in California, and 11.0°C in Washington. Only at the Washington site did average monthly temperatures reach or go below 0°C. At intervals soil cores to a maximum depth of 90 cm were analysed in segments using GLC (combined oxamyl and oxamyl oxime residue) or HPLC (separate oxamyl and oxamyl oxime residues). At the Florida site some oxamyl and oxamyl oxime residues were found in the 60-90 cm core segment and oxamyl degradation exhibited biphasic kinetics: the first phase accounted for the degradation of approximately 90% of the oxamyl with a half-life of 14 days, and the second slower phase had a half-life of 74 days. At the California site neither oxamyl nor oxamyl oxime was found in the 60-90 cm core segments and the oxamyl half-life was 9 days. In Washington trace levels of oxamyl and oxamyl oxime were found in the 60-90 cm core segments, with an oxamyl half-life of 62 days. This longer half-life may be explained by the dry, cool conditions as well as the different application method used in this orchard setting. In the 0-15 cm topsoil segment the average soil concentration of oxamyl oxime reached peaks of 1.2% on day 0 in Florida, 6.6% on day 13 in Washington, and 39.7% on day 59 in California (percentages calculated by dividing the peak average soil concentration of oxamyl oxime expressed as oxamyl equivalents by the maximum average soil concentration of oxamyl at each site).

McLory and Orescan (1996) applied a water-soluble liquid formulation of oxamyl (240 g/l) at a rate of 20.2 kg ai/ha to bare soil in Mississippi, USA. The 0-15 cm surface soil was silt loam (pH 5.8, 1.2% organic matter). Approximately 24 cm of rain fell at the site within two months and a total of 157 cm during the 13-month study period. The average temperature was 18.3°C. Soil cores were collected at intervals from triplicate plots, sectioned into 15-cm depth segments and analysed by HPLC to determine separately the soil concentrations of oxamyl and oxamyl oxime. Owing to the limited mobility of oxamyl, core segments below 45 cm were not analysed. Approximately 98% of the residues were recovered in the 0-15 cm, 1.7% in the 15-30 cm, and 0.5% in the 30-45 cm segment. The calculated half-life of oxamyl in the top 0-15 cm in the triplicate plots (A, B, C) was 7.7 days, 8.5 days, and 10 days. In the 0-15 cm topsoil section the average concentration of oxamyl oxime, expressed as oxamyl equivalents, peaked at 3.3% on day 30 in plot A, 1.7% on day 30 in plot B, and 1.7% on day 15 in plot C. Percentages were based on the maximum average oxamyl soil concentration in each plot.

Mol (2001) applied a granular formulation of oxamyl (10% oxamyl wt/wt) at a rate of 4 kg ai/ha to bare sandy loam soil (pH 6.6, 2.0% organic carbon) in The Netherlands. The granules were broadcast directly onto the soil surface and mechanically incorporated to a depth of approximately 20 cm. At 16 intervals up to 1.4 years after application, soil cores to a depth of 90 cm (30 cm on day 0) were collected, divided into 15-cm segments and homogenised to make composite samples for each depth at each interval. After extraction with acetonitrile/methanol acidified with formic acid, extracts were redissolved in methanol in formic acid, then analysed by HPLC with MS/MS detection. Oxamyl and oxamyl oxime were determined individually and the LOQs were 0.005 mg/kg for both analytes. Owing to rapid degradation only samples collected for the first 194 days were analysed, when the average air temperature was 14.8°C and rain and irrigation totalled 42.7 cm. Rainfall was 3.7 cm in the first week and 6.9 cm in the first month. Oxamyl and oxamyl oxime were not detected below a depth of 30-45 cm. Total residues in the soil profile were converted to mass per hectare, assuming a soil bulk density of 1.5 g/cm^3. From non-linear first order regression analysis of the total mass per hectare with time, the half-life of oxamyl was 9.2 days and of oxamyl oxime 3.6 days. On a molar equivalent basis, oxamyl oxime reached a peak of 6.4% (day 5) of the initial soil mass of oxamyl.

In a similar study Zietz (2001) applied the granular formulation of oxamyl (10% oxamyl wt/wt) at a rate of 5.5 kg ai/ha to bare silt loam soil (pH 7.3, 1.4% organic carbon) in England. The granules were broadcast directly onto the soil surface and mechanically incorporated to a depth of approximately 10 cm. At 18 intervals in the first year soil cores were collected, processed and analysed as described above (Mol, 2001). Owing to rapid degradation, only samples collected in the first 123 days were analysed, when the average air temperature was 15.2°C. It rained every day for the first 17 days (total of 7.3 cm), in the first month the total was 7.8 cm and in the first 123 days 188 cm.

Despite the heavy initial rain only trace levels of oxamyl and oxamyl oxime were found in the 75-90 cm layer on days 21 and 28. Total residues in the soil were converted to mass per hectare as above, and from non-linear first order regression of the total mass per hectare, the half-life of oxamyl was 11.5 days, and of oxamyl oxime 4.3 days. On a molar equivalent basis, oxamyl oxime reached a peak of 24% (day 13) of the initial soil mass of oxamyl.

Under cropped conditions, the half-life of oxamyl ranged from 7 days to 28 days after broadcast incorporation using a variety of commercial equipment to depths from 8 cm to 20 cm (average 14 cm) of the granule (10% oxamyl wt/wt) in ten commercial potato fields (loamy sand or sand, varying in pH from 5.9 to 7.0, average 6.5, organic matter content 2.3% to 5.4%, average 3.2%, in England (Ambrose *et al.*, 2000). Soil samples collected weekly for up to 91 days with an auger (2.5 cm x 30 cm) were homogenised, extracted with methanol and analysed for oxamyl by HPLC. Reported DT-50 values ranged from 7 to 28 days. Recent recalculation of the reported data using non-linear first-order regression by DuPont gave half-lives ranging from 8 to 24 days, with an average of 16 days. It is suggested by the authors that the average or cumulative soil temperature at the sites had a greater influence on degradation than rainfall.

Photolysis

In the study by Barefoot (1985) a silt loam (pH 4.5, 5.6% organic matter) and a sandy loam (pH 6.5, 2.1% organic matter) were air-dried, ground and sieved through a 2 mm screen before [^{14}C]oxamyl (78% initial radiochemical purity) was applied to the surface at a rate equivalent to 6.72 kg oxamyl/ha. Moisture content was adjusted to 75% of 33 kPa and the temperature maintained at 25°C with a constant air flow. Fluorescent sunlamps provided light in the 300 nm to 400 nm range with an intensity equivalent to half that of normal sunlight in Delaware, USA. Irradiated samples were collected on days 0, 2, 7, 12 and 20 and control samples stored in the dark on days 7 and 20. Traps collected volatile degradation products and $^{14}CO_2$. Soils were extracted with methanol, analysed by radio-TLC and identities confirmed by MS. The main irradiated degradation products were a polar fraction and unextractable residues but in the control samples $^{14}CO_2$ was the main product. The intermediate formation of oxamyl oxime and the nitrile IN-N0079 occurred in both irradiated and control samples. The half-lives of oxamyl originally calculated were 3 days in the sandy loam and 5 days in the silt loam, but these values were not corrected for degradation in the controls. The kinetic values recalculated recently by DuPont using linear least-squares regression assuming first order kinetics were 1.7 days ($r^2 = 0.879$) and 17.3 days ($r^2 = 0.431$) in the sandy loam irradiated and control samples respectively, and 3.1 days ($r^2 = 0.774$) and 17.9 days ($r^2 = 0.947$) in the corresponding silt loam samples. For the controls, a day 0 value equivalent to that in the irradiated samples was assumed for the calculation. Correcting for the rate of oxamyl loss in the controls gave a photolysis half-life of oxamyl of 1.9 days in the sandy loam and 3.8 days in the silt loam soil indicating that oxamyl is susceptible to photolytic losses on mineral surfaces.

Mobility

The mobility of oxamyl on soil TLC plates was assessed using four soils with wide ranging organic matter content by Harvey and Han (1978). The soils were an organic muck (pH 6.7, 83.5% organic matter), a silt loam (pH 6.4, 6.0% organic matter), another silt loam (pH 5.4, 2.1% organic matter) and a loamy sand (pH 5.8, 0.7% organic matter). On development with water the R_f value was inversely related to the percentage of organic matter. The R_f values were 0.53 for the organic muck, 0.69 for the silt loam, 0.79 for the second silt loam, and 1.0 for the loamy sand. Oxamyl was mobile or very mobile in all the soils except the organic muck in which mobility was intermediate.

In a column leaching study by Chrzanowski (1980) [^{14}C]oxamyl was applied in two ways to columns 5.1 cm in diameter and 45.7 cm long packed with either a sandy loam (pH 6.6, 0.79% organic matter) or a silt loam (pH 5.0, 4.0% organic matter). It was applied to the surface of one set of wetted columns at a rate equivalent to 9 kg ai/ha and a further 50.8 cm of water was added immediately which took one to two days to leach through the column. Another set of columns was

topped with 100 g of soil (which had been treated with 1.8 mg [^{14}C]oxamyl and aged under greenhouse conditions for 30 days at 65% to 75% of normal moisture capacities. This treated, aged soil was added to make up the top 5.1 cm of the 45.7 cm columns. Leaching was as described above. The column break-through curves and void volumes for the two soils were determined with a ^{36}Cl tracer from Na^{36}Cl. Total ^{14}C was determined in the leachate over time, as was the residual ^{14}C in the columns after leaching. In the sandy loam columns 100% of the applied radioactivity percolated through the freshly-treated and 61% through the aged soil, and in the silt loam the figures were 83% and 63% respectively. Maximum levels of ^{14}C occurred in the leachate from the fresh and aged sandy loam columns at a similar time to the ^{36}Cl tracer, but were later than the traces in the leachate from the fresh and aged silt loam columns. Residual soil radioactivity was not determined in the columns leached immediately after treatment since most of it had eluted. The radioactivity left in the aged soil columns (6.7% in sandy loam and 11% in silt loam) was mainly in the top 5-cm layer, and was presumed to represent fragments incorporated into the soil organic matter during the 30-day greenhouse incubation.

In a more detailed study [^{14}C]oxamyl was applied at a rate equivalent to 4 kg ai/ha with Na^{36}Cl to fresh silt loam (pH 4.3, 1.9% organic matter), loamy sand (pH 5.4, 2.0% organic matter), sandy loam (pH 6.3, 1.0% organic matter) and loam (pH 7.3, 5.1% organic matter) soils in 5.1-cm x 30.5-cm columns (duplicate columns of the silt loam and single columns of the others). A further two columns of the silt loam were used for aged leaching (Rhodes *et al.*, 1987). Treatment with oxamyl was followed immediately by the application of 1000 ml of deionised water at a rate of approximately 50 ml per hour. For the additional aged leaching silt loam was treated with [^{14}C]oxamyl (4 kg ai/ha) and aged for 7 or 18 days at 25°C and a moisture content of 21.7% by weight, then Na^{36}Cl was mixed into the soil which was added as the top 5-cm segment to the 30.5-cm columns. Water was then added as described above. Leachate from the fresh and aged columns was collected and analysed for total ^{14}C and ^{36}Cl radioactivity. Pooled fractions of the column leachates were concentrated and further analysed by HPLC, and residues in the soil were determined and characterized by HPLC and radio-TLC following extraction with methanol and water. More than 90% of the applied radioactivity was recovered in the leachate from all of the freshly treated soil columns. The nature of the ^{14}C compounds in the leachate was clearly related to the pH of the soils. In that from the pH 7.3 loam oxamyl was not detected and the hydrolysis product, oxamyl oxime, was the major component (79.7%). In the pH 6.3 sandy loam leachate, oxamyl 18.8% and oxamyl oxime represented 54.1% of the ^{14}C, and in the pH 5.4 loamy sand leachate oxamyl and oxamyl oxime accounted for 50.3% and 27.4%. Oxamyl in the leachate from the duplicate silt loam columns represented 81.1% and 82.8%, and oxamyl oxime was not observed. The acid IN-D2708 was observed at levels from 12.1% to 17.2% in leachates from all the columns. The ^{14}C in the columns after leaching (5% to 11% of that applied) was distributed throughout the columns with slightly more in the top 5-cm layer. These residues were found to be mainly IN-D2708, oxamyl, and oxamyl oxime. In the silt loam soil column aged 7 days, 66.8% of the applied ^{14}C was recovered from the leachate and 21.8% from the soil. In the silt loam column aged 18 days, 36.7% was in the leachate and 27.9% was in the soil. The rest of the ^{14}C was presumed lost as ^{14}CO$_2$ during the ageing process. The leachate from the aged silt loam columns contained mainly oxamyl and IN-D2708. Oxamyl oxime was also found in the leachate from the 18-day aged column. The ^{14}C left in the aged columns after leaching was mainly associated with the top 5-cm layer. Following extraction with methanol and water, these residues were characterized as unextracted, IN-D2708, oxamyl, and oxamyl oxime. The ^{14}C and ^{36}Cl elution curves peaked in the leachate at similar times from all fresh and aged columns except the pH 7.3 loam where oxamyl oxime was the main component.

The adsorption of [^{14}C]oxamyl to a silty clay loam from Illinois, USA (pH 4.8, 4.4% organic matter), a loam from The Netherlands (pH 7.0, 2.4% organic matter), a silt loam from Germany (pH 7.8, 2.1% organic matter), a silt loam from Maryland, USA (pH 6.9, 4.3% organic matter), and a silt loam from Mississippi, USA (pH 7.0, 0.4% organic matter) was studied by Ohm (2001). The adsorption coefficients K$_D$, K$_{OM}$ and K$_{OC}$, and the Freundlich adsorption isotherm parameters K$_F$, K$_{FOM}$, K$_{FOC}$ and 1/n were calculated. The soils were pre-equilibrated with 0.01 M CaCl$_2$, then equilibrated with solutions of [^{14}C]oxamyl at concentrations of 0.05, 0.1, 0.5, 1 and 5 μg/ml in 0.01 M

CaCl$_2$ (1:1 soil:solution) for four hours at 20-25°C, then two four-hour desorption cycles were conducted for soils with an average adsorption of 10% or more at the highest solution concentration by decanting the supernatant, after centrifugation, and replacing it with an equivalent volume of fresh 0.01M CaCl$_2$. Linear adsorption coefficients were calculated from the mean ratios of the sorbed and water concentrations at equilibrium, using the Freundlich equation. Selected samples were analysed by HPLC and combustion/LSC to confirm stability of oxamyl and recovery of ^{14}C. Oxamyl was found to be weakly adsorbed, with linear K$_{OC}$ values, uncorrected for soil organic carbon, ranging from 7 ml/g to 39 ml/g (average 17 ml/g). **????** were marginally related to soil organic matter content being generally higher in the higher organic matter soils. The K$_{FOC}$ values were similar, ranging from 4 ml/g to 37 ml/g (average 16 ml/g) and the 1/n values from 0.946 to 1.27 (average 1.07). Freundlich parameters could not be derived for the loam soil owing to negligible sorption at some concentrations. Of the oxamyl adsorbed, 71% was desorbed from the silty clay loam, 64.1% from the Mississippi silt loam, and 36.5% from the Maryland silt loam in two desorption cycles. The other soils showed <10% adsorption at the highest solution concentration so were not used in the desorption experiment. The results from this study are similar to those reported by Bromilow *et al.* (1980) (average oxamyl K$_{OC}$ in six soils 5 ml/g), and Gerstl (1984) (average K$_{OC}$ in five soils 37 ml/g).

The batch equilibrium sorption of [^{14}C]oxamyl oxime and ^{14}C-IN-D2708, the main soil degradation products of oxamyl, was measured in similar studies to Ohm's (Berg, 2000a,b). Initial concentrations ranged from 0.05 µg/ml to 5.0 µg/ml, 1:1 soil/solution ratios were used, and samples were equilibrated at 20°C for 24 hours. The same five soils were used as above, although the pH and organic contents varied because different batches of the soils were used.

For oxamyl oxime linear K$_{OC}$ values ranged from 4 ml/g to 11 ml/g (average 7 ml/g), Freundlich K$_{FOC}$ from 4 ml/g to 10 ml/g (average 7 ml/g) and 1/n values from 0.87 to 1.24 (average 1.03). In the only soil in which more than 10% of the oxamyl oxime was adsorbed at the highest concentration (silt loam, 6.3% organic matter), 70.3% was desorbed in two desorption cycles.

For IN-D2708, linear K$_{OC}$ values ranged from 2 ml/g to 10 ml/g (average 6 ml/g), Freundlich K$_{FOC}$ from 6 ml/g to 14 ml/g (average 10 ml/g) and 1/n values from 0.532 to 0.762 (average 0.668). The three soils from which more than 5% of the IN-D2708 had been sorbed were subjected to two desorption cycles, when 70.9% to 82.1% was desorbed with 0.01 M CaCl$_2$.

Loss from plant leaves

Ware *et al.* (1978) sprayed a liquid oxamyl formulation (240 g/l) twice onto cotton plants planted with a row spacing of 102 cm (average plant height at application 79 cm) in a field in Arizona, USA, at 276 kPa pressure and a ground speed of 9 km/h for a total application of 94 l spray/ha, 1.12 kg ai/ha, each application at one-half the dose to ensure uniform coverage. Triplicate sets of 100 leaf disks (2.54 cm diameter) were collected 0, 24, 48, 72 and 96 hours after treatment from the top, middle, and bottom of the canopy. During this period daytime high temperatures ranged from 38°C to 41°C and nighttime minimum air temperatures from 15°C to 23°C. No rain was reported. Leaf disks were extracted in the field with water, and the extracts chilled or frozen until analysis in a commercial laboratory (method not given). Dislodgeable leaf surface residues of oxamyl rapidly decreased with 49.4 mg/m^2 on day 0, 49.4 mg/m^2 on day 1, 5.14 mg/m^2 on day 2, 1.68 mg/m^2 on day 3, and 1.48 mg/m^2 on day 4. A half-life of 0.7 days (r^2 = 0.893) was calculated recently by DuPont from the natural log of the water-extractable surface residue with time.

In a similar study by Buck *et al.* (1980) the same formulation of oxamyl (240 g ai/l) was sprayed onto cotton plants with a row spacing of 102 cm (average height of 76 cm) in Arizona, USA, at 276 kPa pressure and a ground speed of 4 km/hr for a total of 122 l spray/ha, 0.41 kg ai/ha. Triplicate sets of 100 leaf disks each were collected as described above and daytime high temperatures ranged from 37°C to 40°C and nighttime from 17°C to 26°C. No rain was reported. Leaf disks were extracted in the field with water and chilled until analysis. Portions of the aqueous extracts

were extracted with hexane, base was then added (converting oxamyl to oxamyl oxime) and samples re-extracted with chloroform and ethyl acetate. The organic extracts were discarded and triethylamine was added before the processed aqueous fractions were concentrated for analysis by GLC. Dislodgable leaf surface residues decreased with 1.5 $\mu g/cm^2$ on day 0, 1.1 $\mu g/cm^2$ on day 1, 1.2 $\mu g/cm^2$ on day 2, 0.85 $\mu g/cm^2$ on day 3, and 0.76 $\mu g/cm^2$ on day 4. From these data, a half-life of 4.3 days ($r^2 = 0.883$) was calculated recently by DuPont.

Residues in rotational crops

Rotational crop studies (three confined and one field) with either labelled or unlabelled oxamyl applied to soil aged for 30, 120 and/or 363 days before planting. Significant oxamyl residues remained in the soil at planting, allowing the assessment of the potential for accumulation of residues in the rotational crops.

In a greenhouse trial Harvey (1978) applied [^{14}C]oxamyl directly to sandy loam soil (0.73% organic matter, pH 5.9) in New Castle, DE, USA, at 9 kg ai/ha. The soil was aged for 30 and 120 days and beets, sorghum and cabbage planted at the end of each period and grown to maturity. Soil samples were taken at planting and segmented into 0-10.2 cm, 10.2-20.3 cm and 20.3-30.5 cm sections. The 0-10.2 cm layers were extracted with methanol and water for analysis by silica gel TLC (ethyl acetate). Oxamyl was degraded rapidly in the soil and the plants. At the end of each ageing period the 0-10.2 cm layer contained 88-96% of the recovered radioactivity. After thirty days the soil contained approximately 48% of the applied radioactivity (19% oxamyl and 1.1% oxamyl oxime) and after 120 days approximately 12% (0.3% oxamyl and 0.1% oxamyl oxime). At maturity beet foliage and roots, sorghum fodder and grain, and cabbage heads were analysed for total and extractable (methanol, hexane, ethyl acetate) radioactivity. The concentrated extracts were partitioned with hexane and ethyl acetate, and the fractions analysed by TLC. The combined concentration of oxamyl plus oxamyl oxime was low (≤0.12 mg oxamyl equivalents/kg) in crops planted 30 days after treatment, and insignificant in crops planted after 120 days (<0.02 mg oxamyl equivalents/kg).

Hawkins *et al.* (1990) applied [^{14}C]oxamyl directly to sandy loam soil (Somersham, Cambridgeshire, UK, 2.7% organic matter, pH 6.3) in a growth room at 20 kg ai/ha. The soil was aged for 30, 120 and 363 days before barley, lettuce and beets were planted. Soil samples were collected at the time of application, the time of sowing (30, 120, or 363 days after application), at immature harvest (barley forage) and at mature crop harvest. At mature harvest barley grain, chaff and straw, whole lettuce, and beet foliage, root and root peel were separated. The total ^{14}C and the residues extractable with methanol, methanol/water, and water in the crop and air-dried soil samples were determined by normal and reverse-phase TLC. Plant extracts were also incubated with β-glucosidase and the barley straw was hydrolysed with acid. Unextractable plant residues were further processed by enzyme treatments.

The total ^{14}C in the treated soil decreased from about 18 mg oxamyl equivalents /kg immediately after treatment to about 13, 7.2 and 0.64 mg oxamyl equivalents/kg at the 30-, 120- and 363-day planting intervals respectively. 90% of the residue in the soil was available for crop uptake (64% as oxamyl) after thirty days, approximately 57% was solvent-extractable (24% as oxamyl) after 120 days, and less than 4% was extractable after 363 days. Other residues included oxamyl oxime, IN-D2708 and several polar components.

The TRR in crops sown 30 days after treatment ranged from 3.1 mg/kg oxamyl equivalents in lettuce to 38 mg/kg in barley straw, for crops sown after 120 days from 0.27 mg/kg in lettuce to 6.8 mg/kg in mature beet foliage, and for crops sown after 363 days from 0.03 mg/kg in lettuce to 0.29 mg/kg in barley straw. Oxamyl, oxamyl oxime and IN-D2708 were identified at concentrations above 0.01 mg/kg (oxamyl equivalents) in barley, beet, cabbage and lettuce planted 30 and 120 days after treatments. These components were previously identified in plant metabolism studies and were all

present in the soil at planting. Polar unknowns 1, 3, and 7 were at significant levels in plant tissues from the 30- and 120-day plantings, but not in the soil.

In an attempt to characterize the unknown polar residues 1, 3 and 7 a separate greenhouse study (Brown *et al.,* 2001b) was conducted with barley planted in soil treated with [^{14}C]oxamyl 30 days earlier. Radiolabelled residues in the soil and barley were analysed by TLC and by more sophisticated HPLC analyses than were possible for the previous study. A solution of [^{14}C]oxamyl containing inert formulation ingredients to simulate a Vydate® 10L formulation was applied once to Sassafras sandy loam soil (1% organic matter, pH 6.3) at 8 kg ai/ha in New Castle, Delaware, USA, and the soil aged for 30 days in the field before barley (the rotational crop) was planted. Soil samples were taken on the day of treatment, the day of planting (day 30), hay sampling (93 days after treatment) and final harvest (166 days after treatment) and crop samples included barley forage (taken 20 days after planting), hay (sampled after 63 days) and straw and grain (sampled after 136 days). The TRR and extractable residues (methanol and aqueous methanol for plants; acetonitrile and aqueous acetonitrile for soils) were determined. Barley hay, straw, and forage extracts were also subjected to β-glucosidase and acid hydrolyses. All extracts were analysed by LSC, HPLC and/or TLC.

Solvent-extractable residues in the soil decreased steadily. After thirty days only 32.9% of the radiolabelled residue was solvent-extractable, with 14.8% of the TRR (0.1 mg/kg) present as oxamyl, and at harvest only 7% was extractable (1% of the TRR, 0.01 mg/kg oxamyl). Other extractable compounds included IN-D2708 and oxamyl oxime. The TRR (expressed as oxamyl) exceeded 0.01 mg/kg in all the barley fractions. Most of the radioactivity was extracted from the forage (88.8 % of the TRR, 5.96 mg oxamyl equivalents/kg), hay (84.3% of the TRR, 1 mg oxamyl equivalents/kg), straw (71.7% of the TRR, 1.13 mg oxamyl equivalents/kg) and grain (60.3% of the TRR, 0.19 mg oxamyl equivalents/kg). The main extractable residue in the grain (51.3% of the TRR, 0.16 mg oxamyl equivalents /kg) was IN-D2708. No oxamyl or oxamyl oxime was detected (<0.01 mg oxamyl equivalents/kg); other polar components were detected at 4.0 % of the TRR (0.01 mg oxamyl equivalents/kg). The chromatographic profiles of the barley forage, hay and straw were similar. Residues included IN-D2708 (2.9-8.2% of the TRR; 0.05-0.23 mg oxamyl equivalents/kg), oxamyl oxime (4.6-13.4% of the TRR; 0.06-0.90 mg oxamyl equivalents/kg) and oxamyl (5.9-24.0% of the TRR; 0.07-1.61 mg/kg). Other foliar components were tentatively identified as IN-KP532, IN-L2953, IN-T2921 and IN-N0079. Unknown foliar components were typically present at ≤2 % of the TRR (≤0.14 mg oxamyl equivalents/kg), but a major unknown compound in the forage, hay and straw constituting 24.4-40.4% of the TRR (0.45-1.64 mg oxamyl equivalents/kg) was resistant to enzyme and acid hydrolysis, and was more polar than oxamyl oxime on the evidence of HPLC chromatography. It was similar in behaviour to the metabolite oxamyl oxime glucoside.

In a field study by Lin and Tomic (1992) at three US sites (Florida, sand, 1.6% organic matter, pH 5.5; Indiana, clay loam, 2.7% organic matter, pH 5.8; California, loam, 0.9% organic matter, pH 8.3) oxamyl was formulated as Vydate L [24% w:v]. At each site three broadcast applications at 4.5 kg ai/ha were applied weekly directly to the soil. Representative small grain, leafy vegetable, and root crops were planted 30 and 120 days after the last application and grown to maturity.

Soil samples (0-30.5 cm depth) were taken before and immediately after the final application, at planting and at harvest, and analysed for oxamyl and oxamyl oxime by a GC-MS method (Holt and Pease, 1976) in which oxamyl is hydrolysed to oxamyl oxime. Total residues, reported as oxamyl equivalents, decreased steadily in the soil from 1-3.5 mg/kg at all sites immediately after the last application to 0.042-1.25 mg/kg after 30 and 120 days and <0.02-0.12 mg/kg after 120 days.

At maturity crops were harvested and analysed for oxamyl and the oxime by the GC-MS method of Holt and Pease (1976). Lettuce and mustard greens planted approximately 30 and 120 days after the last application contained a maximum of 0.27 and 0.026 mg oxamyl equivalents/kg respectively. Radish roots (representative of the edible portion of root crops) planted at these intervals

contained a maximum of 0.34 and <0.02 mg/kg, and wheat and oat grain 0.068 and <0.02 mg/kg crop respectively.

All three trials demonstrated that oxamyl as well as the degradation products oxamyl oxime and IN-D2708 were taken up by the succeeding crop. The tentative identification of IN-KP532, IN-T2921, IN-L2953 and IN-N0079 further indicates that metabolism in rotated crops is consistent with that found in plant studies. Oxamyl was hydrolysed to oxamyl oxime, which was ultimately metabolized to IN-D2708 (presumably via IN-N0079 and IN-T2921) and other polar metabolites. Oxamyl oxime can also be demethylated to give IN-L2953, which can be metabolized to IN-KP532. It is proposed that a major component in rotated crops (specifically barley) is the glucose conjugate of oxamyl oxime, a major plant metabolite. The field rotational crop study confirms that succeeding crops take up oxamyl and/or oxamyl oxime when planted 30 days after oxamyl application, but no significant oxamyl residues were found in the edible parts of crops planted 120 days after oxamyl application.

Environmental fate in water and water-sediment systems

Hydrolysis in water

The stability of [14C]oxamyl in buffered aqueous solutions at pH 4.7, pH 6.9, and pH 9.1 (0.01M mixtures of sodium acetate, sodium chloride, or sodium bicarbonate) containing approximately 1200 mg oxamyl/l was evaluated by Harvey and Han (1978). Solutions were incubated in plugged glass flasks at room temperature in the laboratory and at intervals samples were analysed by radio-TLC. The stability of oxamyl was markedly related to pH: at pH 4.7 [14C]oxamyl was stable for at least 11 days, at pH 6.9 hydrolysis to oxamyl oxime was observed (3% after 24 hours and 9% after 48 hours) and at pH 9.1 30% was converted in the first six hours, but the hydrolysis reaction neutralised the sodium carbonate buffer and hydrolysis was comparable to that at pH 6.9 for the remainder of the experiment. The oxime was the only degradation product and conversion of oxamyl to oxamyl oxime was quantitative.

In a further study by McNally and Wheeler (1988b) buffered solutions containing 20 mg/l [14C]oxamyl were incubated for up to 31 days at 25°C in the dark. Solutions were adjusted to pH 5 (0.01 M acetate buffer), pH 7 (0.01 M phosphate buffer), and pH 9 (0.01 M borate buffer) before treatment and the pH did not change (<0.1 unit) during the course of the experiments. Samples were analysed by LSC and HPLC and the identity of the degradation product was confirmed by GC-MS. Quantitative conversion of [14C]oxamyl to oxamyl oxime was observed at pH 7 and pH 9. [14C]oxamyl oxime represented >93% of the applied radioactivity in the solutions by day 30, and at pH 5 [14C]oxamyl was stable and oxamyl oxime was not observed over a 31-day period. The half-lives of oxamyl were >30 days (stable) at pH 5, 7.9 days at pH 7 and 2.9 hours at pH 9.

Hydrolysis studies were also conducted with the two main soil degradation products of oxamyl, oxamyl oxime and the acid IN-D2708 by Lee and Berg (2001a,b). Sterile buffered solutions at pH 4, 7, and 9 containing either 1.7 mg/l of [14C]oxamyl oxime or 1.0 mg/l of [14C-]IN-D2708 were incubated in the dark for 30 to 34 days, and both compounds were stable at all pH levels throughout the tests.

Photolysis in water

Two experiments investigating the stability of [14C]oxamyl in aqueous solutions exposed to sunlight were reported by Harvey and Han (1978).

In the first, solutions of 1 mg oxamyl/l and 1000 mg oxamyl/l in distilled water (pH 6.2) and river water (Brandywine river, Delaware, USA, pH 6.5) were exposed to artificial light at wavelengths between 300 and 400 nm with an intensity of 1200 μW/cm^2 at a distance of 18 cm (stated to be equivalent to about half the intensity of summer sun at midday in Delaware, USA) for

seven days at 31°C. Control samples were incubated in the dark for 10 days at 24°C. At 0, 3 h, 19 h, and 2, 4 and 7 days samples were analysed by LSC and radio-TLC-MS. Control samples were analysed only after 10 days. Major compounds were identified by MS. The degradation of [^{14}C]oxamyl was substantially more rapid in irradiated samples than in the controls. The levels of [^{14}C]oxamyl remaining in the irradiated solutions after 7 days were 61% (distilled water 1 mg/kg), 2% (river water 1 mg/kg), 28% (distilled water 1000 mg/kg) and 22% (river water, 1000 mg/kg), whereas levels in the controls ranged from 84% to 98%. More conversion was noted in the river than in the distilled water, which suggests that inorganic and/or organic components in natural water play a role in the degradation, possibly *via* indirect photolysis. Oxamyl oxime and a polar fraction were the main degradation products, at levels of up to 67% and 23% respectively.

In the second study, a container was filled with 20 l of river water with [^{14}C]oxamyl at a concentration of 1 mg/l and the surface exposed outdoors to direct natural sunlight for six weeks. The water was moderately agitated and any lost by evaporation was replaced at frequent intervals with distilled water. At intervals small aliquots were removed for analysis by LCS and radio-TLC. At the end of the six weeks a 12-l portion was rotary-evaporated without ^{14}C loss to 500 ml, and the concentrate analysed by HPLC, radio-TLC and MS. Total radioactivity decreased by 17% and was presumed lost as ^{14}CO$_2$. While only 3% of the oxamyl was hydrolysed to oxamyl oxime during the first 16 hours (overnight), complete conversion occurred during sunlight hours the next day, and oxamyl oxime was gradually converted into a mixture of polar compounds. After six weeks the composition of the ^{14}C residue in the water was 80% oxamyl oxime and a geometrical oxime isomer, 14% IN-D2708, and 6% unidentified polar fractions.

The photolytic degradation of [^{14}C]oxamyl was also studied in sterile aqueous solutions exposed to artificial sunlight (<290 nm excluded) equivalent to summer sunlight in Delaware, USA (McNally and Wheeler, 1988a). The purified buffered water, pH 5 (0.01M acetate), containing 18.4 mg oxamyl/l was continually exposed for 16 days to xenon light and maintained at 25°C, with controls incubated in the dark. Samples were collected at 0, 1, 3, 7, 9, 14 and 16 days for analysis by LSC and HPLC, with identification by GC-MS. Oxamyl conversion in irradiated samples was rapid, and the half-life was 7.4 days. Oxamyl oxime was the only product in irradiated samples (maximum 75.3% by day 16) and no conversion occurred in the controls demonstrating that the formation of oxamyl oxime in irradiated samples was due to the presence of light, presumably because of a photo-hydrolytic reaction. Oxamyl oxime was resistant to further hydrolysis, as shown by the absence of any other degradation products.

In a supplement to this study Schmuckler (2001) calculated the quantum efficiency of oxamyl in aqueous systems to be 0.0187, with a predicted half-life (calculated at midday for maximum sun exposure and 30°, 40°, and 50° latitude) from 6.6 days in summer to 27.0 days in winter.

Fate in water/sediment systems

The degradation of [^{14}C]oxamyl was studied in stream and pond samples (Spare, 1995). The stream water had a pH of 7.3, hardness of 164 mg CaCO$_3$ equivalents/l, and an organic matter content of 726 mg/l, and the sediment was sandy loam (pH of 6.7, organic matter content 2.4%). The pond water had pH 7.5, hardness 89 mg CaCO$_3$ equivalents/l and organic matter content 402 mg/l; its sediment was also sandy loam (pH 6.1 and 5.1% organic matter). Containers filled with water and sediment (4:1 ratio) were equilibrated for 51 days in a flow-through apparatus. Aerobic water and anaerobic sediment phase conditions were established in each system. [^{14}C]oxamyl was applied at a concentration of 2 mg/l and the samples incubated in the dark at 20°C for 100 days were either vigorously hand-shaken to mix the water and sediment initially, or the water and sediment were not mixed. Samples were taken at 0 and 6 h from both systems and on days 1, 2, 7, 14, 30, 61 and 100 from the unmixed system. Polyurethane foam plugs, ethanolamine, and caustic traps which collected organic volatiles and ^{14}CO$_2$ were changed at each sampling or twice per month. Sediment samples were exhaustively extracted with methanol/water and methanol after separation, and the extracts and

water phase samples were analysed by LSC and HPLC. Foam plugs were analysed by solvent extraction and LSC, and ethanolamine and caustic traps by LSC before and after the addition of $BaCl_2$ to confirm $^{14}CO_2$. The identities of degradation products were confirmed by radio-TLC. In the mixed samples 15% to 20% of the dose was immediately associated with the sediment, decreasing to 11% in the stream and 18% in the pond system in 6 hours. The unmixed samples required 14 to 61 days' incubation to reach similar levels. Not more than 21% of the dose was associated with the sediment from either system over the 100-day incubations. The same pattern of degradation was found in the stream and pond systems, although the product ratios varied. The primary degradation products were oxamyl oxime, the acid IN-D2708, the nitrile IN-N0079 and $^{14}CO_2$. The substantial amounts of the nitrile are likely to have resulted from the Fe^{II}-oxamyl reduction (Dean, 2000; Strathmann and Stone, 2001). The anaerobic sediment phase would be a likely source of Fe^{II}. Only limited partitioning of the degradation products (about 10% or less for individual compounds) to the sediment was observed, with most of residues remaining in the water phases. Maximum levels of the major degradation products in the stream water were 48.8% oxamyl oxime (day 2), 66.8% IN-D2708 (day 30), and 11.3% IN-N0079 (day 7), and in the pond water 25.3% (day 2), 64.2% (day 30), and 45.1% (day 7) respectively. Through day 61 both systems produced similar $^{14}CO_2$ levels (20.9%-25.6%) but from day 61 to day 100 the stream system generated 43.3% of the dose and the pond only 8.7%, with totals of 68.9% for the stream and 29.6% for the pond system. Unextractable residues followed a similar trend and rose to 18% in the stream system and 9% in the pond after 100 days. Oxamyl degradation was rapid in both systems (non-first order DT-50 values 1 day in the stream and 0.4 days in the pond).

In a study by Barnes (2001) 26.1 mg/l of unlabelled oxamyl was dissolved in aqueous nutrient medium inoculated with activated sewage sludge bacteria (30 mg solids/l) with sodium benzoate as a positive control. The samples incubated for 29 days at 20°C to 24°C were aerated with CO_2-free air, which was then passed through a 0.025 N barium hydroxide solution, trapping any CO_2 produced. The production of CO_2 was determined by acid titration of the residual barium hydroxide. By day 6, 59% and by day 29, 99.0% of the theoretical maximum CO_2 had been produced from the sodium benzoate control. Similar results were observed for sodium benzoate in the presence of 26.1 mg/l of oxamyl, demonstrating that oxamyl had no effect on the sewage sludge organisms under these conditions. In the oxamyl samples without sodium benzoate 19% of the theoretical maximum CO_2 level was produced by day 29, showing that the sewage sludge microbes were able to utilise oxamyl appreciably, though not as readily as sodium benzoate.

RESIDUE ANALYSIS

Analytical methods

Oxamyl is considered the only relevant compound in the total toxic residue, but earlier GLC methods converted oxamyl to oxamyl oxime and determined total residues of the two analytes.

<u>Animal materials</u>

Methods for generation of residue data

Oxamyl and oxamyl oxime residues in urine, faeces, liver, kidney, lean meat, fat and milk are determined by extraction with ethyl acetate, followed by liquid-liquid partitioning clean-up, and alkaline hydrolysis of oxamyl to the more volatile oxamyl oxime. Final determination is by GLC with sulfur-sensitive flame photometric detection (Holt and Pease, 1976). The LOQ is 0.02 mg/kg for milk and 0.04 for fat, liver, kidney and meat (Table 11).

Bacher (2001) developed a method for milk, bovine muscle and eggs based on this method in which gel permeation chromatography (GPC) is used for clean-up. Samples are extracted with ethyl acetate, concentrated and brought up in a solution of 1:1 ethyl acetate:cyclohexane. Additional clean-

up by GPC is followed by conversion of oxamyl to oxamyl oxime in basic solution and analysis by GC-MS. The LOQ is 0.01 mg/kg (Table 11).

Table 11. Performance of analytical methods for the determination of oxamyl in animal materials.
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Reference	Fortification (mg/kg)	Mean recovery (%)	SD (%)	Range (%)		Sample	Control interference
				low	high		
Holt and Pease, 1976	0.4-4	100		75	130	Urine	Insignificant to none
	0.2-2	83		72	100	Faeces	
	0.04-0.4	90		75	102	Liver	
	0.04-0.4	89		82	100	Kidney	
	0.04-0.4	107		85	114	Lean meat	
	0.04-0.4	98		80	130	Fat	
	0.02-0.2	83		70	110	Milk	
Bacher, 2001	0.01-0.1	82	8	73	93	Whole milk	None
	0.01-0.1	91	9	80	101	Bovine muscle	
	0.01-0.1	101	6	90	106	Whole egg	

Methods for enforcement

Ali (1989) developed an HPLC method to determine the carbamates aldicarb, aldicarb sulfoxide, aldicarb sulfone, bufencarb, carbofuran, 3-hydroxycarbofuran, carbaryl, methiocarb, methiocarb sulfoxid, and methomyl in liver, and recommended it for the determination of other methylcarbamates such as oxamyl.

Anhydrous sodium sulfate is added to samples of bovine, pig, or duck liver and samples are then extracted twice with methylene chloride (CH_2Cl_2) using a homogeniser and the combined extracts passed twice through a filter paper with anhydrous sodium sulfate. The filtered extract is concentrated and dissolved in 1:1 methylene chloride/hexane solution, filtered again, and passed through a GPC SX-3 gel column for clean-up. The residue fraction is evaporated to dryness, reconstituted in CH_2Cl_2, passed through an aminopropyl solid-phase extraction cartridge, and eluted with 1.5% methanol in CH_2Cl_2. The eluate is evaporated to dryness, reconstituted in methanol and filtered. Determination is by reverse-phase HPLC with fluorescence detection (340 and 455 nm).

Liver samples were fortified with 0.005, 0.01 and 0.02 mg/kg of mixed carbamate standards. The average of 10 recoveries of 10 carbamates at all 3 levels of fortification was greater than 80% with coefficients of variation less than 17%.

Plants

Methods for generation of residue data

Oxamyl and oxamyl oxime residues in plants are determined by initial extraction with ethyl acetate, liquid-liquid partitioning clean-up, alkaline hydrolysis to the oxime, and final determination by GLC with sulfur-sensitive flame photometric detection (Holt and Pease, 1976; Rühl, 2000). The LOQ is 0.02 mg/kg for dry and watery crops (Table 12).

In the method of Thean *et al.* (1978) oxamyl is extracted with ethyl acetate in a homogeniser, water is added to the extract and the ethyl acetate evaporated under vacuum. Clean-up is by liquid-liquid extractions. Reverse-phase HPLC with UV detection under isocratic conditions is used to quantify oxamyl *per se*. Fortifications were at 1-2 mg/kg; the LOQ was not reported (Table 12).

Françon *et al.* (2000) extracted samples with acetone in an accelerated solvent extractor. The extract is passed through an ENVI-Carb® SPE cartridge to remove pigments and other interfering materials. The whole SPE eluate is then evaporated to about 0.5 ml, dissolved in a mixture of 10%

acetone in cyclohexane and applied to a Silica Mega Bond Elut® SPE cartridge to complete clean-up. The final extract is filtered and analysed by HPLC with column-switching and UV detection. Oxamyl and its oxime are determined separately. The LOQ of oxamyl was 0.02 mg/kg (Table 12).

Françon *et al.* (2001) validated The Netherlands' multi-residue HPLC method 2 for methylcarbamate pesticides for oxamyl only. Oxamyl is extracted with acetone and partitioned into dichloromethane/light petroleum. An aliquot of the extract is evaporated to dryness and if necessary purified by solid-phase extraction on aminopropyl-bonded silica cartridges. Oxamyl is determined by HPLC with post-column hydrolysis of oxamyl to form methylamine, reaction of this with *o*-phthalaldehyde reagent, and fluorescence detection of the derivative. The LOQ of oxamyl was 0.01 mg/kg (Table 12).

Table 12. Performance of analytical methods for the determination of oxamyl in crops.

Reference	Fortification (mg/kg)	Mean recovery (%)	SD (%)	Recoveries (%)		Sample	Control interference
				low	high		
Holt and Pease, 1976	0.02-0.8	103		78	120	Peanut, kernel	Insignificant to none
	0.10-5	99		82	120	Peanut, hull	
	0.05-10	93		70	113	Peanut, foliage	
	0.02-5	93		72	110	Tobacco	
	0.04-0.4	98		94	100	Apples	
	0.02-4	94		76	105	Turf grass	
	0.04-2	76		54	105	Peaches	
	0.04-1	92		81	99	Lettuce	
	0.04-2	83		70	90	Oranges	
	0.02-0.2	95		84	102	Cotton seed	
	0.08-0.4	79		72	85	Grapefruit	
	0.02-2	98		96	100	Coffee beans	
	0.02-2	85		70	94	Grapes	
	0.02-4	91		76	120	Potatoes	
	0.02-2	83		71	109	Tomatoes	
	0.04-2	84		70	109	Celery	
	0.02-1	91		84	100	Peppers	
	0.02 – 0.2	88		79	96	Carrots	
Thean *et al.*, 1978	1-2	65	21			Celery	None
		76	7			Cabbage	
		63				Collard	
		62	3			Turnip	
		61	11			Mustard	
		69	6			Grapefruit	
		77	6			Nectarines	
		74	7			Tomatoes	
		73	5			Apple	
		61	16			Maize	
Rühl, 2000	0.02-0.2	86	14	71	112	Potato	None
	0.02-10	90	16	70	110	Carrot root	
	0.02-50	87	10	76	99	Onion foliage	
	0.02-10	87	15	71	108	Onion bulb	
	0.01-10	84	13	62	120	Celery	
	0.02-5	91	14	77	112	Tobacco fresh	
	0.02-5	98	18	75	120	Tobacco cured	
	0.02-1	82	12	70	100	Apples	
	0.02-0.2	91	11	77	104	Stone Fruit	
	0.02-5	90	13	74	104	Oranges	
	0.02-10	110	10	99	120	Grapefruit	
	0.02-3	83	12	67	110	Tomato	
Françon *et al.*, 2000	0.02-0.2	83	16	60	104	Lettuce	None
	0.02-0.2	83	16	65	106	Melon peel	
	0.02-0.2	79	10	59	88	Melon pulp	
	0.02-0.2	88	15	67	104	Potato tuber	
	0.02-0.2	81	19	59	106	Sugar beet roots	
	0.02-0.2	84	12	68	96	Sugar beet leaves/tops	

Reference	Fortification (mg/kg)	Mean recovery (%)	SD (%)	Recoveries (%) low	Recoveries (%) high	Sample	Control interference
Françon et al., 2001	0.01-0.1	94	15	76	116	Melon peel	None
	0.01-0.1	83	14	67	98	Melon pulp	
	0.01-0.1	85	20	57	108	Lettuce	
	0.01-0.1	98	9	84	112	Sugar beet root	
	0.01-0.1	87	17	62	105	Sugar beet leaves/tops	
	0.01-0.1	99	11	82	112	Potato tuber	
	0.01-0.1	84	9	75	97	Citrus peel	
	0.01-0.1	80	18	60	111	Citrus pulp	

Methods for enforcement

In the method of Fillion *et al*. (2000) extraction with acetonitrile is followed by a salting-out step, then solid-phase extraction on an octadecyl cartridge followed by a coupled carbon and aminopropyl SPE cartridges. Determination is by gas chromatographywith mass-selective detection or by liquid chromatography with post-column reaction and fluorescence detection. The LOQ was 0.1 mg/kg and the LOD 0.03 mg/kg for apples, carrots and lettuce.

De Kok *et al*. (1987) extracted fruit or vegetable samples with acetone and subsequently 50/50 dichloromethane/petroleum ether using a homogeniser. After centrifugation, the organic extract is decanted, evaporated to dryness, and redissolved in dichloromethane using an ultrasonic bath. The sample is passed through an aminopropyl-bonded silica SPE column and the eluate collected. After elution with 99/1 dichloromethane/methanol, the eluate is evaporated to dryness, reconstituted in 28/72 acetonitrile/water and filtered. Processed grain samples are extracted with 50/50 dichloromethane/acetone by soaking overnight. The organic extract is concentrated and purified as for fruits and vegetables. Determination is by reverse-phase HPLC with fluorescence detection (340 and 455 nm). The multi-residue method was developed for 21 pesticides (Table 13).

In a development of the method (de Kok and Hiemstra, 1992) the SPE clean-up is automated and determination is by on-line SPE-HPLC (Table 13).

Table 13. Validation of The Netherlands' multi-residue regulatory method 2 for the determination of oxamyl in crops.

Reference	Sample	No.	Recoveries, %, at fortification, mg/kg, and (coefficient variation) 0.05	0.2	0.5	0.13
de Kok et al., 1987	Grain	5	98 (1.2)		102 (0.4)	
	Apple	2	73		77	
	Beans	2	68		76	
	Carrot	2	68		70	
	Carrot	5		73 (3.4)		
	Cauliflower	2	66		73	
	Cauliflower	5		73 (1.8)		
	Celery	2	66		73	
	Cucumber	2	66		69	
	Leek	2	65		71	
	Onion	2	67		77	
	Orange	2	81		86	
	Potato	2	67		75	
	Spinach	2	68		73	
	Strawberry	2	77		77	
de Kok and Hiemstra, 1992	Apple	5				75 (2.57)
	Beans	5				72 (2.48)
	Carrot	5				70 (2.86)
	Cauliflower	5				72 (2.54)
	Endive	5				70 (1.75)
	Onion	5				71 (3.59)
	Orange	5				69 (1.90)

Reference	Sample	No.	Recoveries, %, at fortification, mg/kg, and (coefficient variation)			
			0.05	0.2	0.5	0.13
	Paprika	5				71 (1.55)
	Peach	5				67 (1.64)
	Potato	5				71 (2.53)
	Strawberry	5				66 (1.72)
	Rice	5				101 (2.05)

Soil

The residues of oxamyl and oxamyl oxime in soil are determined as in crops by the method of Holt and Pease (1976). The LOQ is 0.02 mg/kg with a 25-g sample.

In the method by Prince (1982) liquid chromatography is used based upon a HPLC measurement of the intact oxamyl and oxamyl oxime after clean-up on a Sep-Pak™ silica cartridge. The LOQ is 0.01 mg/kg with a 50-g sample.

The method of Cicotti (1996) is essentially that of Holt and Pease (1976) with modifications to improve clean-up and complete the hydrolysis of oxamyl. Oxamyl and oxamyl oxime are extracted with a mixture of ethyl acetate and water. After initial partition with hexane, oxamyl is completely hydrolyzed by heating in an aqueous alkali. A second partition with chloroform is followed by extraction of oxamyl oxime with a mixture of ethyl acetate and methanol. The evaporated extract redissolved in an appropriate solvent is analysed by HPLC with UV detection. The LOQ is 0.01 mg/kg with a 50-g sample.

In the method of Brisbin (2001) extraction is with 80:20 acetonitrile/methanol mixture in an accelerated solvent extractor. The extract is concentrated and then diluted with aqueous 0.01% formic acid for analysis by reverse-phase LC/MS/MS detection. The LOQ is 0.001 mg/kg with a 13-g sample.

Powley and Mol (2001) confirmed Brisbin's method but carried out extraction without a pressurized extractor. Soil samples are extracted with a pre-heated solution of formic acid in methanol/acetonitrile with mechanical shaking. An aliquot of the extract is transferred to 10/90 methanol/0.1% formic acid in 10 mM ammonium acetate and analysed by LC/MS/MS. The method has an LOQ of 0.005 mg/kg for oxamyl and oxamyl oxime with a 20-g sample.

Water

Methods for generation of residue data

Oxamyl is determined by direct injection of ground water samples onto a reverse-phase HPLC column with on-line derivatization and fluorescence detection. The LOQ is 0.002 mg/l with a 0.5-ml sample (McIntosh, 1988).

Oxamyl and oxamyl oxime are determined by direct HPLC of water samples, following acidification with an aqueous solution of formic acid. No other concentration or clean-up steps are required. Analysis is with LC/MS/MS using an atmospheric pressure ionization (API). The method has a LOQ of 0.001 mg/l with a 0.1-ml sample (Wickremesinhe *et al.*, 1999).

Oxamyl is concentrated [??? Tenses] from the water sample and selectively purified by solid-phase extraction. Detection and quantitative analyses by reverse-phase HPLC and LC/MS/MS gave an LOQ of 0.0001 mg/l with a 100-ml sample (Hill *et al.*, 2001).

Methods for enforcement

Oxamyl is extracted and separated on a graphite carbon solid-phase extraction cartridge in the method of Johnson *et al.* (1997). After elution with methanol, the eluate is diluted with methanol and filtered. Determination is by reverse-phase HPLC with post-column hydrolysis forming methylamine and derivatization with *o*-phthalaldehyde and 2-mercaptoethanol and fluorescence detection at excitation and emission wavelengths of 330 and 466 nm respectively. The LOQ is 0.005 mg/l.

Surface water samples are filtered through 0.45 μm PTFE fibre-glass and concentrated on a C-18 bonded silica disk (Honing *et al.*, 1996). After elution with acetonitrile, the eluate is evaporated almost to dryness and dissolved in methanol. Determination is by LC-MS and the LOQ is 0.0001 mg/l.

The method of Chiron *et al.* (1995) for monitoring of carbamate insecticides in groundwater was applied to 17 pesticides including oxamyl. The sample is filtered, acidified to pH 2 with sulfuric acid, and separated by means of an automated on-line SPE system. Determination is by reverse-phase HPLC as described by Johnson *et al.* (1997). The LOD is 0.00001 mg/l; the LOQ was not reported.

The determination of oxamyl is included in US EPA method 531.1 (Munch, 1995). The water sample is treated with monochloroacetic acid buffer to adjust the pH to 3, then filtered through a 0.45 μm millipore type HA filter. An aliquot is chromatographed on a reverse-phase HPLC column with gradient elution. After elution from the column, the analytes are hydrolyzed with 0.05 N sodium hydroxide at 95°C and derivatized and detected by fluorescence as above. The LOQ for oxamyl is 0.002 mg/l.

Stability of pesticide residues in stored analytical samples

Oxamyl is to be considered the only toxicologically significant compound in the total residue. Recent supervised residue trials have been conducted using liquid chromatography methods capable of determining oxamyl directly (Françon *et al.*, 2000). But the historic and current gas chromatographic enforcement method for crops (Holt and Pease, 1976) converts oxamyl to oxamyl oxime and reports the total residues of the two analytes. Owing to the use of this method, current national MRLs for plant commodities are based on the total residues of oxamyl and oxamyl oxime. The stabilities of oxamyl and oxamyl oxime have been studied separately in a variety of frozen crops. The results are shown in Tables 19 (oxamyl) and 20 (oxamyl oxime).

Table 14. Freezer storage stability of oxamyl in crops.

Sample	Spike, mg/kg	Storage, months	% remaining after storage		% procedural recovery	% remaining, adjusted [1]		Reference report-no.	Method
Apple	2	0	80	90	85	94	106	Lin and Tomic, 1991i	Holt and
		6	95	110	95	100	116	AMR 1398-89;	Pease,
		12	75	75	100	75	75	McClory *et al.*, 1992c	1976
		18	90	85	100	90	85	AMR 1398-89	
		24	100	90	105	95	86	Supplement no. 1	
		30	110	105	110	100	105		
		36	90	90	90	100	90		
Celery	3	0	93	93	100	93	93	Lin and Kennedy, 1990b	Holt and
		6	77	80	93	83	86	AMR-1012-87;	Pease,
		12	100	100	95	105	105	Lin and Tomic, 1991a	1976
		18	100	100	113	88	88	AMR-1012-87,	
		24	77	73	107	72	68	Supplement no. 1	
		30	87	103	80	109	129		
		36	113	93	87	130	107		
Cotton seed	0.2	0	75	95	75	100	127	Lin and Kennedy, 1990e	Holt and
		6	70	70	80	88	88	AMR-1016-87;	Pease,
		12	70	60	100	70	60	Lin and Tomic, 1991e	1976
								AMR-1016-87,	
								Supplement no. 1	

Sample	Spike, mg/kg	Storage, months	% remaining after storage		% procedural recovery	% remaining, adjusted [1]		Reference report-no.	Method
		25.5	80	75	90	89	83		
		30	100	75	95	105	79		
		36	90	85	105	86	81		
Cotton seed oil	0.2	0	95	75	95	100	79	Lin and Kennedy, 1990f	Holt and Pease, 1976
		6	90	80	90	100	89	AMR-1017-87;	
		12	100	100	95	105	105	Lin and Tomic, 1991f	
		18	95	95	95	100	100	AMR-1017-87,	
		24	110	105	115	96	91	Supplement no. 1	
		30	90	85	90	100	77		
		36	110	110	95	116	116		
Cucumber	2	0	95	95	90	106	106	Lin and Tomic, 1991m	Holt and Pease, 1976
		6	100	90	85	118	106	AMR 1402-89;	
		12	100	100	95	105	105	McClory et al., 1992g	
		18	75	65	80	94	81	AMR 1402-89	
		24	95	90	95	100	95	Supplement no. 1	
		30	80	75	80	100	94		
		36	110	105	110	100	95		
Leaf lettuce	0.5	0			97			Dubey, 2001	Françon et al., 2000
		3	77	91	81	95	112	DuPont-4235	
		6	90	86	84	107	102		
		12	86	90	91	95	99		
Mint	10	0	82	94	91	90	103	Lin and Tomic, 1991g	Holt and Pease, 1976
		6	76	77	110	69	70	AMR 1396-89;	
		12	71	87	110	65	79	McClory et al., 1992a	
		18	76	72	110	69	65	AMR 1396-89	
		24	92	85	110	84	77	Supplement no. 1	
		30	88	90	77	114	117		
		36	92	96	94	98	102		
Onion	0.1	0	74	74	82	90	90	Lin and Tomic, 1991h	Holt and Pease, 1976
		6	74	81	87	85	93	AMR 1397-89;	
		12	73	70	86	85	81	McClory et al., 1992b	
		18	90	100	110	82	91	AMR 1397-89	
		24	92	97	120	77	81	Supplement no. 1	
		30	82	87	79	104	110		
		36	76	86	93	82	92		
Orange	3	0	87	77	77	103	100	Lin and Tomic, 1990	Holt and Pease, 1976
		6	87	77	77	103	100	AMR 1013-87;	
		12	93	107	103	90	103	Lin and Tomic, 1991b	
		18	107	100	107	100	93	AMR 1013-87	
		24	83	93	83	100	112	Supplement no. 1	
		30	97	97	117	83	83		
		36	107	110	110	97	100		
Orange peel	0.5	0						Dubey, 2001	Françon et al., 2000
		6	95	87	73	130	119	DuPont-4235	
		12	94	91	93	101	98		
Peanut	0.2	0	80	90	90	89	100	Lin and Tomic, 1991k	Holt and Pease, 1976
		6	70	80	120	58	67	AMR 1400-89;	
		19	65	65	85	76	76	McClory et al., 1992e	
		24	85	80	95	89	84	AMR 1400-89	
		30	120	110	100	120	110	Supplement no. 1	
		36	95	85	85	112	100		
Pine-apple	1	0	93	93	94	99	99	Lin and Tomic, 1991l	Holt and Pease, 1976
		6	73	96	100	73	96	AMR 1401-89;	
		12	86	89	82	105	109	McClory et al., 1992f	
		18	84	100	95	88	105	AMR 1401-89	
		24	110	97	88	125	110	Supplement no. 1	
		30	63	62	91	69	68		
		36	61	64	96	64	67		
Potato tuber	0.5	0			83			Dubey, 2001	Françon et al., 2000
		3	73	80	82	89	98	DuPont-4235	
		6	82	73	77	106	95		
		12	88	70	75	117	93		
Potato tuber	0.1	0	93	85	96	97	89	Lin and Kennedy, 1990c	Holt and Pease, 1976
								AMR 1014-87;	
								Lin and Tomic, 1991c	
								AMR 1014-87	
								Supplement no. 1	

Sample	Spike, mg/kg	Storage, months	% remaining after storage		% procedural recovery	% remaining, adjusted [1]		Reference report-no.	Method
		6	97	88	88	110	100		
		12	110	110	94	117	117		
		18	80	80	101	79	79		
		24	85	120	110	77	109		
		30	81	85	86	94	99		
		36	81	93	97	84	96		
Soya bean	0.2	0	95	95	110	86	86	Lin and Tomic, 1991j AMR 1399-89; McClory *et al.*, 1992d AMR 1399-89 Supplement no. 1	Holt and Pease, 1976
		5.5	75	85	90	83	94		
		10.5	90	110	95	95	116		
		15	80	65	110	73	59		
		18	75	85	90	83	94		
		24	65	70	95	68	74		
		30	120	105	80	150	131		
Sugar beet root	0.5	0			74			Dubey, 2001 DuPont-4235	Françon *et al.*, 2000
		3	81	83	80	101	104		
		6	85	83	81	105	104		
		12	73	70	73	100	96		
Tomato	2	0	90	75	70	129	107	Lin and Kennedy, 1990d AMR 1015-87; Lin and Tomic, 1991d AMR 1015-87 Supplement no. 1	Holt and Pease, 1976
		6	110	100	95	116	105		
		12	90	100	90	100	111		
		18	80	95	100	80	95		
		24	75	90	80	94	113		
		30	80	80	80	100	100		
		36	100	100	110	91	91		
Tomato	0.5	0			90			Dubey, 2001 DuPont-4235	Françon *et al.*, 2000
		3	87	78	83	105	94		
		6	83	91	79	105	115		
		12	85	76	85	100	89		

[1] (% remaining after storage ÷ % procedural recovery) x 100

Table 15. Freezer storage stability of oxamyl oxime in crops fortified at 1 mg/kg (Sumpter and Orescan, 1994; method of Holt and Pease, 1976).

Storage, months	Apples		Celery		Cotton seed		Cucumbers	
	% rem	% recov	% rem	% recov	% rem	% recov	% rem	% recov
0	90	86	92	87	90	96	78	72
1	80	94	86	90	76	87	84	87
3	80	72	90	84	89	92	82	101
6	89	74	92	101	84	73	76	78
12	93	97	86	72	76	95	86	100
18	100	106	92	106	85	81	98	106
24	90	85	110	112	110	111	88	88
	Onions		Oranges		Peanuts		Pineapples	
0	98	99	94	84	86	82	88	91
1	93	101	92	91	79	88	92	94
3	110	102	77	72	76	83	88	78
6	98	85	89	94	83	76	86	77
12	75	116	90	83	82	86	95	73
18	110	104	87	80	80	105	96	98
24	98	105	90	82	88	82	92	84
	Potatoes		Soya beans		Spearmint		Tomatoes	
0	94	74	76	79	89	86	78	82
1	84	74	82	80	75	74	84	82
3	86	79	74	71	72	75	100	96
6	88	97	88	82	86	102	94	81
12	100	99	84	73	88	84	94	88
18	98	102	76	86	80	77	110	93
24	84	84	82	90	84	120	90	95

% rem: % remaining after storage, mean of 2 samples
% recov: % procedural recovery, 1 sample

The stability of oxamyl in animal samples stored frozen was not separately investigated as metabolism studies have shown that no oxamyl residues are to be expected in animal food commodities (milk, eggs, edible tissues). But it was shown by Ali *et al.*(1993) that low levels of oxamyl were not stable in frozen beef liver. The initial residues of 0.017 or 0.013 mg/kg were lost completely within two weeks.

The stabilities of oxamyl and oxamyl oxime have been determined in frozen soil and water samples both by the GLC method of Holt and Pease (1976) and the HPLC method of Wickremesinhe *et al.* (1999). The results are shown in Tables 16 (oxamyl) and 17 (oxamyl oxime).

Table 16. Freezer storage stability of oxamyl in soil and water.

Sample	Fortification, mg/kg	Storage, months	% remaining after storage		% procedural recovery		Reference report-no.	Method
Soil	1	0	77	100	87	112	Lin and Kennedy, 1990a AMR-999-87	Holt and Pease, 1976
		6	66	65	89	88		
		12	100	100	100	100		
		18	78	92	69	81		
Soil	0.01	13 – 14	75 70 71 75				Rühl, 2001 AMR 4713-97	Wickremesinhe *et al.*, 1999
	0.06	13-14	97 83 89 85					
	0.1	13-14	78 75 70 88					
Ground water	0.01	0	114	97			Rühl, 2001 AMR 4713-97	Wickremesinhe *et al.*, 1999
		1	96	98				
		4	99	100				
		6	74	72				
Soil-pore water	0.01	0	88	100			Rühl, 2001 AMR 4713-97	Wickremesinhe *et al.*, 1999
		1	100	100				
		4	99	103				
		6	101	107				

Table 17. Freezer storage stability of oxamyl oxime in soil and water.

Sample	Fortification, mg/kg	Storage, months	% remaining after storage	Reference, report-no.	Method reference
Soil	0.01	13 – 14	53 52 29 84	Rühl, 2001 AMR 4713-97	Wickremesinhe *et al.*, 1999
	0.06	13-14	60 59 53 70		
	0.1	13-14	67 63 54 54		
Ground water	0.01	0	96 95	Rühl, 2001 AMR 4713-97	Wickremesinhe *et al.*, 1999
		1	103 102		
		4	97 95		
		6	82 85		
Soil-pore water	0.01	0	84 94	Rühl, 2001 AMR 4713-97	Wickremesinhe *et al.*, 1999
		1	97 99		
		4	88 96		
		6	91 93		

Definition of the residue

Oxamyl is rapidly metabolized in rats, goats and hens and has not been isolated intact in animal products. None of the metabolites contain the carbamoyl moiety. The main metabolite identified in milk, eggs and livestock tissues was thiocyanate.

On the basis of the metabolism studies on potatoes, peanuts, tobacco, tomatoes, oranges, and apples, oxamyl oxime (IN-A2213) and *N*-demethyl oxime (IN-L2953) are the main degradation products of oxamyl. None of the metabolites contains the carbamoyl moiety responsible for cholinesterase inhibition. The Meeting concluded that the only residue of toxicological concern in any

plant tissue is oxamyl. But most of the supervised trials samples were analysed by the GLC method of Holt and Pease (1976) which converts oxamyl to oxamyl oxime and reports the total residues of the two analytes as oxamyl.

Owing to the nature of the residues in the reported supervised trials the Meeting recommended that the definition of the residue for compliance with MRLs should be the sum of oxamyl and oxamyl oxime expressed as oxamyl.

For the estimation of dietary intake the definition of the residue should be oxamyl *per se*. Because the estimated STMRs and HRs are based on the sum of oxamyl and oxamyl oxime, the Meeting noted that an overestimate in the dietary intake calculations could not be excluded.

This definition of the residue applies to both plant and animal commodities.

USE PATTERN

Oxamyl is used as a systemic insecticide and nematicide to control chewing and sucking insect pests on a wide range of crops in many countries. Information on registered uses was reported to the Meeting and is shown in Table 18. Unless otherwise stated certified labels and their English translations were submitted.

Table 18. Registered uses of oxamyl.

Crop	Country	F/G	Form	Amount Ai/form.	Application				PHI, days
					Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Apple	Chile	F	SL	240 g/l	foliar		0.048-0.06	repeat as necessary	14
	Mexico	F	SL	240 g/l	foliar	0.48			14
	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow application	3 g/tree			-
	Saudi Arabia	F	SL	240 g/l	spray at roots during spring, repeat after 3 month	3.6 g/tree		2[2]	14-21
	Saudi Arabia	F	SL	240 g/l	foliar	0.96			14-21
	USA	F	SL	240 g/l	foliar, repeat at 7-14 day intervals, max. 3741 l and min. 467 l water/ha	0.56-2.2 max. 2.2 kg ai/ha per season		1-4	14
Apple (non-bearing)	Canada	F	SL	240 g/l	drench at tree base		0.03	1	-
	Canada	F	SL	240 g/l	foliar treatment after drench	max. 2.2 kg/ha per applic.	0.1- 0.2	1-3	-
	Canada	F	SL	240 g/l	foliar	max. 2.2 kg/ha per applic.	0.036-0.072	apply as needed	-
	Chile	F	SL	240 g/l	soil incorporation, pre-planting	9.6		1	-
	Chile	F	SL	240 g/l	foliar	max. 2.2 kg ai/ha per season	0.06-0.12	repeat every 2-3 weeks	-
	USA	F	SL	240 g/l	foliar	2.2	0.23	4	-
					soil incorporation, pre-planting	8.9	4.8	1	-
Banana	Australia	F	SL	240 g/l	spotgun, apply at 3-month interval	1.8-3 g/plant		3	-
	Central America[1]	F	SL	240 g/l	rhizome immersion		0.032	1	-
					spotgun at base, 7-14 days interval	2.4 g/plant		3-4	-
	Colombia	F	SL	240 g/l	spotgun	1.8 g/plant			-
	Costa Rica	F	SL	240 g/l	rhizome immersion		0.03	1	-
					spotgun	2.4 g/plant		3-4	-
	Ecuador	F	SL	240 g/l	spotgun	2.4 g/plant			1
	Egypt	F	SL	240 g/l	applied to corm	3.6 g/plant		2	1
	France	F	SL	240 g/l	in planting hole	1.8 g/plant		1	-
	France	F	SL	240 g/l	on soil around base of plant, 3-4 months interval	1.8 g/plant		3	-
	Morocco	F	GR	100 g/kg	soil incorporation	5		if necessary	30
	Morocco	F	SL	240 g/l	soil drip irrigation, 30 days interval	1.4		3-4	30

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	Portugal	F	SL	240 g/l	soil drip irrigation, 2-3 months interval	1.9 g/plant			-
	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow application	3 g/plant			-
	Saudi Arabia	F	SL	240 g/l	spray at root zone during spring, repeat after 3 months	3.6 g/plant		2[2]	14-21
					foliar	0.96			14-21
	Spain	F	SL	240 g/l	soil drip irrigation, 3 weeks interval	1.9		3	30
	USA (Puerto Rico only)	F	SL	240 g/l	spotgun, apply to corm, at or post planting, 3-4 months interval	1.4 g/plant, max. 4.5 kg/ha per season			1
	Venezuela	F	SL	240 g/l	soil, post-planting		1.2 – 2.4		
					soil, at planting around plant base	2.4 g/plant			
					soil, at planting	0.24-0.48	0.06 – 0.12		
Beans	Peru	F	SL	240 g/l	foliar		0.096	2	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
Beets	Chile	F	SL	240 g/l	soil in furrow at seeding	0.96		1	
Black currants	New Zealand	F	SL	240 g/l	foliar, 1st applic. pre-flowering 2nd immediately post-flowering	0.48	0.048	2	42
Burdock	Japan	F	GR	8 g/kg	soil incorporation, before seeding	3		1	-
Cabbage	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
Cantaloupe	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	USA	F	SL	240 g/l	soil incorporation, band or broadcast, pre-planting or at planting	4.5 max. 6.7 kg/ha per season			1
	USA	F	SL	240 g/l	foliar	1.1 max. 6.7 kg/ha per season		1-6[2]	1
Carrot	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	14
	Chile	F	SL	240 g/l	soil incorporation, pre-planting	8.9		1	14
	Chile	F	SL	240 g/l	soil in furrow at planting	4.3		1	14
	Hungary	F	GR	100 g/kg	soil in furrow at sowing	3		1	-
	Japan	F	GR	8 g/kg	soil incorporation, before seeding	4		1	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	New Zealand	F	SL	240 g/l	soil in furrow at seeding	4.8	1.2-2.4	1	-
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	USA (except California)	F	SL	240 g/l	soil in furrow at planting	4.5 max. 8.9 kg/ha per season	2.4	1	14
	USA (except California)	F	SL	240 g/l	soil broadcast, pre-planting	8.9 max. 8.9 kg/ha per season	4.8	1	14

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	USA (except California)	F	SL	240 g/l	soil directed spray, 2-3 weeks interval	1.1 max. 8.9 kg/ha per season	0.6	3	14
Celery	Central America[1]	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	14
	Chile	F	SL	240 g/l	soil incorporation, pre-planting	4.4		1	14
	Chile	F	SL	240 g/l	in irrigation water, 2-4 weeks after planting and 2-3 weeks later	2.3		2	14
	Chile	F	SL	240 g/l	foliar, 2-4 weeks after planting and 2-3 weeks later	2.3		2	14
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days intervals	1.2		4	3
	Kuwait	F	GR	100 g/kg	at plant base with incorporation	0.1 g/plant			14
	Mexico	F	SL	240 g/l	foliar	0.96			14
	Mexico	F	SL	240 g/l	soil drip irrigation	0.96			14
	Mexico	F	SL	240 g/l	soil in furrow pre-planting	4.8		2	14
	Oman	F	GR	100 g/kg	soil at plant base	0.1 g/plant			14
	Peru	F	SL	240 g/l	soil		0.12	2	3
	Peru	F	SL	240 g/l	foliar		0.12	3	3
	Poland	F	SL	240 g/l	poured onto seedlings, pre-planting		0.096	1	14
	Qatar	F	GR	100 g/kg	at plant base	0.1 g/plant			14
	United Arab Emirates	F	GR	100 g/kg	at plant base with soil incorporation	0.1 g/plant			14
	USA (AZ, CA, FL)	F	SL	240 g/l	foliar, ground or aerial, 5-7 days interval	0.56-1.1 max. 6.7 kg/ha per season	0.6-1.2		21
	USA (FL, OH, PA, MI, TX)	F	SL	240 g/l	transplant treatment	1.1-2.2 max. 6.7 kg/ha per season	0.24	1	21
	USA (FL, OH, PA, MI, TX)	F	SL	240 g/l	foliar, 3 weeks after transplanting, repeat 3 weeks after first applic.	2.2 max. 6.7 kg/ha per season	0.24	2	21
	USA (FL, OH, PA, MI, TX)	F	SL	240 g/l	pre-planting row treatment with soil incorporation	4.5 max. 6.7 kg/ha per year	2.4	1	21
	USA (FL, OH, PA, MI, TX)	F	SL	240 g/l	foliar treatment as extension of pre-planting treatment	1.1 max. 6.7 kg/ha per season	0.6	2	21
	USA (CA only)	F	SL	240 g/l	band treatment or shank injection, after seeding or transplanting, repeat 21-30 days after first treatment	1.1 max. 6.7 kg/ha per season		2	21
Cereals	Jordan	F	SL	240 g/l	foliar, 3 weeks interval		0.12-0.24		14
	Kuwait	F	SL	240 g/l	foliar, 3 weeks interval		0.12-0.24		14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96-1.9			21
	United Arab Emirates	F	SL	240 g/l	foliar, 3 weeks intervals		0.06-0.24	repeat if necessary	14
Cherry (non-bearing)	USA	F	SL	240 g/l	foliar	2.2	0.23	4	-
	USA	F	SL	240 g/l	soil incorporation, pre-planting	8.9	4.8	1	-
Citrus	Central America[1]	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Chile	F	SL	240 g/l	foliar, before flowering		0.024-0.06	repeat as necessary	7
	Chile	F	SL	240 g/l	foliar	2..3-4.4 max. 13 kg/ha per season		3-6	7
	Chile	F	SL	240 g/l	soil drip irrigation	2.3		repeat every 30 days	7
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Egypt	F	GR	100 g/kg	soil incorporation, row/band treatment	6		1	7
	Egypt	F	SL	240 g/l	not stated in the label	0.48		2	7
	Jordan	F	SL	240 g/l	foliar		0.12-0.24		14
	Kuwait	F	SL	240 g/l	foliar		0.12-0.24		14

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow soil application	3 g/tree			-
	Saudi Arabia	F	SL	240 g/l	spray at root zone during spring, repeat after 3 months	3.6 g/tree		2	14-21
	Saudi Arabia	F	SL	240 g/l	foliar	0.96			14-21
	Spain	F	SL	240 g/l	soil drip irrigation	1.4 g/tree			30
	Taiwan	F	SL	240 g/l	foliar	0.24-0.72			7
	Thailand	F	SL	240 g/l	foliar		0.12		7
	United Arab Emirates	F	SL	240 g/l	foliar		0.12-0.24	repeat if necessary	14
	USA	F	SL	240 g/l	foliar, 2-3 weeks interval	1.1 max. 6.7 kg/ha per season	0.0075-0.03		7
	USA	F	SL	240 g/l	foliar, apply in early spring before flower, at petal fall and midsummer, ground or aerial	0.56-1.1 max. 6.7 kg/ha per season	0.02 – 0.12 (by ground) 0.6 – 1.2 aerial		7
	USA	F	SL	240 g/l	soil irrigation, do not apply more than 2.2 kg/ha in any 30 day period	0.56-2.2 max. 6.7 kg/ha per season			7
Citrus (non-bearing)	USA	F	SL	240 g/l	foliar	2.2	0.23	4	-
	USA	F	SL	240 g/l	soil incorporation, pre-planting	8.9	4.8	1	-
Coffee	Central America[1]	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
Corn (maize)	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	in furrow or at base of plant	1.5-2.5		1	14
Cotton	Colombia	F	SL	240 g/l	foliar, ground or aerial	0.48	0.85-1.7		-
	Mexico	F	SL	240 g/l	foliar	0.48-2.4			21
	Peru	F	SL	240 g/l	foliar	0.48-0.72			21
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96-1.9			21
	Spain	F	GR	100 g/kg	broadcast, pre-planting	4-6		1	30
	USA	F	SL	240 g/l	foliar, 6-8 days interval	0.28-1.1 max. 4.5 kg/ha per season		4	21
	USA (AL, AR, GA, LA, MS)	F	SL	420 g/l	soil broadcast or foliar, ground or aerial, with covering, 7-14 days interval	0.28-0.52	0.7-1.1	2	14
	USA	F	SL	420 g/l	foliar, 6-8 days interval	0.52 – 1.1 max. 4.5 kg/ha per season	0.14 – 1.1	4	14
	Venezuela	F	SL	240 g/l	foliar	0.48	0.12 – 0.24	4	
Courgette, see Squash, Summer									
Cucumber	Belgium	G	GR	100 g/kg	soil incorporation, pre-planting	5-10		1	-
	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution, 7-day interval	0.25			
	Bulgaria	G	GR	100 g/kg	soil incorporation, pre-planting	1500 g/plant		1	-
	Central America[1]	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	3
	Chile	F	SL	240 g/l	foliar	0.6-1.1			
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Hungary	F	GR	100 g/kg	soil incorporation broadcast, pre-planting	3		1	-
	Japan	F	GR	8 g/kg	direct at base of plant, nursery period	0.02 g/plant		2	-
	Japan	F	GR	8 g/kg	soil incorporation, pre-planting	4		2	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Lebanon	F	GR	100 g/kg	at plant base	0.1 g/plant			-

| Crop | Country | F/G | Form | Amount Ai/form. | Application | | | | PHI, days |
					Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
	Oman	F	GR	100 g/kg	soil broadcast. with incorporation, pre-planting	3-5		1	14
	Poland	G	SL	240 g/l	foliar		0.024		7
	Qatar	F	GR	100 g/kg	soil broadcast. with incorporation	3-5		1	14
	Romania	G	GR	100 g/kg	after transplanting	6.4		1	-
	Romania	F	SL	240 g/l	not stated on the label		0.048		14
	Saudi Arabia	F/G	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F/G	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F/G	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar spray on leaf, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Spain	F	SL	240 g/l	soil drip irrigation after planting, repeat after 3 weeks	1.4-1.9		4	3
	Syria	F	GR	100 g/kg	foliar	0.1 g/plant			3
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	3
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	3
	Syria	F	GR	100 g/kg	soil band application at planting	5-18 g/ 100 m row		1	3
	Turkey	F	GR	100 g/kg	soil incorporation, pre-planting	5		1	3
	United Arab Emirates	F/G	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2-5		1	14
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1- 3
	USA	F	SL	240 g/l	soil band or broadcast with incorporation, pre-planting or at planting	2.2-4.5 max. 6.7 kg/ha per season			1
	USA	F	SL	240 g/l	foliar, repeat weekly	0.56-1.1 max. 6.7 kg/ha per season		repeat as necessary	1
	Venezuela	F	SL	240 g/l	foliar	0.24-0.72			
Cucurbits	Chile	F	SL	240 g/l	soil incorporation, pre-planting	4.3		1	
	Chile	F	SL	240 g/l	foliar, 2-4 weeks after planting and 2-3 weeks later	1.1		2	7
	Lebanon	F	GR	100 g/kg	foliar	0.1 g/plant			1- 3
	Morocco	F	SL	240 g/l	soil drip irrigation, 2 weeks interval	1.4-1.9		1-4	7
	Syria	F	GR	100 g/kg	foliar	0.1 g/plant			3
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	3
	Syria	F	GR	100 g/kg	soil incorporation at furrow, post-planting	1-3		1	3
	Syria	F	GR	100 g/kg	soil band application at planting	5-18 g/ 100 m row		1	3
Date palm	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow soil application	3 g/tree			-
Egg plant	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution, 7 days interval	0.25		3-4	3
	Japan	F	GR	8 g/kg	direct application at base of plant, nursery period	0.02 g/plant		1	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	GR	100 g/kg	at plant base with soil incorporation	0.1 g/plant			14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Lebanon	F	GR	100 g/kg	foliar	0.1 g/plant			1-3
	Oman	F	GR	100 g/kg	at plant base	0.1 g/plant			14
	Poland	G	SL	240 g/l	foliar		0.024		7
	Qatar	F	GR	100 g/kg	at plant base	0.1 g/plant			14

Crop	Country	F/G	Form	Amount Ai/form.	Application				PHI, days
					Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
	Romania	G	GR	100 g/kg	after transplant	6.4		1	-
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar spray, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	SL	240 g/l	soil drip irrigation after planting, repeat after 3 weeks	1.4-1.9		4	3
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Syria	F	GR	100 g/kg	foliar	0.1 g/plant			3
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	3
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1 – 3		1	3
	Syria	F	GR	100 g/kg	soil band application at planting	5-18 g/ 100 m row		1	3
	United Arab Emirates	F/G	GR	100 g/kg	at plant base with soil incorporation	0.0001 kg/plant			14
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	USA	F	SL	240 g/l	foliar, ground, repeat at 1-3 weeks intervals	0.56-1.1 max. 6.7 kg ai/ha			1
	USA (except CA)	F	SL	240 g/l	soil (band), 2-3 weeks after transplanting, 2nd applic. 4 weeks later (additional foliar treatment at 1- to 2-week intervals 2 to 4 weeks after the second soil treatment possible)	2.2 soil 1.1 foliar max. 6.7 kg ai/ha		2	7
Figs	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow soil application	3 g/tree			-
Fodder beet	Austria	F	SL	245 g/l	soil band application at planting and before emergence	1.2	0.6-0.96	2	-
	Poland	F	SL	240 g/l	foliar	0.12-4.8		1	14
Garlic	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	14
	Chile	F	SL	240 g/l	immersion		0.12	1	
	Colombia	F	SL	240 g/l	immersion		0.18	1	-
	Hungary	F	GR	100 g/kg	soil, in furrow at sowing	3		1	-
	Kuwait	F	GR	100 g/kg	soil at planting	2-4		1	14
	Oman	F	GR	100 g/kg	soil at planting	2-4		1	14
	Peru	F	SL	240 g/l	immersion		1.2	1	-
	Peru	F	SL	240 g/l	foliar		0.12		-
	Qatar	F	GR	100 g/kg	soil at planting	2-4		1	14
	Romania	F	GR	100 g/kg	soil, in furrow at planting	15		1	-
	Romania	F	GR	100 g/kg	soil broadcast at planting	5-6		No.	-
	Romania	F	SL	240 g/l	not stated on label		0.048		14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-plant	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar spray on leaf, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	United Arab Emirates	F	GR	100 g/kg	soil at planting	2-4		1	14
	USA (OR)	F	SL	240 g/l	foliar, ground or aerial, repeat at 5-7 days interval, aerial 4 days interval	0.56-1.1 max. 5 kg/ha per season	0.6 – 2.4		14
	USA (CA, OR)	F	SL	240 g/l	in-furrow drench or in-furrow spray at planting	2.2	0.24 – 1.2	1	14
	USA (CA, OR)	F	SL	240 g/l	in-furrow band spray at planting	4.5	2.4	1	14
	USA (CA, OR)	F	SL	240 g/l	soil post emergence band, 14-21-day interval	2.2	1.2	2-3	14
	USA (CA, OR)	F	SL	240 g/l	pressurized sprinkler system after planting	2.2 max. 5 kg/ha per season			14
Gherkins	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution, 7 day intervals	0.25		3-4	3

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Application Rate, kg ai/ha or g ai/plant	Application Spray conc., kg ai/hl	Application No.	PHI, days
Ginger root	USA (HI)	F	SL	240 g/l	preplanting broadcast or band with incorporation	4.5		1	30
	USA (HI)	F	SL	240 g/l	foliar or post-planting band, apply at monthly	1.1 max. 11 kg/ha per growing season		6	30
Gourd, white	Japan	F	GR	8 g/kg	direct application to dibble, at planting	0.02 g/plant		1	-
Grapes	Bulgaria	F	GR	100 g/kg	soil incorporation, pre-planting	0.2 g/plant		1	-
	Egypt	F	SL	240 g/l	ns	1.2		2	14
	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow soil application	3 g/tree			-
Grapevine (non-bearing)	Chile	F	SL	240 g/l	immersion, pre-planting		0.6	1	-
	Chile	F	SL	240 g/l	foliar, post-planting		0.12	4	-
Indian poke	Japan	F	GR	8 g/kg	soil application with incorporation, before seeding	4		1	-
Leaf mustard	Taiwan	F	SL	240 g/l	foliar	0.24-0.48			6
Lettuce	Central America[1]	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days interval	1.2	0.3	4	3
	Guatemala	F	SL	240 g/l	foliar	1.2	0.3	4	7
	Japan	F	GR	8 g/kg	soil incorporation, pre-planting	4		1	-
Melons	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution, 7 days interval	0.25			
	Central America[1]	F	SL	240 g/l	foliar	1.2		4	3
	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	7
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7
	Ecuador	F	SL	240 g/l	foliar, 15 days after planting	0.48-1.2	0.12-0.6	4	1
	Japan	F	GR	8 g/kg	direct at base of plant, nursery period	0.02 g/plant		1	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1- 3
	Kuwait	F	GR	100 g/kg	broadcast with incorporation, pre-planting	3-5		1	14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Oman	F	GR	100 g/kg	broadcast with incorporation, pre-planting	3-5		1	14
	Portugal	G	SL	240 g/l	drip irrigation from May-September, 2-3 months interval	1.9		2-3	-
	Qatar	F	GR	100 g/kg	broadcast with incorporation	3-5		1	14
	Spain	F	GR	100 g/kg	broadcast pre-planting	4-6		1	30
	Spain	F	SL	240 g/l	drip irrigation	1.4-1.9		4	3
	United Arab Emirates	F/G	GR	100 g/kg	broadcast with incorporation, pre-planting	2-5		1	14
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	USA	F	SL	240 g/l	soil band/broadcast pre-planting or at planting	2.2-4.5		1	1
	USA	F	SL	240 g/l	foliar	0.56-1.1 max. 6.7 kg/ha per season			1
	Venezuela	F	SL	240 g/l	foliar	0.24-0.72			
Mint	USA (ID, MI, MT, OR, WA, WI)	F	SL	240 g/l	foliar	3.4 max. 4.5 kg/ha per season		2	21
Olives	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow	3 g/tree			-
Onion	Central America[1]	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	7

Crop	Country	F/G	Form	Amount Ai/form.	Application				PHI, days
					Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	14
	Chile	F	SL	240 g/l	in furrow pre-planting	4.8		1	14
	Chile	F	SL	240 g/l	irrigation water plus	1.7		2-3	14
	Chile	F	SL	240 g/l	foliar at a 4-21 days interval	0.72			
	Costa Rica	F	SL	240 g/l	foliar, 7-14 days interval	1.2		4	3
	Hungary	F	GR	100 g/kg	soil in furrow at sowing	3		1	-
	Kuwait	F	GR	100 g/kg	at planting	2-4		1	14
	Lebanon	F	GR	100 g/kg	soil incorporation, pre-planting	2.5-5		1	-
	Lebanon	F	GR	100 g/kg	soil in furrow post planting	1-3		1	-
	Lebanon	F	GR	100 g/kg	soil band at planting	50-180 g/ 100 m row		1	-
	Oman	F	GR	100 g/kg	soil at planting	2-4		1	14
	Poland	F	SL	240 g/l	foliar	0.48	0.08-0.24	2	14
	Qatar	F	GR	100 g/kg	at planting	2-4		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4 – 6		1	30
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5 – 5		1	14
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	14
	Syria	F	GR	100 g/kg	band at planting	5-18 g/ 100 m row		1	14
	Turkey	F	GR	100 g/kg	soil incorporation, pre-planting	6		1	21
	United Arab Emirates	F	GR	100 g/kg	soil at planting	2 – 4		1	14
	USA (MI, NM,TX)	F	SL	240 g/l	foliar, repeat applic. at 5-7 days interval	0.28-0.56 max. 5 kg/ha per season	0.6 – 2.4		14
	USA (MI, TX, ID, OR, WA)	F	SL	240 g/l	soil in-furrow drench or in-furrow spray at planting	1.1-2.2 max. 5 kg/ha per season	0.24 – 1.2	1	14
	USA (MI, TX, ID, OR, WA)	F	SL	240 g/l	soil in-furrow band spray at planting	3.4-4.5 max. 5 kg/ha per season	2.4	1	14
	USA (MI, TX, ID, OR, WA,CA)	F	SL	240 g/l	soil post emergence band, 14-21 days interval	1.1-2.2 max. 5 kg/ha per season	1.2	2-3	14
	USA (ID, OR, WA, CA)	F	SL	240 g/l	pressurized sprinkler system after planting	2.2 max. 5 kg/ha per season		2-3[2]	14
Parsley	Hungary	F/G	GR	100 g/kg	soil in furrow at sowing	3		1	-
	Lebanon	F	GR	100 g/kg	foliar	0.1 g/plant			14
	Lebanon	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	14
	Lebanon	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	14
	Lebanon	F	GR	100 g/kg	soil band at planting	5-18 g/ 100 m row		1	14
Pasture	New Zealand	F	SL	240 g/l	foliar or before cultivation or direct drilling	0.48	0.1-0.48		-
Peach (non-bearing)	USA	F	SL	240 g/l	foliar	2.2	0.23	4	-
					soil, pre-planting with incorporation	8.9	4.8	1	-
Peanut	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96-1.92			21
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	in furrow or at base of plant	1.5-2.5		1	14
	Taiwan	F	SL	240 g/l	in trench, foliar 20-40 days after planting	0.3-0.36		3	-
	USA (except CA)	F	SL	240 g/l	soil, band with incorporation, pre-planting	1.1-3.4 max. 5.6 kg/ha per season	3.6	1	-
	USA (except CA)	F	SL	240 g/l	soil at planting, band	1.1-3.4 max. 5.6 kg/ha per season	3.6	1	-

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	USA (except CA)	F	SL	240 g/l	foliar, post-planting, 1st applic. 3 weeks after emergence, 2nd 3 weeks later	0.56-1.1 max. 5.6 kg/ha per season	0.6	2	-
	USA (except CA)	F	SL	420 g/l	soil, band, pre- or at planting with incorporation	1.1-3.4 max. 5.6 kg/ha per season	3.3	1	-
	USA (except CA)	F	SL	420 g/l	foliar	0.56-1.1 max. 5.6 kg/ha per season	0.56	2	-
Pear	Chile	F	SL	240 g/l	foliar		0.048-0.06	repeat as necessary	7
	Saudi Arabia	F	GR	100 g/kg	soil broadcast at base of tree	3 g/tree			-
	Saudi Arabia	F	SL	240 g/l	spray at roots during spring, repeat after 3 months	3.6 g/tree			14-21
	Saudi Arabia	F	SL	240 g/l	foliar	0.96			14-21
	USA (except CA)	F	SL	240 g/l	foliar, ground only	1.7-2.2 max. 2.2 kg/ha per season	0.24		14
Pear (non-bearing)	Chile	F	SL	240 g/l	soil incorporation, pre-planting	9.6		1	-
	Chile	F	SL	240 g/l	foliar	max. 2.2 kg/ha per season	0.06 -0.12	repeat every 2-3 weeks	-
	USA	F	SL	240 g/l	foliar	0.56-2.2 max. 8.9 kg /ha	0.23	4	-
	USA	F	SL	240 g/l	soil, pre-planting with incorporation	6.7-8.9	4.8	1	-
Peas	Lebanon	F	GR	100 g/kg	soil incorporation, pre-planting	2.5 – 5		1	-
	Lebanon	F	GR	100 g/kg	soil in furrow post planting	1-3		1	-
	Lebanon	F	GR	100 g/kg	soil band at planting	50-180 g/ 100 m row		1	-
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	-
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	-
	Syria	F	GR	100 g/kg	soil band at planting	5-18 g/ 100 m row		1	-
Peppers	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution	0.38		3-4	3
	Central America[1]	F	SL	240 g/l	foliar	1.2	0.3	4	7
	Hungary	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3		1	-
	Japan	F	GR	8 g/kg	direct at base of plant, nursery period	0.02 g/plant		1	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Lebanon	F	GR	100 g/kg	foliar	0.1 g/plant			7
	Mexico	F	SL	240 g/l	foliar	0.96		1-4	7
	Mexico	F	SL	240 g/l	soil drip irrigation	0.96			7
	Oman	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Poland	G	SL	240 g/l	foliar		0.024		7
	Qatar	F	GR	100 g/kg	soil broadcast, incorporation	3-5		1	14
	Romania	G	GR	100 g/kg	soil after transplanting	6.4		1	-
	Romania	F	SL	240 g/l	not stated on the label		0.048		14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Application Rate, kg ai/ha or g ai/plant	Application Spray conc., kg ai/hl	Application No.	PHI, days
	Saudi Arabia	F	SL	240 g/l	foliar, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Spain	F	SL	240 g/l	soil drip irrigation after planting, repeat after 3 weeks	1.4-1.9		4	3
	Syria	F	GR	100 g/kg	foliar	0.1 g/plant			7
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	7
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	7
	Syria	F	GR	100 g/kg	soil band at planting	5-18 g/ 100 m row		1	7
	United Arab Emirates	F/G	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2-5		1	14
	United Arab Emirates	F	SL	240 g/l	Foliar		0.06-0.18		1-3
	USA	F	SL	240 g/l	transplanting water treatment,	0.56	0.03	1	7
	USA	F	SL	240 g/l	soil drip irrigation, 14 days after transplanting, repeat at 1-2-weeks interval	0.56-1.1 max. 6.7 kg/ha per season	0.06		7
	USA	F	SL	240 g/l	foliar spray, 1-2 weeks interval	0.56-1.1 max. 6.7 kg/ha per season			7
Pineapple	Ecuador	F	SL	240 g/l	foliar PHI: 30 days nematodes, 7 days mealy bugs, weevils	1.2	0.3-0.6	4-8	7 – 30
	Kenya	F	SL	240 g/l	soil drip irrigation, pre-planting, 4-5 months interval	9.6			30
	Kenya	F	SL	240 g/l	soil drip irrigation, pre-planting, 30 days interval	2.4			30
	Kenya	F	SL	240 g/l	foliar	0.96 – 1.9			14
	Mexico	F	SL	240 g/l	soil incorporation, pre-planting	0.96		1	30
	Mexico	F	SL	240 g/l	soil after planting	0.96		1	30
	Mexico	F	SL	240 g/l	soil drip irrigation	0.96			30
	Mexico	F	SL	240 g/l	foliar	0.96			30
	Saudi Arabia	F	SL	240 g/l	spray at roots during spring, repeat after 3 month	3.6 g/plant			14-21
	Saudi Arabia	F	SL	240 g/l	foliar	0.96			14-21
	USA (except CA)	F	SL	240 g/l	soil drip irrigation or broadcast, within 1 week after planting	1.1-4.5 max. 8.9 kg/ha per season		1	30
	USA (except CA)	F	SL	240 g/l	foliar (ground) post-planting, 2-4 weeks interval	1.1-2.2 max. 8.9 kg/ha per season			30
	USA (except CA)	F	SL	240 g/l	drip irrigation, post-planting, 2-, 4-, or 8 weeks intervals	0.56-2.2 max. 8.9 kg/ha per season			30
Pome fruit	Jordan	F	SL	240 g/l	foliar		0.12-0.24		14
	Kuwait	F	SL	240 g/l	foliar		0.12-0.24		14
	New Zealand	F	SL	240 g/l	foliar, 10-14 days interval	0.048 – 0.06			7
	United Arab Emirates	F	SL	240 g/l	foliar		0.12-0.24	repeat if necessary	14
Pome-granate	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow soil	3 g/tree			-
Poppy	Romania	F	SL	240 g/l	foliar	0.24	0.12		-
Potato	Austria	F	SL	245 g/l	soil incorporation, pre-planting	4.8	1.2	1	-
	Belgium	F	GR	100 g/kg	soil incorporation, pre-planting	3-6		1	-
	Canada	F	SL	240 g/l	foliar	0.73	0.08-0.024	weekly or as needed	7
	Chile	F	SL	240 g/l	foliar, 5 day intervals		0.36	4	
	Chile	F	SL	240 g/l	soil incorporation, pre-planting	4.1		1	
	Japan	F	GR	8 g/kg	soil incorporation, pre-planting	2.4		1	-
	Japan	F	GR	8 g/kg	in furrow, pre-planting	1.6		1	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	GR	100 g/kg	soil incorporation, pre-planting	2.5-5		1	14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Lebanon	F	GR	100 g/kg	soil incorporation, pre-planting	2.5-5		1	7
	Mexico	F	SL	240 g/l	foliar	0.96			7
	Mexico	F	SL	240 g/l	soil drip irrigation	0.96			7
	Oman	F	GR	100 g/kg	soil incorporation, pre-planting	2.5-5		1	14
	Peru	F	SL	240 g/l	foliar	0.48-0.72			7
	Peru	F	SL	240 g/l	soil		0.12	2	1
	Poland	F	SL	240 g/l	soil, after planting	3.6		1	14
	Qatar	F	GR	100 g/kg	soil incorporation, pre-planting	2.5-5		1	14
	Romania	F	GR	100 g/kg	soil broadcast at planting	1.8-3		1	-
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Spain	F	SL	240 g/l	soil drip irrigation at 2-3-leaf-stage, repeat every 2-3 weeks	1.4-1.9			30
	Syria	F	GR	100 g/kg	broadcast with incorporation, pre-planting	2.5-5		1	-
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	-
	Syria	F	GR	100 g/kg	band application at planting	5-18 g/ 100 m row		1	-
	United Arab Emirates	F	GR	100 g/kg	soil, at planting	2.5-5		1	-
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	UK	F	GR	100 g/kg	soil incorporation, pre-planting	4-5.5		1	-
	USA (except CA)	F	SL	240 g/l	soil, in-furrow pre-planting	2.2-4.5 max. 10 kg/ha per season	2.4	1	7
	USA (except CA)	F	SL	240 g/l	foliar, ground or I, apply at 5-7-day intervals	0.56-1.1 max. 10 kg/ha per season	3	6	7
	USA (CT, DE, MA, MD, ME, NH, NJ, NY, PA, RI, VA,VT)	F	SL	420 g/l	foliar, ground or I, apply at 5-7-day intervals	0.56-1 max. 6.7 kg/ha per season	2.8	6	7
	USA (except Northeast, Mid Atlantic States, CA)	F	SL	420 g/l	soil, in-furrow pre-planting	2.2-4.5 max. 10 kg/ha per season	2.2	1	7
	USA (except Northeast, Mid Atlantic States, CA)	F	SL	420 g/l	foliar spray or I, apply at 5-7-day intervals	0.56-1 max. 10 kg/ha per season	2.8	6	7
	Venezuela	F	SL	240 g/l	foliar	0.48 – 1.2			
					soil	0.24 – 0.48			
Prunes	Egypt	F	SL	240 g/l	foliar	0.48		2	14
	Saudi Arabia	F	GR	100 g/kg	broadcast at base of tree or in furrow soil	3 g/tree			-
Pumpkin	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	7
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1- 3
	Kuwait	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Oman	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Qatar	F	GR	100 g/kg	soil broadcast with incorporation	3 – 5		1	14

Crop	Country	F/G	Form	Amount Ai/form.	Application				PHI, days
					Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar spray on leaf, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	United Arab Emirates	F/G	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2-5		1	14
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	USA	F	SL	240 g/l	soil band or broadcast with incorporation, pre-planting or at planting	2.2-4.5 max. 6.7 kg/ha per season			1
	USA	F	SL	240 g/l	foliar, repeat weekly	0.56-1.1 max. 6.7 kg/ha per season		repeat as necessary	1
Radish	Japan	F	GR	8 g/kg	soil with incorporation in furrow, before seeding	4		1	-
Raspberry	Canada	F	SL	240 g/l	soil drench, apply only once during 12 month	2.2		1	-
Rice	Ecuador	F	SL	240 g/l	foliar, 15-30 days after germination	0.48	0.12-0.24	1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	in furrow or at base of plant	1.5-2.5		1	14
Rice, paddy	Taiwan	F	GR	100 g/kg	in seed box	6 g/box		1	-
	Taiwan	F	GR	100 g/kg	soil after transplanting	3		1	-
	Taiwan	F	SL	240 g/l	seed soak, pre-planting		0.024	1	-
	Taiwan	F	SL	240 g/l	foliar, 60 days after transplanting		0.032	1	-
Ryegrass	New Zealand	F	SL	240 g/l	foliar or before cultivation or direct drilling	0.48	0.1-0.48		-
Seed potato	Bulgaria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	7		1	-
Soya bean	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96-1.92			21
	USA (except CA)	F	SL	240 g/l	soil in furrow/band, pre- or at planting with incorporation	0.56-1.1	1.2	1	-
	USA (except CA)	F	SL	240 g/l	soil incorporated broadcast pre-planting	2.2-4.5	4.8	1	-
	USA (except CA)	F	SL	420 g/l	soil in furrow/band, pre- or at planting with incorporation	0.56-1	1.1	1	-
	USA (except CA)	F	SL	420 g/l	incorporated broadcast pre-planting	2.2-4.5	4.5	1	-
Squash, Summer (Courgette)	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution, 7 days interval	0.25		3-4	3
	Kuwait	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3 – 5		1	14
	Oman	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Qatar	F	GR	100 g/kg	soil broadcast with incorporation	3-5		1	14
	United Arab Emirates	F/G	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2-5		1	14
	USA	F	SL	240 g/l	soil band or broadcast with incorporation, pre-planting or at planting	2.2-4.5 max. 6.7 kg/ha per season			1
	USA	F	SL	240 g/l	foliar, repeat weekly	0.56-1.1 max. 6.7 kg/ha per season		repeat as necessary	1
	Venezuela	F	SL	240 g/l	foliar	0.24-0.72			
Stone fruit	Chile	F	SL	240 g/l	immersion, pre-planting		0.6	1	-
		F	SL	240 g/l	foliar, post-planting		0.12	4	-
Strawberry sapling	Bulgaria	F	GR	100 g/kg	soil incorporation, pre-planting	0.2 g/plant			1	-
Sugar beet	Austria	F	SL	245 g/l	soil band application at planting and before emergence	1.2	0.6-0.96	2	-

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	Bulgaria	F	GR	100 g/kg	soil in furrow at drilling	2		1	-
	Italy	F	GR	50 g/kg	soil broadcast with incorporation, pre-planting	8		1	-
	Italy	F	GR	50 g/kg	soil in furrow at planting	2		1	-
	Lebanon	F	GR	100 g/kg	soil incorporation, at planting	1 – 1.2		1	-
	Poland	F	SL	240 g/l	foliar	0.12-4.8		1	14
	Romania	F	GR	100 g/kg	soil at sowing in rows	1.5		1	-
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	-
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	-
	Syria	F	GR	100 g/kg	soil band application at planting	5-18 g/ 100 m row		1	-
	Syria	F	GR	100 g/kg	soil at sowing	1-1.2		1	-
	UK	F	GR	100 g/kg	soil in seed furrow, at drilling	0.6 – 0.9		1	-
Sugar cane	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	in furrow or at base of plant	1.5-2.5		1	14
	Taiwan	F	GR	100 g/kg	during planting	4 – 6		1	-
Sweet potato	Japan	F	GR	8 g/kg	soil with incorporation, pre-planting	4		1	-
	USA (except CA)	F	SL	240 g/l	soil broadcast, pre-planting with incorporation	4.5-6.7 max. 6.7 kg/ha per season	3.6	1	-
	USA (except CA)	F	SL	240 g/l	in furrow at planting	2.2-4.5	0.24	1	-
Tobacco	Bulgaria	F	GR	100 g/kg	broadcast with incorporation, pre-planting	10		1	-
	Central America[1]	F	SL	240 g/l	foliar	0.72			3
	Central America[1]	F	SL	240 g/l	ground treatment to seed plot		0.12		
	Central America[1]	F	SL	240 g/l	in furrow treatment after transplant		0.96		
	Chile	F	SL	240 g/l	foliar, 2 weeks after plan-ting and 2-3 weeks later	1.1	0.36 – 0.6	2	
	Chile	F	SL	240 g/l	soil incorporation, pre-planting	4.3	1.1	1	
	Chile	F	SL	240 g/l	transplanting water	0.48		1	
	Ecuador	F	SL	240 g/l	foliar, 15 days after transplanting	1.2	0.3-0.6		-
	Japan	F	GR	8 g/kg	soil incorporation in furrow, pre-planting	3		1	-
	Jordan	F	SL	240 g/l	foliar, repeat applic. at 3 weeks interval		0.12-0.24		14
	Kuwait	F	SL	240 g/l	foliar, 3 weeks interval		0.12-0.24		14
	Macedonia	F	SL	240 g/l	foliar, pre-planting	4.8		1	-
	Mexico	F	SL	240 g/l	soil drip irrigation	0.96			-
	Mexico	F	SL	240 g/l	foliar	0.96		3-4	-
	Mexico	F	SL	240 g/l	soil band	1.4			-
	Mexico	F	SL	240 g/l	soil drench	2.4	1.2		-
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Spain	F	SL	240 g/l	bed or soil drip irrigation after planting, repeat every 3-4 weeks	1.4-1.9		3	30
	Taiwan	F	GR	100 g/kg	soil during planting	6		1	-
	United Arab Emirates	F	SL	240 g/l	foliar, 3 week intervals		0.06-0.24	repeat if necessary	14
	USA	F	SL	240 g/l	soil row with incorporation	2.2 max. 2.2 kg/ha per season	1.2	1	-
	USA	F	SL	240 g/l	soil broadcast and bed, pre-planting	2.2 max. 2.2 kg/ha per season	0.6	1	-
	USA	F	SL	420 g/l	soil row, pre-planting with incorporation	2.2 max. 2.2 kg/ha per season	1.1	1	-

Crop	Country	F/G	Form	Amount Ai/form.	Application				PHI, days
					Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
	USA	F	SL	420 g/l	soil broadcast and bed treatment	2.2 max. 2.2 kg/ha per season	0.56		-
	Venezuela	F	SL	240 g/l	foliar	0.72			
	Venezuela	F	SL	240 g/l	soil irrigation at planting	0.36 – 0.6			
	Venezuela	F	SL	240 g/l	soil incorporation, pre-planting	2.4 – 7.2			
Tomato	Australia	F	SL	240 g/l	soil drip irrigation at planting	4.3		1	-
	Australia	F	SL	240 g/l	soil drip irrigation, 14 days interval	0.48		4	-
	Belgium	G	GR	100 g/kg	soil incorporation, pre-planting	5-10		1	-
	Belgium	G	SL	250 g/l	foliar		0.05 – 0.075	3-4	3
	Belgium	G	SL	250 g/l	add'n to nutrient solution, 7 days interval	0.25		3-4	3
	Bulgaria	G	GR	100 g/kg	soil incorporation, pre-planting	0.0015 g/plant		1	-
	Bulgaria	G	GR	100 g/kg	soil incorporation, pre-planting	10		1	-
	Central America[1]	F	SL	240 g/l	foliar	1.2		4	3
	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	7
	Chile	F	SL	240 g/l	foliar, 1 week after planting, 2 week intervals	0.12		3-6	7
	Chile	F	SL	240 g/l	irrigation water, 1 week after planting, 2 weeks interval	4.3		as necessary	7
	Chile	F	SL	240 g/l	soil incorporation, pre-planting	8.9		1	7
	Costa Rica	F	SL	240 g/l	foliar, 7-14 day intervals	1.2		4	7
	Ecuador	F	SL	240 g/l	foliar, 15 days after planting	0.48	0.12-0.24	4	-
	Egypt	F	GR	100 g/kg	soil in furrow/broadcast	4.8		1	1-3
	Egypt	F	SL	240 g/l	not stated on the label	0.2		2	1
	Hungary	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3		1	-
	Japan	F	GR	8 g/kg	soil incorporation, pre-planting	4		1	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	GR	100 g/kg	broadcast w/incorporation	3-5		1	-
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Lebanon	F	GR	100 g/kg	foliar	0.1 g/plant			1-3
	Macedonia	F	SL	240 g/l	foliar, chemigation	0.48	0.048	1	3
	Mexico	F	SL	240 g/l	foliar	0.96		1-4	1
	Mexico	F	SL	240 g/l	soil drip irrigation	0.96			1
	Morocco	F	GR	100 g/kg	soil after transplanting	5			-
	Morocco	F	SL	240 g/l	soil drip irrigation, 2 weeks interval	1.4-1.9		1-4	7
	Oman	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	3-5		1	14
	Peru	F	SL	240 g/l	soil		0.12	2	1
	Peru	F	SL	240 g/l	foliar		0.12	4	1
	Poland	G	SL	240 g/l	foliar		0.024		7
	Portugal	G	SL	240 g/l	soil drip irrigation, 2-3 month intervals	1.9		2-3	-
	Qatar	F	GR	100 g/kg	soil broadcast with incorporation	3-5		1	14
	Romania	G	GR	100 g/kg	soil after transplanting	6.4		1	-
	Romania	F	SL	240 g/l	not stated on the label		0.048		14
	Saudi Arabia	F/G	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F/G	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F/G	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	GR	100 g/kg	soil broadcast pre-planting	4-6		1	30
	Spain	F	SL	240 g/l	soil drip irrigation after planting, repeat after 3 weeks	1.4-1.9		4	3
	Syria	F	GR	100 g/kg	foliar	0.1 g/plant			3

Crop	Country	F/G	Form	Amount Ai/form.	Method	Rate, kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	PHI, days
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	3
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	3
	Syria	F	GR	100 g/kg	soil band at planting	5-18 g/ 100 m row		1	3
	Taiwan	F	GR	100 g/kg	in trench at planting	2-4		1	-
	Taiwan	F	SL	240 g/l	foliar, 20 and 40 days after transplanting	0.48		2	-
	Turkey	F	GR	100 g/kg	soil incorporation, pre-planting	5		1	3
	Turkey	G	SL	100 g/l	soil drip irrigation, at planting and 15 days after planting	7		2	3
	United Arab Emirates	F/G	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2-5		1	14
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	USA	F	SL	240 g/l	foliar, 5-7-day intervals	0.56-1.1 max. 8.9 kg/ha per season	1.2 – 3		3
	USA	F	SL	240 g/l	soil drip irrigation, 1-2-week intervals	0.56-2.2 max. 8.9 kg/ha per season			3
	USA (CA only)	F	SL	240 g/l	shank injection	0.84-1.4		3	3
	Venezuela	F	SL	240 g/l	seed bed and 30 days after planting	12	0.08-0.11		
	Venezuela	F	SL	240 g/l	10, 20, 40, 60 days after planting		0.12	4	
	Venezuela	F	SL	240 g/l	foliar	0.48 – 1.2			
Turnip	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
Watermelon	Chile	F	SL	240 g/l	foliar	0.6-1.1		every 5-10 days	7
	Ecuador	F	SL	240 g/l	foliar, 15 days after planting	0.48-1.2	0.12-0.6	4	1
	Japan	F	GR	8 g/kg	direct at base of plant	0.02 g/plant		2	-
	Japan	F	GR	8 g/kg	soil incorporation, pre-planting	2.8		2	-
	Jordan	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Jordan	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Kuwait	F	SL	240 g/l	foliar, after transplanting and 3 weeks later (mites)		0.06		7-14
	Kuwait	F	SL	240 g/l	foliar or soil, after transplanting and 3 weeks later (nematodes)		0.18		1-3
	Lebanon	F	GR	100 g/kg	foliar	0.1 g/plant			1-3
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast, at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
	Saudi Arabia	F	SL	240 g/l	soil drip irrigation or foliar	0.96 – 1.9			7-10
	Saudi Arabia	F	SL	240 g/l	foliar spray on leaf, 2 weeks after germination	0.48 – 0.96		repeat if necessary	7-10
	Spain	F	SL	240 g/l	soil drip irrigation after planting, repeat after 3 weeks	1.4-1.9		4	3
	Syria	F	GR	100 g/kg	foliar	0.1 g/plant			3
	Syria	F	GR	100 g/kg	soil broadcast with incorporation, pre-planting	2.5-5		1	3
	Syria	F	GR	100 g/kg	soil incorporation in furrow, post planting	1-3		1	3
	Syria	F	GR	100 g/kg	soil band application at planting	5-18 g/ 100 m row		1	3
	Taiwan	F	SL	240 g/l	foliar		0.04		6
	United Arab Emirates	F	SL	240 g/l	foliar		0.06-0.18		1-3
	USA	F	SL	240 g/l	soil band or broadcast with incorporation, pre-planting or at planting	4.5 max. 6.7 kg /ha per season			1
	USA	F	SL	240 g/l	foliar, repeat weekly	1.1 max. 6.7 kg /ha per season		repeat as necessary	1

Crop	Country	F/G	Form	Amount Ai/form.	Application Method	Application Rate, kg ai/ha or g ai/plant	Application Spray conc., kg ai/hl	Application No.	PHI, days
	Venezuela	F	SL	240 g/l	foliar	0.24-0.72			
Wheat	Saudi Arabia	F	GR	100 g/kg	soil broadcast, pre-planting	5		1	14
	Saudi Arabia	F	GR	100 g/kg	soil broadcast at planting	2		1	14
	Saudi Arabia	F	GR	100 g/kg	soil in furrow or at base of plant	1.5-2.5		1	14
Yam	Japan	F	GR	8 g/kg	soil incorporation, pre-planting	4		1	-
	USA (Puerto Rico only)	F	SL	240 g/l	seed piece dip, pre-planting	1.1-2.2	0.24	1	60
	USA (Puerto Rico only)	F	SL	240 g/l	foliar, 2 month after planting, 2-week-intervals	0.56 max. 13.5 kg/ha per season)	0.24	12	60

[1] Countries include Guatemala, Belize, El Salvador, Honduras, Nicaragua, Panama and the Dominican Republic
[2] Not specified on the label
✈   Aerial application

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials on

Fruits	Table 19.	Citrus fruits
	Table 20.	Apple
	Table 21.	Banana
	Table 22.	Pineapple
Vegetables	Table 23.	Cucumber
	Table 24.	Melons, watermelons and squash
	Table 25.	Peppers
	Table 26.	Tomato
	Table 27.	Carrot
	Table 28.	Potato
	Table 29.	Celery
Oilseed	Table 30.	Cotton seed
	Table 31.	Peanut

For older trials conducted between 1971 and 1979 before GLP was required field reports were not available, so there was no information on crop variety, plot and sample sizes, or freezer storage. But the laboratory reports included method validation and documented recoveries at spiking levels similar to those occurring in samples from supervised trials. Although dates of analyses were not reported the trials were carried out according to the guidelines of the time. Because there are no doubts about the validity, these trials were not excluded from the evaluation. Newer trials carried out between 1989 and 1999 were generally well documented with full laboratory and field reports.

Where residues were undetected, the results are recorded in the Tables as below the LOQ, e.g. <0.01 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Although trials included control plots, control data are only included when residues exceeded the LOQ. Residues are recorded unadjusted for procedural recoveries.

Most trials were with single plots. Replicate residues are from split plots. The period of freezer storage between sampling and analysis were recorded for the newer trials (1989–1999) and were within the acceptable determined stability period of 3 years. Because the reported trials used the analytical method of Holt and Pease (1976) based on the hydrolysis of oxamyl to the oxime, residues are reported as the sum of oxamyl and oxamyl oxime expressed as oxamyl except in Table 25

(analyses by HPLC method of Thean *et al*., 1977). Double underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels, STMRs and HRs.

Table 19. Residues in whole citrus fruits from supervised trials in the USA, foliar broadcast spray treatment. (Reports 3G 1349, 7F 1909, du Pont de Nemours and Co., 1972 and 1976b). Field report unavailable.

Location, Year	Application						PHI, days	Residues, mg/kg	Report no.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval			
GRAPEFRUIT									
Bradenton, FL 1972	SL	2.1	0.015	14031	1		2 4 8	0.06 0.08 0.08	3G 1349, 7F 1909
Bradenton, FL 1972	SL	4.3	0.03	14031	1		2 4 8	0.08 0.07 0.05	3G 1349, 7F 1909
Bradenton, FL 1972	SL	2.1 4.3	0.015 0.03	14031 14031	2 2	1 month 1 month	14 14	<0.02 <0.02	3G 1349, 7F 1909
Davenport, FL 1972	SL	0.71	0.015	4676	1		3 6 9	0.03 0.03 <0.02	3G 1349, 7F 1909
Ft. Pierce, FL 1972	SL	0.60	0.03	1983	1		1 5 13	0.25 0.11 0.03	3G 1349, 7F 1909
Mesa, AZ 1972	SL	1.1	0.012	9353	1		1 3 7 14	0.14 0.14 <u>0.13</u> 0.1	3G 1349, 7F 1909
Woodlake, CA 1972	SL	2.2	0.06	3742	1		1 2 5 7	1.3 0.67 0.84 0.3	3G 1349, 7F 1909
Woodlake, CA 1972	SL	4.5	0.12	3742	1		1 2 5 7	1.5 1.1 0.64 0.64	3G 1349, 7F 1909
Monte Alto, TX 1972	SL	0.71	0.015	4676	1		2 7 14	0.63 0.29 0.08	3G 1349, 7F 1909
Monte Alto, TX 1972	SL	1.5	0.031	4676	1		7 14	<u>1.2</u> 0.81	3G 1349, 7F 1909
Weslaco, TX 1972	SL	0.71	0.015	4676	1		2 7 14	2.1 1.2 0.3	3G 1349, 7F 1909
Weslaco, TX 1972	SL	1.4	0.03	4676	1		2 7 14	3.6 <u>2.0</u> 0.46	3G 1349, 7F 1909
Waverly, FL 1975 2 replicate plots	SL	2.2 2.2 2.2	2.4 0.048 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	<0.02 <0.02 <0.02	7F 1909
		2.2 2.2 2.2	2.4 0.048 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	<0.02 <0.02 <0.02	
Waverly, FL 1975 2 replicate plots	SL	2.2 4.5 2.2	2.4 0.096 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	0.06 <0.02 <0.02	7F 1909
		2.2 4.5 2.2	2.4 0.096 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	<0.02 <0.02 <0.02	
Lake Wales, FL 1975 2 replicate plots	SL	2.2 2.2 2.2	0.19 0.048 0.048	1169 4676 4676	3	7 months 4 months	3 5 7	0.26 0.27 0.18	7F 1909

Location, Year	Application						PHI, days	Residues, mg/kg	Report no.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval			
		2.2	0.19	1169	3	7 months	3	0.24	
		2.2	0.048	4676		4 months	5	0.42	
		2.2	0.048	4676			7	0.19	
Lake Wales, FL 1975 2 replicate plots	SL	2.2	0.19	1169	3	7 months	3	0.31	7F 1909
		4.5	0.096	4676		4 months	5	0.17	
		4.5	0.096	4676			7	0.1	
		2.2	0.19	1169	3	7 months	3	0.56	
		4.5	0.096	4676		4 months	5	0.34	
		4.5	0.096	4676			7	0.19	
LEMON									
Yuma, AZ 1972	SL	1.1			1		1	0.29	3G 1349, 7F 1909
							2	0.25	
							4	0.18	
							8	<u>0.05</u>	
Yuma, AZ 1972	SL	2.2			1		1	0.39	3G 1349, 7F 1909
							2	0.29	
							4	0.04	
							8	0.07	
Yuma, AZ 1972	SL	4.5			1		1	0.55	3G 1349, 7F 1909
							2	0.36	
							4	0.32	
							8	0.36	
Los Gatos, CA 1972	SL	1.1	0.03	3742	1		1	0.21	3G 1349, 7F 1909
							3	0.17	
							7	<u>0.17</u>	
ORANGE									
Ft. Pierce, FL 1971	SL	1.1	0.03	3742	1		7	<u>0.16</u>	3G 1349, 7F 1909
							14	0.06	
							21	<0.04	
Cutler, CA 1971	SL	1.1	0.24	468	5	1 month	1	0.13	3G 1349, 7F 1909
							2	0.11	
							4	<0.04	
							8	<u><0.04</u>	
Bradenton, FL 1972	SL	2.1	0.015	14031	1		2	0.12	3G 1349, 7F 1909
							4	0.15	
							8	0.05	
Bradenton, FL 1972	SL	4.3	0.03	14031	1		2	0.22	3G 1349, 7F 1909
							4	0.25	
							8	0.13	
Bradenton, FL 1972	SL	2.1	0.015	14031	2	1 month	14	<0.02	3G 1349, 7F 1909
							21	<0.02	
Bradenton, FL 1972	SL	4.3	0.03	14031	2	1 month	14	<0.02	3G 1349, 7F 1909
							21	0.03	
Los Gatos, CA 1972	SL	1.1	0.03	3742	1		1	0.15	3G 1349, 7F 1909
							3	0.05	
							7	<u>0.14</u>	
Lake Alfred, FL 1972	SL	1.4	0.015	9353	1		2	0.51	3G 1349, 7F 1909
							4	0.42	
							8	<u>0.34</u>	
							15	0.06	
							21	0.04	
Davenport, FL 1972	SL	0.71	0.015	4676	1		3	0.02	3G 1349, 7F 1909
							6	0.02	
							9	<0.02	
Woodlake, CA 1972	SL	2.2	0.06	3742	1		1	0.31	3G 1349, 7F 1909
							2	0.52	
							5	0.92	
							7	0.6	
Woodlake, CA 1972	SL	4.5	0.12	3742	1		1	2.0	3G 1349, 7F 1909
							2	0.85	
							5	0.6	
							7	0.85	

Location, Year	Application					PHI, days	Residues, mg/kg	Report no.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval		
Mesa, ARIZ. 1972	SL	1.1	0.012	9353	1		1 3 7 14	0.6 0.44 <u>0.3</u> 0.25	3G 1349, 7F 1909
Mission, TX 1972	SL	1.1	0.024	4676	1		2 7 14 21	1.1 <u>0.8</u> 0.37 0.3	3G 1349, 7F 1909
Mission, TX 1972	SL	2.2	0.048	4676	1		7 14	3.0 0.86	3G 1349, 7F 1909
Bradenton, FL 1972	SL	2.2	0.24	935	6	6 months 4 months 3 months 6 months 6 months	3 7	0.27 0.14	3G 1349, 7F 1909
Clermont, 1974	SL	4.5	0.064	7015	3	2 months 3 months	1 2 3 7 14	4.8 4.4 3.6 2.0 0.67	7F 1909
Waverly, FL 1975 2 replicate plots	SL	2.2 2.2 2.2	2.4 0.048 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	<0.02 0.06 0.03	7F 1909
		2.2 2.2 2.2	2.4 0.048 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	0.02 <0.02 0.03	
Waverly, FL 1975 2 replicate plots	SL	2.2 4.5 2.2	2.4 0.096 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	0.14 0.09 <0.02	7F 1909
		2.2 4.5 2.2	2.4 0.096 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	0.04 0.09 0.02	
Lake Wales, FL 1975 2 replicate plots	SL	2.2 2.2 2.2	0.19 0.048 0.048	1169 4676 4676	3	7 months 4 months	3 5 7	0.42 0.3 0.61	7F 1909
		2.2 2.2 2.2	0.19 0.048 0.048	1169 4676 4676	3	7 months 4 months	3 5 7	0.33 0.18 0.19	
Lake Wales, FL 1975 2 replicate plots	SL	2.2 4.5 4.5	0.19 0.096 0.096	1169 4676 4676	3	7 months 4 months	3 5 7	0.75 0.65 0.37	7F 1909
		2.2 4.5 4.5	0.19 0.096 0.096	1169 4676 4676	3	7 months 4 months	3 5 7	1.1 0.54 0.2	
TANGELO									
Weslaco, TX 1972	SL	4.2	0.03	14031	3	1 month 2 months	84	0.04	3G 1349, 7F 1909
Haines City, FL 1975	SL	2.2 2.2 2.2	0.06 0.038 0.038	3742 5846 5846	3	2 months 4 months	4 6 8	0.06 0.02 0.04	7F 1909
		2.2 4.5 4.5	0.06 0.077 0.077	3742 5846 5846	3	2 months 4 months	4 6 8	0.88 0.24 0.36	
TANGERINE									
Waverly, FL 1975 2 replicate plots	SL	2.2 2.2 2.2	2.4 0.048 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	<0.02 0.02 <0.02	7F 1909
		2.2 2.2 2.2	2.4 0.048 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	0.1 0.03 0.03	
Waverly, FL 1975 2 replicate plots	SL	2.2 4.5 2.2	2.4 0.096 2.4	94 ✈ 4676 94 ✈	3	3 months 2 months	3 5 7	0.04 <0.02 <0.02	7F 1909

Location, Year	Application						PHI, days	Residues, mg/kg	Report no.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval			
		2.2	2.4	94 ✈	3	3 months	3	<0.02	
		4.5	0.096	4676		2 months	5	0.02	
		2.2	2.4	94 ✈			7	<0.02	
Haines City, FL 1975	SL	2.2	0.06	3742	3	2 months	4	0.28	7F 1909
		2.2	0.038	5846		4 months	6	0.1	
		2.2	0.038	5846			8	0.02	
Haines City, FL 1975	SL	2.2	0.06	3742	3	2 months	4	0.57	7F 1909
		4.5	0.077	5846		4 months	6	0.16	
		4.5	0.077	5846			8	0.4	

✈ Aerial application

Table 20. Residues in apples from supervised trials in the USA, foliar broadcast spray treatment. Field report unavailable for trials marked in bold (Report 3G 1349, du Pont de Nemours and Co., 1972).

Location, year (variety)	Application						PHI, days	Residues, mg/kg	Reference, report no.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval, days			
Wapato, WA, 1972 (Golden Delicious)	SL	1.1	0.03	3742	1		3	0.84	3G 1349
							7	0.68	
	SL	2.2	0.06	3742	1		3	1.5	
							7	1.4	
	SL	4.5	0.12	3742	1		3	1.9	
							7	1.4	
Wapato, WA, 1972 (Red Bisbee)	SL	1.1	0.03	3742	1		7	0.37	3G 1349
		2.2	0.06	3742	1		7	0.8	
		4.5	0.12	3742	1		7	1.2	
Fennville, MI, 1972 (Red Delicious)	SL	1.1	0.03	3742	2	2 months	2	0.32	3G 1349
							4	0.23	
							8	0.11	
							14	0.09	
							21	0.09	
	SL	2.2	0.06	3742	2	2 months	2	0.52	3G 1349
							4	0.31	
							8	0.43	
							14	0.3	
							21	0.27	
Biglerville, PA, 1972 (Golden Delicious)	SL	0.84	0.03	2806	2	1 month	14	1.2	3G 1349
Arlington, WI, 1972 (Red Delicious)	SL	0.76	0.032	2338	1		2	0.34	3G 1349
							7	0.09	
							10	0.08	
							14	0.04	
							20	<0.02	
Stevens City, VA, 1988 (Golden Delicious)	SL	2.2	0.46	495	2	14	14	0.39	Lin and Hay, 1990d
		4.5	0.91	495	2	14	14	0.72	AMR 1062-88
Fairfield, PA, 1988 (Golden Delicious)	SL	2.2	0.23	963	2	14	14	0.69	Lin and Hay, 1990d
		4.5	0.47	963	2	14	14	0.73	AMR 1062-88
Newburgh, NY, 1988 (Red Delicious)	SL	2.2	0.30	750	2	14	14	0.08	Lin and Hay, 1990d
		4.5	0.60	750	2	14	14	0.18	AMR 1062-88
Wenatchee, WA, 1988 (Red Delicious)	SL	2.2	3.4	65 ✈	2	14	14	0.03	Lin and Hay, 1990d
		4.5	6.8	65 ✈	2	14	14	0.22	AMR 1062-88

Location, year (variety)	Application						PHI, days	Residues, mg/kg	Reference, report no.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval, days			
Madera, CA, 1993 (Rome)	SL	1.1 1.1	0.23 0.25	477 450	2	7	14 14 14 21 28	0.11 0.17 0.15 0.13 <0.1	McClory and Tomic, 1995 AMR 2569-93
	SL	2.2	0.5	450	1	7	14 14 14 21 28	0.19 0.52 0.28 0.12 0.18	
Zillah, WA, 1993 (Granny Smith)	SL	1.1	0.24	468	2	7	14 14 14 21 28	1.2 1.2 1.1 1.1 0.68	McClory and Tomic, 1995 AMR 2569-93
	SL	2.2	0.48	468	1	7	14 14 14 21 28	0.81 1.1 0.73 0.58 0.22	
Granger, WA, 1993 (Red Delicious)	SL	1.1	0.24	468	2	7	14 14 14 21 28	0.39 0.49 0.49 0.34 0.15	McClory and Tomic, 1995 AMR 2569-93
	SL	2.2	0.48	468	1	7	14 14 14 21 28	0.59 0.52 1.2 0.54 0.17	
Zillah, WA, 1993 (Red Delicious)	SL	1.1	0.24	468	2	7	14 14 14 21 28	0.34 0.17 <0.1 0.24 0.2	McClory and Tomic, 1995 AMR 2569-93
	SL	2.2	0.48	468	1	7	14 14 14 21 28	0.44 0.2 <0.1 0.29 0.36	
Ponderay, ID, 1993 (Royal Gala)	SL	1.1	0.21	533	2	7	14 14 14 21 28	<0.1 <0.1 <0.1 <0.1 <0.1	McClory and Tomic, 1995 AMR 2569-93
	SL	2.2	0.42	533	1	7	14 14 14 21 28	≤0.1 <0.1 <0.1 <0.1 <0.1	
North Rose, NY, 1993 (Cortland)	SL	1.1	0.24	468	2	7	14 14 14 21 28	0.15 <0.1 0.15 <0.1 0.24	McClory and Tomic, 1995 AMR 2569-93
	SL	2.2	0.48	468	1	7	14 14 14 21 28	0.12 0.14 0.12 <0.1 0.24	

Location, year	Application						PHI, days	Residues, mg/kg	Reference, report no.
(variety)	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval, days			
Fennville, MI, 1993 (Red Delicious)	SL	1.1	0.24	468	2	7	14	0.11	McClory and Tomic, 1995 AMR 2569-93
							14	0.31	
							14	0.11	
							21	0.12	
							28	0.1	
	SL	2.2	0.48	468	1	7	14	0.21	
							14	0.20	
							14	0.25	
							21	<0.1	
							28	0.12	
North Wilkesboro, NC, 1993 (Spur Red Delicious)	SL	1.1	0.24	468	2	7	14	0.29	McClory and Tomic, 1995 AMR 2569-93
							14	0.23	
							14	0.16	
							21	<0.1	
							28	<0.1	
	SL	2.2	0.48	468	1	7	14	0.49	
							14	0.34	
							14	0.33	
							21	0.31	
							28	0.11	
Romney, WV, 1993 (Red Delicious)	SL	1.1	0.21	525	2	7	14	0.34	McClory and Tomic, 1995 AMR 2569-93
							14	0.25	
							14	0.2	
							21	<0.1	
							28	0.22	
	SL	2.2	0.42	525	1	7	14	<0.1	
							14	<0.1	
							14	0.22	
							21	<0.1	
							28	0.26	
Upper Black Eddy, PA, 1993 (Empire)	SL	1.1	0.23	477	2	7	14	0.18	McClory and Tomic, 1995 AMR 2569-93
							14	<0.1	
							14	0.16	
							21	0.1	
							28	<0.1	
	SL	2.2	0.47	477	1	7	14	0.18	
							14	0.15	
							14	0.16	
							21	0.17	
							28	<0.1	

✈ Aerial application

Table 21. Residues in bananas from supervised trials in Australia after spotgun treatment to base of plant (du Pont de Nemours and Co., 1994; reference of all reports).

Location, year	Application				PHI, days	Residues, mg/kg			Report no.
(variety)	Form	g ai/stool	No.	Interval, days		Pulp	Peel	Whole fruit calc.	
Caboolture, Queensland, 1990	SL	1.8	3	120 90	0	<0.01	<0.01	<0.01	2318/90/5
					1	<0.01	0.03	0.02	
					4	<0.01	<0.01	<0.01	
					7	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	
					28	<0.01	<0.01	<0.01	
	SL	2.4	3	120 90	0	<0.01	<0.01	<0.01	
					1	<0.01	0.02	0.01	
					4	<0.01	<0.01	<0.01	
					7	<0.01	0.01	0.01	
					14	<0.01	0.01	0.01	
					28	<0.01	<0.01	<0.01	

Location, year	Application				PHI,	Residues, mg/kg			Report no.
(variety)	Form	g ai/stool	No.	Interval, days	days	Pulp	Peel	Whole fruit calc.	
	SL	4.8	3	120	0	0.01	0.03	0.02	
				90	1	0.01	0.2	0.08	
					4	<0.01	0.03	0.02	
					7	<0.01	0.03	0.02	
					14	<0.01	0.05	0.03	
					28	<0.01	0.02	0.01	
Murwillumbh, NSW, 1990	SL	1.8	3	100	0	<0.01	<0.01	<0.01	2364/90/5
				90	1	<0.01	<0.01	<0.01	
					4	<0.01	<0.01	<0.01	
					7	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	
					28	<0.01	<0.01	<0.01	
	SL	2.4	3	100	0	<0.01	0.02	0.01	
				90	1	<0.01	<0.01	<0.01	
					4	<0.01	<0.01	<0.01	
					7	<0.01	<0.01	<0.01	
					14	<0.01	0.03	0.02	
					28	<0.01	<0.01	<0.01	
	SL	4.8	3	100	0	<0.01	0.1	0.04	
				90	1	<0.01	<0.01	<0.01	
					4	<0.01	0.02	0.01	
					7	<0.01	<0.01	<0.01	
					14	<0.01	0.05	0.03	
					28	<0.01	<0.01	<0.01	
Innisfail, Queensland, 1992	SL	3	6	100	0	<0.01	<0.01	<0.01	92/0253
				188	1	<0.01	<0.01	<0.01	
				89	7	<0.01	<0.01	<0.01	
				100	14	<0.01	0.01	<0.01	
				237	28	<0.01	0.01	<0.01	
	SL	6	6	100	0	<0.01	<0.01	<0.01	
				188	1	<0.01	0.08	0.03	
				89	7	<0.01	<0.01	<0.01	
				100	14	<0.01	<0.01	<0.01	
				237	28	<0.01	0.05	0.02	
Wamuran, Queensland, 1993	SL	3	1		0	<0.01	<0.01	<0.01	93/2788
					1		0.03		
					7		<0.01		
	SL	6	1		0	<0.01	<0.01	<0.01	
					1		0.03		
					7		<0.01		
Wamuran, Queensland, 1994	SL	3	1		0	<0.01	<0.01	<0.01	94/2361
					1	<0.01	<0.01	<0.01	
					2	<0.01	<0.01	<0.01	
					4	<0.01	<0.01	<0.01	
					7	<0.01	<0.01	<0.01	
	SL	6	1		0	<0.01	<0.01	<0.01	
					1	<0.01	<0.01	<0.01	
					2	<0.01	<0.01	<0.01	
					4	<0.01	<0.01	<0.01	
					7	<0.01	<0.01	<0.01	

Table 22. Residues in pineapples from supervised trials in the USA, spray treatment (du Pont de Nemours and Co., 1977). Field reports unavailable for trials marked in bold. No information on water quantities reported. Lanai, 1975: 2 replicate plots; Kunia, 1975: 4 replicate plots. (Report no. 7F 2000).

Location, year	Application				PHI,	Residues, mg/kg	
	Form	kg ai/ha	No.	Interval	days	Whole fruit	Bran
Lanai, HI, 1975 replicate plot 1 (RAB-7-75)	SL	1.1	6	1 month	13	0.11	0.21
					27	0.43	1.7

Location, year	Application				PHI, days	Residues, mg/kg	
	Form	kg ai/ha	No.	Interval		Whole fruit	Bran
	SL	2.2	6	1 month	13	0.73	3.5
					27	0.38	1.1
	SL	4.5	6	1 month	13	2.5	2.1
					27	0.91	4.5
	SL	1.1	5	1 month	13	0.25	0.57
					27	0.12	1.5
	SL	2.2	5	1 month	13	0.38	0.71
					27	0.14	2.7
	SL	4.5	5	1 month	13	1.6	2.4
					27	0.48	4.4
Lanai, HI, 1975 replicate plot 2 (RAB-8-75)	SL	1.1	6	1 month	13	0.22	0.29
					27	0.06	0.28
	SL	2.2	6	1 month	13	0.27	0.54
					27	0.39	3.2
	SL	4.5	6	1 month	13	1.2	1.9
					27	0.79	5.2
	SL	1.1	5	1 month	13	0.46	0.33
					27	0.15	0.88
	SL	2.2	5	1 month	13	1.1	1.1
					27	0.59	1.4
	SL	4.5	5	1 month	13	1.4	3.0
					27	0.57	2.5
Kunia, HI, 1975 replicate plot 1 (RAB-74)	SL	1.1	1	1 month	14	<0.02	0.06
			2		14	0.03	0.12
			3		14	<0.02	0.1
			4		14	<0.02	0.04
			5		14	0.09	0.1
			6		14	0.03	0.11
	SL	2.2	1	1 month	14	0.03	0.1
			2		14	0.1	0.19
			3		14	0.05	0.15
			4		14	0.06	0.13
			5		14	0.05	0.4
			6		14	0.11	0.11
	SL	4.5	1		14	0.24	0.53
			2	1 month	14	0.19	0.47
			3		14	0.38	0.51
			4		14	0.29	0.44
			5		14	0.21	0.22
			6		14	0.16	0.36
	SL	9	1		14	0.55	1.6
			2	1 month	14	0.55	0.8
			3		14	0.29	1.2
			4		14	0.68	0.72
			5		14	1.2	1.2
			6		14	0.14	0.57
Kunia, HI, 1975 replicate plot 2 (RAB-74)	SL	1.1	1		14	0.02	0.14
			2	1 month	14	0.02	0.12
			3		14	0.04	0.11
			4		14	0.04	0.1
			5		14	<0.02	0.05
			6		14	0.03	0.18
	SL	2.2	1		14	0.1	0.31
			2	1 month	14	0.1	0.23
			3		14	0.12	0.15
			4		14	0.1	0.16
			5		14	0.12	0.22
			6		14	0.12	0.15
	SL	4.5	1		14	0.33	0.27
			2	1 month	14	0.13	0.32
			3		14	0.18	0.39
			4		14	0.21	0.68
			5		14	0.34	0.26
			6		14	0.13	0.4

Location, year	Application				PHI, days	Residues, mg/kg	
	Form	kg ai/ha	No.	Interval		Whole fruit	Bran
	SL	9	1		14	0.82	1.2
			2	1 month	14	0.15	1.2
			3		14	0.15	0.69
			4		14	0.48	1.2
			5		14	0.74	1.7
			6		14	0.47	1.1
Kunia, HI, 1975 replicate plot 3 (RAB-2-74)	SL	1.1	3	1 month	23	0.05	
			4		23	0.16	
			5		23	0.02	
			6		23	0.04	
	SL	2.2	3	1 month	23	0.06	
			4		23	0.05	
			5		23	0.17	
			6		23	0.37	
	SL	4.5	3	1 month	23	0.09	
			4		23	0.32	
			5		23	0.31	
			6		23	0.27	
	SL	9	3	1 month	23	0.43	
			4		23	0.44	
			5		23	0.37	
			6		23	0.26	
Kunia, HI, 1975 replicate plot 4 (RAB-3-74)	SL	1.1	1	1 month	23	<0.02	
			2		23	0.02	
			3		23	0.03	
			4		23	0.02	
			5		23	0.03	
			6		23	0.05	
	SL	2.2	1		23	0.02	
			2	1 month	23	0.02	
			3		23	0.02	
			4		23	0.05	
			5		23	0.04	
			6		23	0.02	
	SL	4.5	3	1 month	23	0.33	
			4		23	0.16	
			5		23	0.14	
			6		23	0.08	
	SL	9	3	1 month	23	0.46	
			4		23	0.25	
			5		23	0.36	
			6		23	0.17	
Kunia, HI, 1976 Plot RAB-8-74	SL	1.1	19	1 month	35	0.09	
		2.2	19	1 month	35	0.36	
Molokai, HI, 1976 Plot RAB-10-74	SL	1.1	18	1 month	42	0.03	
		2.2	18	1 month	42	0.05	
Poamoho, HI, 1976 Plot RAB-18-76	SL	2.2	19	1 month	42	0.04	

Table 23. Residues in cucumbers from outdoor supervised trials in the USA, foliar broadcast SL treatment (du Pont de Nemours and Co., 1979a; Interregional Research Project No. 4, 1985b). Only partially unreadable hand-written field reports were available.

Location, year	Application					PHI, days	Residues, mg/kg	Report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Poston, AR, June 1976	2.2			5		1	0.52	OF 2288 + 4E 3057
						3	0.06	
						7	0.02	

Location, year	Application					PHI, days	Residues, mg/kg	Report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Blythe, CA, June 1976	2.2			5		1	0.37	OF 2288 + 4E 3057
						3	0.03	
						5	<0.02	
San Luis Ray, CA, May 1976	0.56	0.07	814	5		control	0.08	OF 2288 + 4E 3057
						1	0.16	
						6	0.19	
						14	0.07	
	1.1	0.14	814	5		1	0.37	
						6	0.32	
						14	0.15	
	2.2	0.24	935	7	7	1	1.1	
						7	0.78	
	4.5	0.48	935	7	7	1	2.2	
						7	1.8	
Bradenton, FL, November 1976	0.56	0.06	935	7	7	1	0.24	OF 2288 + 4E 3057
						7	0.28	
	1.1	0.12	935	7	7	1	0.47	
						7	0.36	
	2.2	0.24	935	7	7	1	1.1	
						7	0.78	
	4.5	0.48	935	7	7	1	2.2	
						7	1.8	
Bradenton, FL, June 1977	0.56	0.06	935	6	3-5	1	0.36	OF 2288 + 4E 3057
						3	0.38	
						7	0.28	
	1.1	0.12	935	6	3-5	control	0.09	
						1	0.54	
						3	0.39	
						7	0.36	
Bradenton, FL, November 1977	0.56	0.06	935	6	7	control	0.03	OF 2288 + 4E 3057
						1	0.15	
						3	0.18	
						7	0.19	
	1.1	0.12	935	6	7	1	0.22	
						3	0.26	
						7	0.38	
	2.2	0.24	935	6	7	1	0.57	
						3	0.7	
						7	0.51	
Bradenton, FL, May 1978	0.56	0.06	935	5	7	control	0.03	OF 2288 + 4E 3057
						1	0.24	
						3	0.18	
						7	0.18	
	1.1	0.12	935	5	7	1	0.3	
						3	0.3	
						7	0.26	
	2.2	0.24	935	5	7	1	0.45	
						3	0.45	
						7	0.39	

Table 24. Residues in melons, watermelons and squash from outdoor supervised trials in the USA, in furrow or foliar broadcast SL treatment, (du Pont de Nemours and Co., 1979a; Interregional Research Project No. 4, 1985b). Only partially unreadable hand-written field reports were available.

Location, year, treatment	Application					PHI, days	Residues, mg/kg	Report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
CANTALOUPE								
Blythe, CA, 1976 foliar spray	2.2			5		3	0.03	OF 2288
Yuma, AZ, 1976 foliar spray	2.2			5		3	0.04	OF 2288
						7	0.05	

Location, year, treatment	Application					PHI, days	Residues, mg/kg	Report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Greenfield, CA, 1976 foliar spray	0.56	0.3	187	5	7	1 4 7	0.04 0.05 0.09	OF 2288 + 4E 3057
	1.1	0.6	187	5	7	1 4 7	0.1 0.06 <u>0.16</u>	
	2.2	1.2	187	5	7	1 4 7	0.19 0.12 0.38	
Greenfield, CA, 1976 in furrow (trial not valid, treatment unclear)	0.56	0.3	187	5	7	1 4 7	0.19 0.09 0.06	OF 2288 + 4E 3057 4E 3057
	1.1	0.6	187	5	7	1 4 7	0.62 0.19 0.07	
	2.2	1.2	187	5	7	1 4 7	0.76 0.28 0.15	
Bradenton, FL, 1977 foliar spray	0.56	0.06	935	6		control 3 7	0.05 0.26 0.25	OF 2288 + 4E 3057
	1.1	0.12	935	6		3 7	0.48 0.59	
Bradenton, FL, 1977 foliar spray	0.56	0.06	935	8	7	control 1 3 7	0.04 0.25 0.19 0.14	OF 2288 + 4E 3057
	1.1	0.12	935	8	7	1 3 7	0.5 0.25 <u>0.26</u>	
	2.2	0.24	935	8	7	1 3 7	0.91 0.66 0.45	
Bradenton, FL, 1978 foliar spray	0.56	0.06	935	8	7	control 1 3 7	0.05 0.2 0.23 0.16	OF 2288 + 4E 3057
	1.1	0.12	935	8	7	1 3 7	0.25 <u>0.26</u> 0.25	
	2.2	0.24	935	8	7	1 3 7	0.64 0.58 0.5	
Vincennes, IN, 1978 foliar spray	2.2	0.67	336	5	7 7 17 14	control 1 3 7	0.2 0.23 0.05 0.16	OF 2288 + 4E 3057
	6.7	2	336	5	7 7 17 14	1 3 7	0.44 0.12 0.3	
	13.4	4	336	5	7 7 17 14	1 3 7	1.4 0.44 0.87	
Vincennes, IN, 1978 foliar spray	1.1	0.33	336	5	14	control 1 3 7	0.07 0.15 0.12 <u>0.2</u>	OF 2288 + 4E 3057
	4.5	1.3	336	5	14	1 3 7	0.69 0.52 0.32	

Location, year, treatment	Application					PHI, days	Residues, mg/kg	Report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
	9	2.7	336	5	14	1	0.49	
						3	1.4	
						7	0.41	
HONEYDEW MELON								
Blythe, CA, 1976 foliar spray	2.2			5		1	0.03	OF 2288 + 4E 3057
						3	<0.02	
						5	<0.02	
Poston, AZ, 1976 foliar spray	2.2			5		1	0.02	OF 2288 + 4E 3057
						3	<0.02	
						7	<0.02	
Bradenton, FL, 1976 foliar spray	0.56	0.06	935	9	6-8	1	0.21	OF 2288 + 4E 3057
						7	0.21	
	1.1	0.12	935	9	6-8	1	0.38	
						7	0.32	
	2.2	0.24	935	9	6-8	1	0.68	
						7	0.7	
	4.5	0.48	935	9	6-8	1	1.0	
						7	0.92	
Bradenton, FL, 1977 foliar spray	0.56	0.06	935	6	3-5	control	0.03	OF 2288 + 4E 3057
						1	0.24	
						3	0.28	
						7	0.23	
	1.1	0.12	935	6	3-5	1	0.4	
						3	<u>0.5</u>	
						7	0.44	
Bradenton, FL, 1978 foliar spray	0.56	0.06	935	8	6-8	control	0.05	OF 2288 + 4E 3057
						1	0.2	
						3	0.18	
						7	0.19	
	1.1	0.12	935	8	6-8	1	0.36	
						3	<u>0.39</u>	
						7	0.35	
	2.2	0.24	935	8	6-8	1	0.71	
						3	0.68	
						7	0.67	
WATERMELON								
Yuma, AZ, 1976 foliar spray	2.2			5		1	0.11	OF 2288 + 4E 3057
						3	<0.02	
						7	<0.02	
Bard, CA, 1976 foliar spray	2.2			5		1	0.11	OF 2288 + 4E 3057
						3	<0.02	
						5	<0.02	
San Luis Ray, CA, 1976 foliar spray	0.56	0.07	814	5		control	0.03	OF 2288 + 4E 3057
						1	0.05	
						6	0.03	
	2.2	0.28	814	5		1	0.1	
						6	0.05	
Bradenton, FL, 1978 foliar spray	0.56	0.06	935	8	5-10	control	0.13	OF 2288 + 4E 3057
						1	0.29	
						3	0.27	
						7	0.28	
	1.1	0.12	935	8	5-10	1	<u>0.77</u>	
						3	0.47	
						7	0.56	
	2.2	0.24	935	8	5-10	1	1.2	
						3	0.87	
						7	0.78	
SQUASH, VINING (Winter squash, pumpkin)								
Bard, CA, 1976 foliar spray	2.2			5		1	<0.02	OF 2288 + 4E 3057
						3	<0.02	
						5	<0.02	

Location, year, treatment	Application					PHI, days	Residues, mg/kg	Report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Yuma, AZ, 1976 foliar spray	2.2			5		1 3 7	<0.02 0.06 <0.02	OF 2288 + 4E 3057
SQUASH, SUMMER (Courgette)								
Bard, CA, 1976 foliar spray	2.2			5		3 5	<0.02 <0.02	OF 2288 + 4E 3057
Yuma, AZ, 1976 foliar spray	2.2			5		3 7	<0.02 <0.02	OF 2288 + 4E 3057

Table 25. Residues in peppers from outdoor supervised trials in the USA, foliar broadcast SL treatment. Field reports unavailable for the trials in bold (Report 9F 2266, du Pont de Nemours and Co., 1979b).

Location, year (variety)	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Fort Meyers, FL, 1975 (unknown)	0.56	0.06	935	5	6-7	1 3 5	0.41 0.39 0.47	9F 2266
	1.1	0.12	935	5	6-7	1 3 5	1.1 0.64 0.62	
	2.2	0.24	935	5	6-7	1 3 5	3.0 1.9 1.4	
Bradenton, FL, 1975 (unknown)	0.56	0.06	935	4	6-7	1 3 6	0.1 0.15 0.11	9F 2266
	1.1	0.12	935	4	6-7	1 3 6	0.23 0.14 0.15	
King City, CA, 1975 (unknown)	0.56	0.06	935	4	6-7	1 3 7	0.31 0.23 0.28	9F 2266
Steele, AL, 1976 (unknown)	1.1	0.12	935	5	6-8	1 3	0.69 0.3	9F 2266
	2.2	0.24	935	5	6-8	1 3	1.9 0.46	
Bradenton, FL, 1976 (unknown)	0.56	0.06	935	11	7	1 4 7	0.88 0.7 0.61	9F 2266
	1.1	0.12	935	11	7	1 4 7	2.2 1.5 1.3	
Bradenton, FL, 1976 (unknown)	0.56	0.06	935	6	6-9	1 7	0.4 0.43	9F 2266
	1.1	0.12	935	6	6-9	1 7	0.61 0.73	
	2.2	0.24	935	6	6-9	1 7	1.3 1.2	
Bradenton, FL, 1977 (unknown)	0.56	0.06	935	10	7	3 7	0.51 0.47	9F 2266
	1.1	0.12	935	10	7	3 7	0.95 0.87	
Bradenton, FL, 1978 (unknown)	0.56	0.06	935	18	7	1 3 7	0.51 0.37 0.48	9F 2266
	1.1	0.12	935	18	7	1 3 7	- 1.0 0.84	

Location, year (variety)	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Puerto Rico, 1980 Sweet peppers (Cubanelle)	1.1	0.12	935	8	7	0 0 7 7	0.17 0.28 0.09 <u>0.13</u>	Interregional Research Project No. 4, 1985a, 4E 3048
2 replicate plots	2.2	0.24	935	8	7	0 0 7 7	0.25 1.5 0.74 <0.05	
Bradenton, FL, 1980 Hot peppers (Jalapeno)	0.56[1] 0.56	0.02 0.06	2834 935	1 7	5-8	control 3 7 14	0.06 2.1 1.0 0.82	Interregional Research Project No. 4, 1985a, 4E 3048
	1.1[1] 1.1	0.04 0.12	2834 935	1 7	5-8	3 7 14	4.0 <u>1.8</u> 1.5	
	0.56	0.06	935	8		3 7 14	1.5 1.0 0.99	
	1.1	0.12	935	8		3 7 14	3.0 1.3 <u>1.5</u>	
	2.2	0.24	935	8		3 7 14	5.8 2.6 1.7	
Litchfield, AZ, 1982 Hot peppers (Chili peppers)	0.56	0.06	935	4	12 6 8	1 3	0.32 0.05	Interregional Research Project No. 4, 1985a 4E 3048
	1.1	0.12	935	8	12 6 8	1 3	0.45 0.53	
	2.2	0.24	935	8	12 6 8	1 3	0.93 1.2	
Puerto Rico, 1983 non-bell pepper (Blanco del Pais) 4 replicate plots	0.56	0.06	935	7	7	14 14 14 14	<0.1 <0.1 <0.1 <0.1	Interregional Research Project No. 4, 1985a, 4E 3048 Oxamyl *per se* by HPLC (Thean *et al.*, 1978)
	1.1	0.12	935	7	7	14 14 14 14	0.13 0.22 0.19 0.2	
Puerto Rico, 1985 Sweet peppers (Cubanelle)	0.56			7	6-8	7 7 7 7	0.35 0.38 0.24 0.19	Interregional Research Project No. 4, undated 8E 3604
4 replicate plots	1.1			7	6-8	7 7 7 7	0.4 <u>0.76</u> 0.59 0.59	
Irvine, CA, 1985 Hot peppers (Jalapeno) 4 replicate plots	0.56[1] 1.1	0.12	30 ml/plant 935	1 12	15 7	7 7 7 7	0.66 0.98 0.37 0.93	Biehn, 1988a,b 8E 3604
Bradenton, FL, 1986 Hot peppers (Jalapeno)	0.56[1]	0.015	3742	1		63	0.11 0.27 <0.1 0.16	Biehn, 1988a,b 8E 3604

| Location, year | Application | | | | | PHI, | Residues, | Reference, |
(variety)	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days	days	mg/kg	report no.
4 replicate plots	0.56[1] 1.1	0.015 0.2 0.16 0.12 0.11 0.11 0.09 0.08	3742 561 701 935 1029 1029 1263 1450	1 7	63 7 7 7 7 7 7 7	7 7 7 7	2.9 4.0 <u>4.3</u> 2.2	
Puerto Rico, 1985 Sweet peppers (Aijes dulces)	0.56[1] 1.1			1 9	13 7-14	7 7 7 7	0.69 0.71 0.39 0.55	Interregional Research Project No. 4, undated
4 replicate plots	1.1			7	7-14	7 7 7 7	1.1 <u>0.75</u> 0.57 0.57	8E 3604
Weslaco, TX, 1987 Hot peppers (Jalapena) 4 replicate plots	0.56[1] 1.1	0.6	187	1 7	95 7-14	14 14 14 14	0.28 0.31 0.36 0.32	Biehn, 1988a,b 8E 3604

[1] Transplant water treatment

Table 26. Residues in tomatoes from supervised trials in the USA, foliar broadcast spray treatment (if not otherwise stated), outdoors. Duplicate plots. (Schneiders, 1999, AMR 4347-97).

| Location, year | Application | | | | | | PHI, | Residues, mg/kg |
(variety)	form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days	days	
Germansville, PA, 1997 Better Boy)	SL	1.1	0.56 0.47 0.48 0.48 0.49 0.47 0.49 0.51	199 238 231 232 228 237 227 222	8	6-8	3 3 5 5 7 7 10 10 14 14	0.37 <u>0.48</u> 0.44 0.48 0.19 0.26 0.22 0.17 0.16 0.15
Rose Hill, NC, 1997 (Better Boy)	SL	1.1	0.6	187	8	7	3 3 5 5 7 7 10 10 14 14	<u>0.42</u> 0.33 0.31 0.33 0.22 0.23 0.17 0.24 0.26 0.17
Bradenton, FL, 1997 (Sunpride)	SL	1.1	0.13	870	8	7	3 3 6 6 7 7 10 10 14 14	0.18 0.27 <u>0.27</u> 0.24 0.22 0.25 0.19 0.21 0.17 0.17

Location, year (variety)	Application						PHI, days	Residues, mg/kg
	form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		
Bradenton, FL, 1997 (Sunpride) drip irrigation	SL	1.1			10		2	0.29
		1.1				7	2	0.47
		1.1				7	3	0.5
		1.1				7	3	0.31
		1.1				7	5	0.31
		1.1				7	5	0.33
		1.1				7	7	0.29
		1.1				7	7	0.35
		2.2				2	10	0.38
		2.2				7	10	0.36
							14	0.19
							14	0.22
Duette, FL, 1997 (Agriset)	SL	1.1	0.14	814	8	7	3	0.04
			0.14	832			3	0.04
			0.13	842			5	0.06
			0.14	795			5	0.04
			0.14	823			7	0.06
			0.14	823			7	0.04
			0.14	814			10	0.04
			0.14	823			10	0.03
							14	0.03
							14	0.03
Arcadia, IN, 1997 (TR-12)	SL	1.1	0.6	187	8	7	3	0.99
			0.63	178			3	0.7
			0.67	168			5	0.69
			0.67	168			5	0.82
			0.67	168			7	0.8
			0.67	168			7	0.83
			0.67	168			10	0.54
			0.67	168			10	0.5
							14	0.57
							14	0.5
Madera, CA, 1997 (Champion)	SL	1.1	0.1	1122	8	7	3	0.5
							3	0.46
							5	0.48
							5	0.54
							7	0.39
							7	0.35
							10	0.35
							10	0.46
							14	0.26
							14	0.26
Madera, CA, 1997 (Champion) drip irrigation	SL	1.1			9	7	2	0.56
		1.1					2	0.69
		1.1					3	0.65
		1.1					3	0.68
		1.1					5	0.59
		1.7					5	0.72
		1.7					7	0.69
		2.2					7	0.68
		2.2					10	0.7
							10	0.52
							14	0.55
							14	0.55

Location, year (variety)	Application						PHI, days	Residues, mg/kg
	form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		
Porterville, CA, 1997 (8892) drip irrigation	SL	1.1 1.1 1.1 1.1 1.1 1.7 1.7 2.2 2.2			9	7	2 2 3 3 5 5 7 7 10 10 14 14	0.07 0.04 0.08 0.05 0.12 0.05 0.07 0.07 0.07 0.1 0.05 0.11
San Marcos, CA, 1997 (Big Beef)	SL	1.1	0.25 0.24 0.26 0.24 0.24 0.24 0.24 0.24	449 468 440 468 468 477 477 468	8	5-8	3 3 5 5 7 7 10 10 14 14	0.55 0.36 0.5 0.35 0.34 0.43 0.3 0.29 0.24 0.38
Delevan, CA, 1997 (APT 127)	SL	1.1	1.2 1.1 1.1 1.1 1.1 1.0 1.1 1.1	94 103 103 103 103 112 103 103	8	7-8	3 3 5 5 7 7 10 10 13 13	0.56 0.61 0.36 0.34 0.4 0.21 0.32 0.31 0.29 0.29
Clovis, CA, 1997 (Champion)	SL	1.1	0.1	1122	8	7	3 3 5 5 7 7 10 10 14 14	0.74 0.47 0.6 0.76 0.67 0.54 0.43 0.39 0.44 0.44
San Marcos, CA, 1997 (Hybrid 882)	SL	1.1	0.6 0.63 0.57 0.6 0.6 0.6 0.6 0.6	187 178 196 187 187 187 187 187	8	6-7	3 3 5 5 7 7 10 10 14 14	0.7 0.74 0.57 0.51 0.41 0.37 0.31 0.18 0.15 0.17
Porterville, CA, 1997 (Ace)	SL	1.1	0.4	281	8	7	3 3 5 5 7 7 10 10 14 14	0.83 0.93 0.36 0.49 0.32 0.37 0.26 0.2 0.21 0.3

Location, year (variety)	Application						PHI, days	Residues, mg/kg
	form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		
Fresno, CA, 1997	SL	1.1	0.4	281	8	6-8	3	1.1
			0.4	281			3	0.82
			0.2	561			5	0.67
			0.2	561			5	0.55
			0.16	701			7	0.59
			0.16	701			7	0.36
			0.16	701			10	0.46
			0.16	701			10	0.34
							14	0.33
							14	0.55
Delevan, CA, 1997 (H9175)	SL	1.1	1.2	94	8	7-8	3	0.61
			1.1	103			3	0.39
			1.1	103			5	0.36
			1.1	103			5	0.31
			1.1	103			7	0.21
			1.0	112			7	0.18
			1.1	103			10	0.2
			1.1	103			10	0.34
							14	0.28
							14	0.22
Porterville, CA, 1997 (Ace)	SL	1.1	0.4	281	8	7	3	0.33
							3	0.24
							5	0.19
							5	0.17
							7	0.13
							7	0.11
							10	0.17
							10	0.16
							14	0.19
							14	0.18
Carlsbad, CA, 1997 (Peto Seed)	SL	1.1	0.25	449	8	7-8	3	0.63
			0.25	449			3	0.69
			0.25	449			5	0.52
			0.24	468			5	0.37
			0.24	458			7	0.43
			0.24	468			7	0.58
			0.24	477			10	0.51
			0.24	468			10	0.51
							14	0.41
							14	0.47
Porterville, CA, 1997 (3155)	SL	1.1	0.6	187	8	7	3	0.5
							3	0.4
							5	0.37
							5	0.45
							7	0.1
							7	0.13
							10	0.27
							10	0.24
							14	0.26
							14	0.21
Porterville, CA, 1997 (3155) drip irrigation	SL	1.1			9	7	2	0.48
		1.1					2	0.16
		1.1					3	0.14
		1.1					3	0.1
		1.1					5	0.1
		1.7					5	0.16
		1.7					7	0.41
		2.2					7	0.51
		2.2					10	0.06
							10	0.16
							14	0.07
							14	0.12

Table 27. Residues in carrots from outdoor supervised trials in the USA, one soil treatment pre-plant or pre-emergence followed by directed spray soil treatments.

Location, year (variety)	Application						PHI, days	Residues, mg/kg	Reference, report no.
	form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Haslett, MI, 1988 (Scarlet Nantes)	SL	9.0[1]	4.8	187	4	90	14	0.05	Eble and Powley, 1989, AMR 1027-88
		1.1	0.52	215		14			
		1.1	0.52	215		14			
		1.1	0.48	234					
	SL	4.5[2]	1.0	440	4	90	14	<u>0.07</u>	
		1.1	0.52	215		14			
		1.1	0.52	215		14			
		1.1	0.48	234					
Arkansaw, WI, 1995 (Falcon)	SL	5.6[3]	2.4	234	4	102	14	<u>0.04</u>	McClory, 1999, AMR 3301-95
		1.1	0.6	187		14	14	0.04	
		1.1	0.6	187		14			
		1.1	0.6	187					
	SL	4.5[3]	1.9	234	3	102	28	0.03	
		2.2	1.2	187		14	28	0.03	
		2.2	1.2	187					
Hermiston, OR, 1995 (Bolero F-1)	SL	5.6[3]	2.0	281	4	37	13	<u>0.03</u>	McClory, 1999, AMR 3301-95
		1.1	0.4	281		14	13	0.03	
		1.1	0.4	281		14			
		1.1	0.4	281					
	SL	4.5[3]	1.6	281	3	37	27	<0.02	
		2.2	0.8	281		14	27	0.02	
		2.2	0.8	281					
Donna, TX, 1996 (Imperiator 58 Improved)	SL	5.6[3]	3.0	187	4	130	14	<u>0.02</u>	McClory, 1999, AMR 3301-95
		1.1	0.6	187		14	14	0.02	
		1.1	0.6	187		13			
		1.1	0.6	187					
	SL	4.5[3]	2.4	187	3	130	28	0.02	
		2.2	1.2	187		14	28	<0.02	
		2.2	1.2	187					
Bradenton, FL, 1996 (Asgrow Hicolor-9)	SL	5.6[3]	1.5	374	4	98	12	<u>≤0.02</u>	McClory, 1999, AMR 3301-95
		1.1	0.4	281		13	12	<0.02	
		1.1	0.4	281		14			
		1.1	0.4	281					
	SL	4.5[3]	1.2	374	3	98	26	<0.02	
		2.2	0.8	281		13	26	<0.02	
		2.2	0.8	281					

[1] pre-plant incorporation
[2] in-furrow
[3] pre-emergence

Table 28. Residues in potatoes from supervised trials in the USA. Field reports unavailable for trials in bold (Report 6F 1695, du Pont de Nemours and Co., 1975). All SL formulations.

Location, year (variety), treatment	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Newark, DE, 1973	0.56			6	15, 14, 9, 7, 6	1	<0.02	6F 1695
	1.1			6	15, 14, 9, 7, 6	1	<0.02	
Niles, MI, 1973	1.1	0.2	561	5	7, 9, 18, 8	7	<u>≤0.02</u>	6F 1695
						14	<0.02	
	2.2	0.2	561	5	7, 9, 18, 8	7	0.03	
						14	0.04	
Smyrna, DE, harvest August 1974, foliar spray replicate plot 1	0.56			5	8, 11, 9, 7	7	<0.02	6F 1695
						14	<0.02	
	1.1			5	8, 11, 9, 7	7	0.03	
						14	<u>0.03</u>	
	4.5			5	8, 11, 9, 7	7	0.11	
						14	0.09	

Location, year (variety), treatment	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Smyrna, DE, harvest July 1974	0.56			5	7	control	0.03	6F 1695
						7	<0.02	
						14	0.03	
	1.1			5	7	7	0.03	
						14	0.02	
replicate plot 2	1.1			5	7	7	0.02	
						14	0.02	
	2.2			5	7	7	0.06	
						14	0.05	
Smyrna, DE, 1974	0.5			5	7	7	<0.02	6F 1695
						14	<0.02	
	1			5	7	7	<0.02	
replicate plot 3						14	<0.02	
Smyrna, DE, 1974	0.56			3	8, 11	7	<0.02	6F 1695
						23	<0.02	
replicate plot 4						30	<0.02	
Yelington, FL, 1975	1.1	0.12	935	5	5, 4, 6, 6,	6	<u>≤0.02</u>	6F 1695
Houlton, ME, 1988 (Superior) 1 x pre-plant incorporation, 6 x foliar spray	9	5.6	159	7	88	7	0.03	Eble and Powley, 1990b, AMR 1035-88
	1.1	0.57	196		5			
	1.1	0.60	187		4			
	1.1	0.57	196		6			
	1.1	0.60	187		6			
	1.1	0.60	187		7			
	1.1	0.57	196					
1 x in-furrow, 6 x foliar spray	4.5	2.2	196	7	88	7	<u>≤0.02</u>	
	1.1	0.57	196		5			
	1.1	0.60	187		4			
	1.1	0.57	196		6			
	1.1	0.60	187		6			
	1.1	0.60	187		7			
	1.1	0.57	196					
Arlington, WI, 1988 (Superior) 1 x pre-plant incorporation, 6 x foliar spray	9	3.8	234	7	73	7	0.03	Eble and Powley, 1990b, AMR 1035-88
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168					
1 x in-furrow, 6 x foliar spray	4.5	1.8	243	7	73	7	<u>0.03</u>	
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168		5			
	1.1	0.67	168					
Northwood, ND, 1988 (Russett Burbank) 1 x pre-plant incorporation, 6 x foliar spray	9.0	4.8	187	7	68	7	0.05	Eble and Powley, 1990b, AMR 1035-88
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281					
1 x in-furrow, 6 x foliar spray	4.5	5.3	84	7	68	7	<u>0.05</u>	
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281		5			
	1.1	0.4	281					

| Location, year | Application | | | | | PHI, | Residues, | Reference, |
(variety), treatment	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days	days	mg/kg	report no.
Greeley, CO, 1988	9.0	4.8	187	7	89	7	<0.02	Eble and Powley,
(Monona)	1.1	0.57	196		5			1990b, AMR
	1.1	0.57	196		5			1035-88
1 x pre-plant	1.1	0.57	196		5			
incorporation,	1.1	0.57	196		5			
6 x foliar spray	1.1	0.57	196		5			
	1.1	0.57	196					
1 x in-furrow,	4.5	2.4	187	7	89	7	≤0.02	
6 x foliar spray	1.1	0.57	196		5			
	1.1	0.57	196		5			
	1.1	0.57	196		5			
	1.1	0.57	196		5			
	1.1	0.57	196		5			
	1.1	0.57	196					

Table 29. Residues in celery from supervised trials in the USA, duplicate field samples. Samples were topped (petioles only) according to commercial fresh-market harvest practices, and untopped (entire plant including leaves) according to harvest practices for processing to celery products. All SL formulations. McClory and Summers, 1998, AMR 3437-95.

| Location, year | Application | | | | | PHI, | Residues, mg/kg | |
(variety), treatment	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days	days	topped	untopped
Grant, MI, 1995	4.5	1.2	383	3	48	7	0.54	
(Florida 683 K)	1.1	0.5	224		7	7	0.26	
	1.1	0.52	215			10	0.18	
1x preplant,						10	0.16	
2 x foliar						14	0.15	<0.1
broadcast spray						14	0.12	<0.1
						21		<0.1
						21		<0.1
						28		<0.1
						28		<0.1
2 x banded,	1.1	0.05	2275	6	7	7	0.26	
4 x foliar	1.1	0.05	2275			7	0.16	
broadcast spray	1.1	0.51	220			10	0.11	
	1.1	0.51	220			10	0.12	
	1.1	0.51	220			14	0.18	<0.1
	1.1	0.51	220			14	0.23	<0.1
						21		<0.1
						21		<0.1
						28		<0.1
						28		<0.1
6 x foliar	1.1	0.51	220	6	7	7	0.19	
broadcast spray						7	0.27	
						10	0.13	
						10	0.28	
						14	<0.1	0.11
						14	0.12	<0.1
						21		<0.1
						21		<0.1
						28		<0.1
						28		<0.1

Location, year (variety), treatment	Application					PHI, days	Residues, mg/kg	
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		topped	untopped
Oxnard, CA, 1995 (Conquistador) 1x preplant, 2 x foliar broadcast spray	4.5 1.1 1.1	2.4 0.3 0.3	187 374 374	3	73 7	7 7 11 11 14 14 20 20 27 27	5.0 4.3 5.1 4.2 1.8 2.3	 12 9.9 1.9 1.4 0.95 1.4
2 x banded, 4 x foliar broadcast spray	1.1 1.1 1.1 1.1 1.1 1.1	0.6 0.6 0.3 0.3 0.3 0.3	187 187 374 374 374 374	6	8-14	7 7 11 11 14 14 20 20 27 27	8.8 11 5.2 4.6 1.8 0.96	 13.5 13 1.6 1.5 1.2 1.2
6 x foliar broadcast spray	1.1	0.3	370	6	8-14	7 7 11 11 14 14 20 20 27 27	12 5.2 2.9 5.8 1.8 1.3	 9.5 9.7 1.1 2.1 2.5 2.3
King City, CA, 1995 (Marithon) 1x preplant, 2 x foliar broadcast	4.5 1.1 1.1	1.1 0.29 0.29	421 383 383	3	112 5	7 7 10 10 14 14 21 21 28 28	0.13 0.14 0.12 0.11 0.2 0.19	 1.0 0.78 0.38 0.55 0.41 0.38
2 x banded, 4 x foliar broadcast spray	2.8 2.8 1.1 1.1 1.1 1.1	0.64 0.64 0.29 0.29 0.29 0.29	440 440 383 383 383 383	6	5	7 7 10 10 14 14 21 21 28 28	0.15 0.16 0.21 0.2 0.11 0.14	 0.41 0.67 0.31 0.3 0.21 0.21
6 x foliar broadcast spray	1.1	0.29	383	6	5	7 7 10 10 14 14 21 21 28 28	0.24 0.18 0.32 0.31 0.1 0.12	 0.41 0.28 1.7 1.1 1.1 0.42

Location, year (variety), treatment	Application					PHI, days	Residues, mg/kg	
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		topped	untopped
Irvine, CA, 1995 (5274) 1x preplant, 2 x foliar broadcast spray	4.5 1.1 1.1	2.4 0.29 0.31	187 383 365	3	66 10	7 7 10 10 14 14 22 22 26 26	1.1 1.6 2.3 1.8 0.72 0.73	 0.79 1.2 0.66 0.84 0.47 0.82
2 x banded, 4 x foliar broadcast spray	1.1 1.1 1.1 1.1 1.1 1.1	0.59 0.59 0.3 0.3 0.3 0.3	190 190 370 370 370 370	6	6-10	7 7 10 10 14 14 22 22 26 26	3.4 2.3 2.2 1.7 1.0 2.0	 1.8 2.6 1.3 1.6 1.1 1.2
6 x foliar broadcast spray	1.1	0.3	370	6	6-10	7 7 10 10	8.2 6.0 7.9 3.6	
Watsonville, CA, 1996 (Summit) 1x preplant, 2 x foliar broadcast spray	4.5 1.1 1.1	2.4 0.24 0.24	187 468 468	3	100 7	7 7 10 10 14 14 21 21 28 28	0.97 1.1 0.84 0.63 0.49 0.51	 0.57 0.42 0.31 0.45 0.26 0.33
2 x banded, 4 x foliar broadcast spray	1.1 1.1 1.1 1.1 1.1 1.1	0.57 0.57 0.24 0.24 0.24 0.24	195 195 468 468 468 468	6	7	7 7 10 10 14 14 21 21 28 28	1.6 1.4 0.52 0.69 0.93 0.73	 0.81 0.86 0.71 0.66 0.49 0.54
6 x foliar broadcast	1.1	0.24	468	6	7	7 7 10 10 14 14 21 21 28 28	1.2 1.0 0.81 0.77 0.64 0.56	 0.68 0.7 0.57 0.61 0.56 0.49
Bradenton, FL, 1996 (Duda 1622) 1x preplant, 2 x foliar broadcast spray	4.5 1.1 1.1	1.6 0.31 0.31	271 365 365	3	74 5	7 7 10 10 15 15 21 21 28 28	0.35 0.45 0.41 0.48 0.28 0.38	 1.4 1.0 0.59 0.49 <0.1 0.12

Location, year (variety), treatment	Application					PHI, days	Residues, mg/kg	
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		topped	untopped
2 x banded, 4 x foliar broadcast spray	1.1	0.05	2217	6	5-8	7	0.41	
	1.1	0.05	2217			7	0.34	
	1.1	0.3	370			10	0.66	
	1.1	0.3	370			10	0.82	
	1.1	0.3	370			15	0.38	0.27
	1.1	0.3	370			15	0.46	0.36
						21		0.46
						21		0.51
						28		0.13
						28		0.11
6 x foliar broadcast spray	1.1	0.3	370	6	5-8	7	0.39	
						7	0.47	
						10	0.58	
						10	0.9	
						15	1.4	0.43
						15	0.97	0.39
						21		0.51
						21		0.51
						28		0.11
						28		0.17
King City, CA, 1996 (Conquistador) 2 x banded, 4 x foliar broadcast spray	1.1	0.2	552	6	7	7	0.12	
	1.1	0.2	552			7	0.5	
	1.1	0.4	281					
	1.1	0.4	281					
	1.1	0.4	281					
	1.1	0.4	281					
Belle Glade, FL, 1996 (June Belle) 1x preplant, 2 x foliar broadcast spray	4.5	1.2	374	3	62	7	1.4	
	1.1	0.27	412		7	7	1.0	
	1.1	0.28	402			10	0.59	
						10	0.49	
						14	<0.1	<0.1
						14	0.12	<0.1
						21		0.16
						21		0.14
						28		<0.1
						28		<0.1
2 x banded, 4 x foliar broadcast spray	1.1	0.05	2245	6	7	7	0.27	
	1.1	0.05	2245			7	0.36	
	1.1	0.28	407			10	0.46	
	1.1	0.28	407			10	0.51	
	1.1	0.28	407			14	0.13	<0.1
	1.1	0.28	407			14	0.11	<0.1
						21		<0.1
						21		<0.1
						28		<0.1
						28		<0.1
6 x foliar broadcast spray	1.1	0.28	407	6	7	7	0.43	
						7	0.39	
						10	0.51	
						10	0.51	
						14	0.11	<0.1
						14	0.17	<0.1
						21		<0.1
						21		<0.1
						28		<0.1
						28		<0.1

Location, year (variety), treatment	Application					PHI, days	Residues, mg/kg	
	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days		topped	untopped
Santa Maria, CA, 1996 (Utah) 1x preplant, 2 x foliar broadcast spray	4.5 1.1 1.1	2.4 0.24 0.25	187 468 440	3	79 7	7 7 10 10 14 14 21 21 28 28	1.5 1.6 0.31 0.35 0.22 0.2	 0.53 0.93 0.3 0.48 0.35 0.5
2 x banded, 4 x foliar broadcast spray	1.1 1.1 1.1 1.1 1.1 1.1	0.6 0.6 0.24 0.24 0.24 0.24	187 187 460 460 460 460	6	7-9	7 7 10 10 14 14 21 21 28 28	3.2 3.6 0.28 1.1 0.57 0.27	 1.2 0.91 0.72 0.95 0.58 0.45
6 x foliar broadcast spray	1.1	0.24	460	6	7-9	7 7 10 10 14 14 21 21 28 28	2.6 2.2 0.6 0.59 0.41 0.5	 1.0 1.7 0.72 0.71 0.73 0.66

Table 30. Residues in cotton seed from supervised trials in the USA, foliar broadcast spray treatment. Field reports not available for trials in bold (du Pont de Nemours and Co., 1976a). All SL formulations.

Location, year (variety)	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval, days			
Peoria, AZ, 1974	0.56			3	7 7	7 15	0.04 <0.02	7F 1907
	1.1			3	7 7	7 15	0.14 ≤0.02	
	2.2			3	7 7	7 15	0.23 <0.02	
	4.5			3	7 7	7 15	0.64 0.02	
Beardsly, AZ, 1974	0.56			1		14	0.02	7F 1907
	1.1			1		14	0.04	
Univ. of Ariz., AZ, 1975	1.1			1		1 2 3 4	25 2.6 0.85 0.75	7F 1907
Tempe, AZ, 1975	1.1			5	5 20 10 35	7 14	0.05 ≤0.02	7F 1907
	2.2			5	5 20 10 35	7 14	0.05 0.02	

Location, year (variety)	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval, days			
Scottsdale, AZ, 1975 Field 33 Replicate plot 1	0.56			5	6 15 16 37	7 14	0.07 0.03	7F 1907
	1.1			5	6 15 16 37	7 14	0.15 <u>0.04</u>	
	2.2			5	6 15 16 37	7 14	0.19 0.12	
	4.5			5	6 15 16 37	7 14	0.3 0.38	
Scottsdale, AZ, 1975 Field 26 Replicate plot 2	0.56			5	6 8 21 10	7 14	0.04 0.02	7F 1907
	1.1			5	6 8 21 10	7 14	0.05 <u>0.08</u>	
	2.2			5	6 8 21 10	7 14	0.14 0.12	
Scottsdale, AZ, 1975 Field 119 Replicate plot 3	0.56			5	7 8 21 36	7 14	0.03 <0.02	7F 1907
	1.1			5	7 8 21 36	7 14	0.1 <u>0.07</u>	
	2.2			5	7 8 21 36	7 14	0.19 0.16	
Scottsdale, AZ, 1975 Field 117 Replicate plot 4	0.56			5	15 5 16 37	7 14	0.04 0.02	7F 1907
	1.1			5	15 5 16 37	7 14	0.08 <u>0.05</u>	
	2.2			5	15 5 16 37	7 14	0.25 0.16	
	4.5			5	15 5 16 37	7 14	0.44 0.26	
Arlington, AZ, 1975 Field 1 Replicate plot 1	0.37			3	7 10	7 14	0.04 0.06	7F 1907
	0.75			3	7 10	7 14	0.16 0.11	
Arlington, AZ, 1975 Field 2 Replicate plot 2	0.37			3	10 10	7 14	0.08 0.12	7F 1907
	0.75			3	10 10	7 14	0.09 0.08	

Location, year (variety)	Application					PHI, days	Residues, mg/kg	Reference, report no.
	kg ai/ha	kg ai/hl	Water, l/ha	No.	Interval, days			
Arlington, AZ, 1975 Field 3 Replicate plot 3	0.37			3	7 10	7 14	0.06 0.07	7F 1907
	0.75			3	7 10	7 14	0.16 0.14	
Calipatria, CA, 1975 Gate E.48 Replicate plot 1	0.56			5	13 16 9 12	8 15	<0.02 <0.02	7F 1907
	1.1			5	13 16 9 12	8 15	0.02 <u>0.03</u>	
	2.2			5	13 16 9 12	8 15	0.03 0.02	
	4.5			5	13 16 9 12	8 15	0.23 0.1	
Calipatria, CA, 1975 Gate D. 28 Replicate plot 2	0.56			5	13 7 9 11	8 15	0.02 <0.02	7F 1907
	1.1			5	13 7 9 11	8 15	0.03 <u>0.02</u>	
	2.2			5	13 7 9 11	8 15	0.07 0.05	
Holtsville, AZ, 1975 Replicate plot 1	0.37			4	12 18 7	7 12	0.04 <0.02	7F 1907
	0.75			4	12 18 7	7 12	0.02 <0.02	
Holtsville, AZ, 1975 Replicate plot 2	0.37			4	12 18 7	7 12	0.03 0.02	7F 1907
	0.75			4	12 18 7	7 12	0.05 0.04	
Yuma, AZ, 1988 (Delta Pine 61)	1.1	4.0	28✈	4	7 7 8	21	<0.02	Eble and Powley, 1990c, AMR 1147-88
Helm, CA, 1988 (SJ-2)	1.1	2.4	47✈	4	7 7 7	21	<0.02	Eble and Powley, 1990c, AMR 1147-88

✈ Aerial application

Table 31. Residues in peanuts from supervised trials in the USA. Field reports unavailable for bold marked trials (E.I. du Pont de Nemours and Co., 1979c).

Location, year (variety) treatm	Application						PHI, days	Residues, mg/kg	Reference, report no.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
Yokum, TX, 1970 no information on treatment	10G	3.4			1		127 127 127	<0.02 kernel <0.02 hull 0.38 hay	3G 1316
Raleigh, NC, 1971	10G	2.8			1		184 184 184	<0.02 kernel <0.02 hull <0.02 hay	3G 1316
no information on treatment	SL	2.8			1		184 184	<0.02 kernel <0.02 hay	
	10G	4.5			1		184 184 184	<0.02 kernel <0.02 hull <0.02 hay	
Yokum, TX, 1972 no information on treatment	10G SL SL	3.4 1.7 1.7			3	42	90 90 90 122 122	0.03 kernel <0.02 hull 0.07 hay <0.02 kernel 0.59 hay	3G 1316
	10G	3.4			1		122 122 122	<0.02 kernel <0.02 hull <0.02 hay	
Yokum, TX, 1972 no information on treatment	10G SL	1.7 1.7			2	42	90 90 122 122 122	<0.02 kernel 0.26 hay <0.02 kernel <0.02 hull 0.04 hay	3G 1316
	10G SL	3.4 3.4			2	42	90 90 90 122 122	<0.02 kernel <0.02 hull 1.0 hay <0.02 kernel 0.04 hay	
Emporia, VA, 1973 1 at plant, 5 foliar sprays	10G SL SL SL SL SL	4.4 1.1 1.1 1.1 0.06 0.06			6	30 21 24 20 28	12 12 12	<0.02 kernel <0.02 hull 0.57 hay	3G 1316
Albany, GA, 1973 foliar spray	SL	0.56			2	14	27 27 27	<0.02 kernel <0.02 hull 0.04 hay	3G 1316
Albany, GA, 1973 foliar spray	SL	0.56			3	14 19	8 8 8	<0.02 kernel 0.05 hull 0.06 hay	3G 1316
Atwood, OK, 1975 foliar spray	10G	3.4			1		157 157	0.03 kernel <0.02 hay	3G 1316
	10G	5			1		157 157 157	<0.02 kernel <0.02 hull <0.02 hay	
1 at plant, 2 foliar sprays Replicate plot 1	10G SL	3.4 1.1 1.1			3	34 32	61 61 61	0.03 kernel <0.02 hull 0.04 hay	
	10G SL	5 1.1 1.1			3	34 32	61 61	<0.02 kernel <0.02 hay	
Atwood, OK, 1975	10G	3.4			1	38 32	157 157	0.03 kernel 0.04 hay	3G 1316
1 at plant, 2 foliar sprays Replicate plot 2	10G SL	3.4 1.1 1.1			3	38	61 61	<0.02 kernel 0.03 hay	

Location, year (variety) treatm	Application						PHI, days	Residues, mg/kg	Reference, report no.
	Form	kg ai/ha	kg ai/hl	water, l/ha	no.	interval, days			
	10G	5			3	38	61	<0.02 kernel	
	SL	1.1				32	61	<0.02 hull	
		1.1					61	0.06 hay	
Eakly, OK, 1976	10G	3.4			3	21	75	<0.02 kernel	3G 1316
	SL	1.1				31	75	<0.02 hull	
	SL	1.1					75	<0.02 hay	
1 at plant, 2 foliar sprays	10G	5			3	21	75	<0.02 kernel	
	SL	1.1				31	75	<0.02 hull	
	SL	1.1					75	0.07 hay	
Fort Valley, GA, 1988 (GK-7)	SL	3.4	2.4	140	3	30	78	<0.02 kernel	Eble and Powley, 1990a, AMR 1033-88
		1.1	0.6	187		20	72	<0.02 vine	
		1.1	0.6	187			78	<0.02 hay	
1 at plant in-furrow, 2 foliar sprays	SL	6.7	4.8	140	3	30	78	<0.02 kernel	
		2.2	1.2	187		20	72	0.04 vine	
		2.2	1.2	187			78	0.03 hay	
Bradenton, FL, 1988 (GK-7)	SL	3.4	1.2	281	3	21	82	<0.02 kernel	Eble and Powley, 1990a, AMR 1033-88
		1.1	0.71	159		21	77	0.12 vine	
		1.1	0.34	327			82	0.03 hay	
1 pre-plant incorporated, 2 foliar sprays	SL	6.7	2.4	281	3	21	82	<0.02 kernel	
		2.2	1.4	159		21	77	0.09 vine	
		2.2	0.68	327			82	0.03 hay	
Fayetteville, NC, 1988 (Florigiant)	SL	3.4	0.84	402	3	31	106	<0.02 kernel	Eble and Powley, 1990a, AMR 1033-88
		1.1	0.48	234		22	98	0.07 vine	
		1.1	0.48	234			106	0.05 hay	
1 pre-plant incorporated, 2 foliar sprays	SL	6.7	1.7	402	3	31	106	<0.02 kernel	
		2.2	0.96	234		22	98	0.03 vine	
		2.2	0.96	234			106	<0.02 hay	
1 pre-plant incorporated, 4 foliar sprays	SL	3.4	0.84	402	5	31	58	<0.02 kernel	
		2.2	0.96	234		22			
		2.2	0.96	234		23			
		2.2	0.96	234		19			
		2.2	0.96	234					
Donna, TX, 1988 (Star D-85-31101)	SL	3.4	1.9	178	3	28	118	<0.02 kernel	Eble and Powley, 1990a, AMR 1033-88
		1.1	0.6	187		24	115	<0.02 vine	
		1.1	0.6	187			118	<0.02 hay	
1 at plant in-furrow, 2 foliar sprays	SL	6.7	4.8	140	3	28	118	<0.02 kernel	
		2.2	1.2	187		24	115	<0.02 vine	
		2.2	1.2	187			118	<0.02 hay	

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Oxamyl residues in tomatoes decreased during storage at 15°C in air and modified atmospheres (McGarvey *et al.*, 1994) decreasing in the latter slightly faster. The results are given in Table 32.

Table 32. Oxamyl residues in tomatoes after storage at 15°C (McGarvey *et al.*, 1994).

Atmosphere	Oxamyl residues, mg/kg, duplicate experiments			
	Week 0	Week 1	Week 2	Week 3
Air (21.0% O_2, 79.0% N_2)	0.32	0.35	0.18	0.24
	0.16	0.12	0.07	0.04
1.5% O_2, 98.5% N_2	0.32	0.29	0.19	0.18
	0.16	0.10	0.06	0.03
1.5% O_2, 4.0% CO_2, 79.0% N_2	0.32	0.29	0.15	0.09
	0.16	0.08	0.04	0.02

In processing

Nature of residues

The hydrolysis of [^{14}C]oxamyl was studied in sterile buffered solutions at pH 4, 5, and 6 to determine the effects of processing (Lee, 2001). Oxamyl oxime was the only degradation product. The results are shown in Table 33.

Table 33. Degradation of oxamyl in buffer solutions at different temperatures and incubation times (Lee, 2001). Control experiments without heating showed 100% recovery of oxamyl.

Treatment				Oxamyl	Oxamyl oxime
90°C,	20 min,	pH 4	(Pasteurisation)	100	0
100°C,	60 min,	pH 5	(Baking or boiling)	58	41
120°C,	20 min,	pH 6	(Sterilization)	0	100

Studies on crops

Oxamyl was determined in processed products of 6 distinct raw agricultural commodities (Table 34) using the GLC method of Holt and Pease (1976) and reporting the combined residues of oxamyl and the oxime in oxamyl equivalents. The processing procedures are shown in Figures 5 and 6 for oranges and tomatoes.

In several supervised residue trials on whole pineapples the bran was analysed (see Table 22). Table 35 shows the results of trials with RAC residues >0.2 mg/kg which were selected for estimation of a median processing factor.

Table 34. Oxamyl processing studies on crops.

Raw Agricultural Commodity (RAC) Fractions	Oxamyl concentration, mg/kg		Processing factor	Reference
	Treated samples	Control samples		
Oranges (RAC)	0.55	<0.02		Lin and Hay, 1990b
Dry pomace	<0.02	<0.02	<0.036	AMR 1029-88
Cold pressed oil	<0.02	<0.02	<0.036	
Molasses	1.9	<0.02	3.45	
Wet finisher waste	<0.02	<0.02	<0.036	
Pineapple (RAC)	0.1	<0.02		Lin and Hay, 1990a
Wet skins	0.17	<0.02	1.7	AMR 1390-89
Juice	0.12	<0.02	1.2	
Tomato – frozen in the field	3.1	<0.02		Lin and Hay, 1990c
Tomato – shipped for processing (RAC)				
Washed fruit	0.2	<0.02	0.13	

Raw Agricultural Commodity (RAC)	Oxamyl concentration, mg/kg		Processing factor	Reference
Fractions	Treated samples	Control samples		
Whole canned	0.11	<0.02	0.073	
Hot break	0.24	<0.02	0.16	
Wet pomace	0.06	<0.02	0.04	
Juice	0.18	<0.02	0.12	
Paste	0.54	<0.02	0.36	
Ketchup	0.36	<0.02	0.24	
Purée	0.24	<0.02	0.16	
Dry pomace	0.02	<0.02	0.013	
Potato (RAC)	0.02	<0.02		Eble and Powley, 1990b
Washed potato	<0.02	<0.02	<1	
Peels	0.022	<0.02	1.1	
Peeled rinsed potato	<0.02	<0.02	<1	AMR 1035-88 and Suppl. No. 1
Fresh oil – French fries	<0.02	<0.02	<1	
Frying oil – French fries	<0.02	<0.02	<1	
French fries	0.026	<0.02	1.3	
Fresh oil – chips	<0.02	<0.02	<1	
Frying oil – chips	<0.02	<0.02	<1	
Chips	<0.02	<0.02	<1	
Granules	<0.02	<0.02	<1	
Peanut kernel (RAC)	0.12	<0.02		Lin and Tomic, 1991n
Hulls	0.092	<0.02	0.77	
Meal	<0.02	<0.02	<0.17	AMR 1697-90
Refined Oil	<0.02	<0.02	<0.17	
Crude Oil	<0.02	<0.02	<0.17	
Soapstock	<0.02	<0.02	<0.17	
Peanut kernel	Residues in all treated and control samples were <0.02 mg/kg; processing factors could not be calculated.			Eble and Powley, 1990a
Cotton seed				Powley, 1988
Cotton seed (RAC)	2.4	<0.02		Lin and Hay, 1990e
Delinted cotton seed	0.69	<0.02	0.288	
Hulls	1.0	<0.02	0.417	AMR 1150-88
Meal	0.03	<0.02	0.0125	
Soapstock	<0.02	<0.02	<0.008	
Crude oil	<0.02	<0.02	<0.008	
Refined oil	<0.02	<0.02	<0.008	

Table 35. Residues in pineapple whole fruits and bran from supervised trials in the USA, spray treatment (du Pont de Nemours and Co., 1977, Report 7F 2000).

Location, year	Application				PHI, days	Residues, mg/kg		Processing factor
	form	kg ai/ha	no.	interval		whole fruit	bran	
Lanai, HI, 1975 Plot 1 (RAB-7-75)	SL	1.1	6	1 month	27	0.43	1.7	3.95
		2.2	6	1 month	13	0.73	3.5	4.79
					27	0.38	1.1	2.89
		4.5	6	1 month	13	2.5	2.1	0.84
					27	0.91	4.5	4.94
		1.1	5	1 month	13	0.25	0.57	2.28
		2.2	5	1 month	13	0.38	0.71	0.54
		4.5	5	1 month	13	1.6	2.4	1.5
					27	0.48	4.4	9.2
Lanai, HI, 1975 Plot 2 (RAB-8-75)	SL	1.1	6	1 month	13	0.22	0.29	1.3
		2.2	6	1 month	13	0.27	0.54	2
					27	0.39	3.2	8.21
		4.5	6	1 month	13	1.2	1.9	1.58
					27	0.79	5.2	6.58
		1.1	5	1 month	13	0.46	0.33	0.72

Location, year	Application				PHI, days	Residues, mg/kg		Processing factor
	form	kg ai/ha	no.	interval		whole fruit	bran	
		2.2	5	1 month	13	1.1	1.1	1
					27	0.59	1.4	2.37
		4.5	5	1 month	13	1.4	3.0	2.14
					27	0.57	2.5	4.39
Kunia, HI, 1975 Plot 1 (RAB-74)	SL	4.5	1		14	0.24	0.53	2.21
			3	1 month	14	0.38	0.51	1.34
			4		14	0.29	0.44	1.52
			5		14	0.21	0.22	1.05
		9	1		14	0.55	1.6	2.91
			2	1 month	14	0.55	0.8	1.45
			3		14	0.29	1.2	4.14
			4		14	0.68	0.72	1.06
			5		14	1.2	1.2	1
Kunia, HI, 1975 Plot 2 (RAB-74)	SL	4.5	1		14	0.33	0.27	0.82
			4	1 month	14	0.21	0.68	3.24
			6		14	0.34	0.26	0.76
		9	1		14	0.82	1.2	1.46
			4	1 month	14	0.48	1.2	2.5
			5		14	0.74	1.7	2.3
			6		14	0.47	1.1	2.34

Fractionated study product/sample list and a brief description of samples in Figure 5:

(1) UNWASHED FRUIT— whole fruit as delivered to pilot plant
(2) WASHED FRUIT— whole fruit washed in the Pennwalt packing house
(3) PRE-WASH WATER — initial water rinsed off the unwashed fruit
(4) AFTER-WASH WATER — water rinse after packing house soap applied
(5) FRESH JUICE — single-strength juice from the washed fruit extracted on an F.M.C. 291B extractor
(6) PEEL FRITS —1 mm pieces of peel recovered during oil extraction
(7) FINISHER PULP (WET FINISHER) — juice sacs removed from juice stream by an FMC model 35 juice finisher
(8) OIL EMULSION WATER — aqueous phase of liquid stream from oil extraction process
(9) CONCENTRATED OIL EMULSION— emulsion of liquid stream from oil extraction process after emulsion water drained off
(10) COLD PRESSED OIL— citrus oil produced by centrifuging and filtering concentrated oil emulsion
(11) PEEL— solid fraction from juice extraction containing peel, membrane, rag, and seeds
(12) CHOPPED PEEL—peel uniformly chopped by Fitzpatrick comminuter
(13) DRIED PEEL (DRY POMACE)— chopped peel limed, reacted, pressed, and put through feed mill drier to produce a product of 8-10% moisture
(14) PRESS LIQUOR —pressed from the limed reaction chopped peel
(15) MOLASSES — press liquor concentrated to approximately 50°Brix

Figure 5. Orange processing (Lin and Hay, 1990b).

After -wash water (4)

Unwashed fruit (1) → Fruit washer → Washed fruit (2) → FMC Extractor

Pre-wash water (3)

Fresh juice (5) ← Finisher ←

Finisher pulp (7)

Peel hopper ← Peel ← Peel frits (6) ← Finisher

Cold pressed oil ← Centrifuge ← concentrated oil emulsion (9) ← Oil em.

Water   Oil emulsion water (8)

Shredder → Chopped peel (12)

→ Lime slurry

Reaction conveyer → Press → Press liquor (14)

Press cake   Vacuum still

Dried peel (13) ← Drier   Molasses (15)

Figure 6. Tomato processing (Lin and Hay, 1990c).

```
                    ┌──────────────────┐
                    │  Raw unwashed    │
                    │    tomatoes      │
                    └──────────────────┘
                             │
                             ▼
┌──────────────┐    ┌──────────────────┐    ┌──────────────────┐
│ Fresh water  │───▶│    Washing       │───▶│   Wash water     │
└──────────────┘    └──────────────────┘    └──────────────────┘
                             │
                             ▼
                    ┌──────────────────┐    ┌──────────────────┐
                    │ Washed tomatoes  │───▶│    Peeling       │
                    └──────────────────┘    └──────────────────┘
                             │                        │
                             ▼                        ▼
                    ┌──────────────────┐    ┌──────────────────┐
                    │    Crushing      │    │  Canned whole    │
                    └──────────────────┘    │    tomatoes      │
                             │   Peels       └──────────────────┘
                             ▼
                    ┌──────────────────┐
                    │    Hot break     │
                    └──────────────────┘
                             │
┌──────────────┐    ┌──────────────────┐
│  Wet pomace  │◀───│    Finisher      │
└──────────────┘    └──────────────────┘
       │                     │
       ▼                     ▼
┌──────────────┐    ┌──────────────────┐    ┌──────────────────┐
│   Drying     │    │     Juice        │───▶│  Canned juice    │
└──────────────┘    └──────────────────┘    │    (fresh)       │
       │                     │               └──────────────────┘
       ▼                     ▼
┌──────────────┐    ┌──────────────────┐
│  Dry pomace  │    │  Concentration   │
└──────────────┘    └──────────────────┘
                      │              │
                      ▼              ▼
              ┌──────────┐    ┌──────────┐
              │  Purée   │    │  Paste   │
              └──────────┘    └──────────┘
                              │          │
                              ▼          ▼
                      ┌──────────────┐  ┌──────────┐
                      │ Canned juice │  │ Ketchup  │
                      │   (paste)    │  └──────────┘
                      └──────────────┘
```
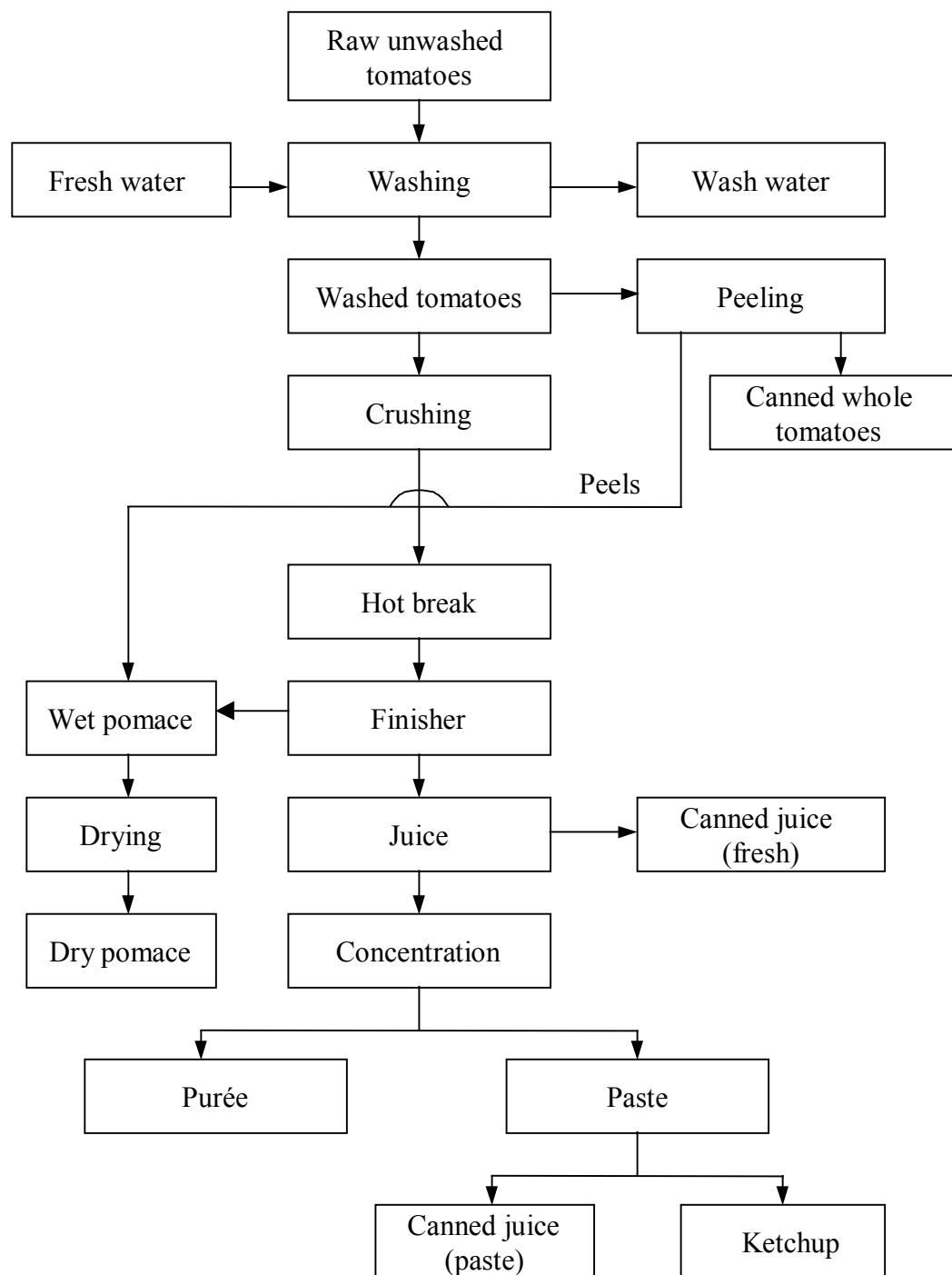
Residues in the edible portion of food commodities

Table 36 shows oxamyl residues in the edible pulp, and whole fruit of bananas (see also Table 21).

Table 36. Oxamyl residues in edible pulp and inedible peel of bananas from trials in Australia (du Pont de Nemours and Co., 1994).

Location, year	Appl., kg ai/hl/plant	Fraction	Oxamyl residues, mg/kg (days after last application)						Report
			0	1-2	4	7	14	28	
Caboolture, Queensland, 1990	0.18	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	Analchem 2318/90/5
		Peel	<0.01	0.03	<0.01	<0.01	<0.01	<0.01	
		Whole fruit	<0.01	0.02	<0.01	<0.01	<0.01	<0.01	
Caboolture, Queensland, 1990	0.24	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	Analchem 2318/90/5
		Peel	<0.01	0.02	<0.01	0.01	0.01	<0.01	
		Whole fruit	<0.01	0.01	<0.01	0.01	0.01	<0.01	
Caboolture, Queensland, 1990	0.48	**Pulp**	**0.01**	**0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	Analchem 2318/90/5
		Peel	0.03	0.20	0.03	0.03	0.05	0.02	
		Whole fruit	0.02	0.08	0.02	0.02	0.03	0.01	
Murwillumbah, New South Wales, 1990	0.18	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	Analchem 2364/90/5
		Peel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
		Whole fruit	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Murwillumbah, New South Wales, 1990	0.24	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	Analchem 2364/90/5
		Peel	0.02	<0.01	<0.01	<0.01	0.03	<0.01	
		Whole fruit	0.01	<0.01	<0.01	<0.01	0.02	<0.01	
Murwillumbah, New South Wales, 1990	0.48	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	Analchem 2364/90/5
		Peel	0.1	<0.01	0.02	<0.01	0.05	<0.01	
		Whole fruit	0.04	<0.01	0.01	<0.01	0.03	<0.01	
Innisfail, Queensland, 1991	0.3	**Pulp**	**<0.01**	**<0.01**	--	**<0.01**	**<0.01**	**<0.01**	Analchem 92/0253
		Peel	<0.01	<0.01	--	<0.01	0.01	0.01	
		Whole fruit	<0.01	<0.01	--	<0.01	<0.01	<0.01	
Innisfail, Queensland, 1991	0.6	**Pulp**	**<0.01**	**<0.01**	--	**<0.01**	**<0.01**	**<0.01**	Analchem 92/0253
		Peel	<0.01	0.08	--	<0.01	<0.01	0.05	
		Whole fruit	<0.01	0.03	--	<0.01	<0.01	0.02	
Wamuran, Queensland, 1993	0.3	**Pulp**	**<0.01**	--	--	--	--	--	Analchem 93/2788
		Peel	<0.01	0.03	--	<0.01	--	--	
		Whole fruit	<0.01	--	--	--	--	--	
Wamuran, Queensland, 1993	0.6	**Pulp**	**<0.01**	--	--	--	--	--	Analchem 93/2788
		Peel	<0.01	0.03	--	<0.01	--	--	
		Whole fruit	<0.01	--	--	--	--	--	
Wamuran, Queensland, 1994	0.3	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	--	Analchem 94/2361
		Peel	<0.01	<0.01	<0.01	<0.01	<0.01	--	
		Whole fruit	<0.01	<0.01	<0.01	<0.01	<0.01	--	
Wamuran, Queensland, 1994	0.6	**Pulp**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	**<0.01**	--	Analchem 94/2361
		Peel	<0.01	<0.01	<0.01	<0.01	<0.01	--	
		Whole fruit	<0.01	<0.01	<0.01	<0.01	<0.01	--	

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Oxamyl is not used for direct animal treatment.

Farm animal feeding studies

Oxamyl was rapidly degraded and excreted in poultry and goats. The five metabolites IN-A2213, IN-N0079, IN-D2708, IN-T2921 and IN-L2953 have been identified from metabolism studies on plants and/or animals. Toxicological studies indicate that the parent compound is the only relevant toxic component of the residue. Transfer coefficients of oxamyl residues from animal feed to animal products are not applicable since oxamyl was not found in any tissue, milk or egg samples in the metabolism studies. Most of the ^{14}C was incorporated into natural components. The conclusion that oxamyl is metabolized and not found in any primary food commodity of animal origin is further supported by two feeding studies on cows and laying hens.

Dairy cattle

Du Pont Report no.:	O/ME 25 (1973)	
Compound dosed:	Oxamyl	
Nature of dose:	Oxamyl admixed with grain concentrate portion of the ration using Vydate® L containing 27% oxamyl.	
Number/type per feeding group:	Lactating Guernsey cows; 2 per dietary level	
Dose period:	30 days	
Depuration period:	7 days	
Feeding levels (mg oxamyl/kg in the feed/dry weight basis):	0, 2, 10, and 20	
Ration (kg feed per cow per day):	4.54 kg grain concentrate/cow/day 9.08 kg dried alfalfa hay/cow/day	
Dose per day:	Feeding level (mg oxamyl/kg feed)	Dose (mg compound/animal/day)
	0	0
	2	27
	10	136
	20	272

This study determined levels of oxamyl in the milk and tissues of lactating cows dosed with oxamyl daily for 30 days using the GLC method of Holt and Pease (1976), which hydrolyzes oxamyl to its oxime metabolite and reports the combined residues in oxamyl equivalents. Half of the cows were killed on day 31, and the remaining were fed unfortified rations a further 7 days before being killed (day 38). The combined residue was undetectable in all the samples from the treated cows (Table 37). The LOQ was 0.02 mg/kg.

Table 37. Oxamyl residues in the milk and tissues of dairy cattle (Du Pont de Nemours and Co., 1973).

Sample	Day sample collected	Oxamyl residues, mg/kg, at dosing level (mg/kg feed)			
		0	2	10	20
Whole milk (milk from cows at same dosing level composited, am and pm milkings)	0 (pre-treatment)	<0.02	<0.02	<0.02	<0.02
	1	<0.02	<0.02	<0.02	<0.02
	3	<0.02	<0.02	<0.02	<0.02
	5	<0.02	<0.02	<0.02	<0.02
	7	<0.02	<0.02	<0.02	<0.02
	10	<0.02	<0.02	<0.02	<0.02
	15	<0.02	<0.02	<0.02	<0.02
	23	<0.02	<0.02	<0.02	<0.02
	25	<0.02	<0.02	<0.02	<0.02
	27	<0.02	<0.02	<0.02	<0.02
	30	<0.02	<0.02	<0.02	<0.02
	31 (am only)	<0.02	<0.02	<0.02	<0.02
	2 days after taken off fortified diet	<0.02	--	--	<0.02
	4 days after taken off fortified diet	<0.02	--	--	<0.02
Milk fat	15	<0.02	--	--	<0.02
	29	<0.02	--	--	<0.02
Milk – aqueous fraction	15	<0.02	--	--	<0.02
	29	<0.02	--	--	<0.02
Liver	31	<0.02	--	--	<0.02
	38 (7 days after taken off fortified diet)	<0.02	--	--	<0.02
Kidney	31	<0.02	--	--	<0.02
	38 (7 days after taken off fortified diet)	<0.02	--	--	<0.02

Sample	Day sample collected	Oxamyl residues, mg/kg, at dosing level (mg/kg feed)			
		0	2	10	20
Lean muscle	31	<0.02	--	--	<0.02
	38 (7 days after taken off fortified diet)	<0.02	--	--	<0.02
Subcutaneous fat	31	<0.02	--	--	<0.02
	38 (7 days after taken off fortified diet)	<0.02	--	--	<0.02

Laying hens

Du Pont Report no.:	O/ME 30
Compound dosed:	Oxamyl
Nature of dose:	Oxamyl admixed with grain concentrate portion of ration using technical oxamyl (97% pure).
Number/type per feeding group:	Leghorn hens; 8 per dietary level
Dose period:	4 weeks
Depuration period:	1 week
Feeding levels (mg oxamyl/kg in the feed on a dry weight basis):	0, 1, and 5
Feed:	Dayett Mills Laying Mash + 1% corn oil

This study by Zahnow (1978) determined levels of oxamyl in the eggs and tissues of hens dosed with oxamyl daily for 4 weeks. All the hens dosed at the 0- and 1-mg/kg feeding levels and half from the 5-mg/kg were killed after the 4 weeks, and those remaining from the 5-mg/kg level were given unfortified rations for an additional week before being killed. Oxamyl was determined as described above for cattle with the same results. The LOQ for eggs and the tissues was 0.02 mg/kg (except skin 0.05 mg/kg owing to limited sample size). The ingestion of oxamyl had no adverse effect upon body weight or egg production. The results are given in Table 38.

Table 38. Oxamyl residues in the eggs and tissues of laying hens (Zahnow, 1978).

Sample	Sample period, weeks	Oxamyl residue, mg/kg, at dosing level (mg/kg feed)		
		0	1	5
Eggs (from hens at same dose, composited)	1	<0.02	<0.02	<0.02
	2	<0.02	<0.02	<0.02
	3	<0.02	<0.02	<0.02
	4	<0.02	<0.02	<0.02
	5 (1 week after taken off fortified diet)	--	--	<0.02
Liver	4	<0.02	<0.02	<0.02
	5 (1 week after taken off fortified diet)	--	--	<0.02
Fat	4	<0.02	<0.02	<0.02
	5 (1 week after taken off fortified diet)	--	--	<0.02
Muscle	4	<0.02	<0.02	<0.02
	5 (1 week after taken off fortified diet)	--	--	<0.02
Skin	4	<0.05	<0.05	<0.05
	5 (1 week after taken off fortified diet)	--	--	<0.05

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

A market basket survey to determine the distribution and level of certain methylcarbamate insecticide residues in single serving samples of fresh fruits and vegetables available for consumption by the US population was submitted by Carringer (2000). The Carbamate Market Basket Study (CMBS) was conducted according to an Environmental Protection Agency (US EPA) approved study design (individual foods eaten as a single-unit serving such as apples and peaches, or a multiple-unit serving

such as grapes and strawberries). Over the one-year study more then 400 samples from eight different crops including apples, tomatoes, lettuce, grapes, peaches, broccoli, and oranges, were collected from grocery stores throughout the USA and analysed for aldicarb, its sulfone and sulfoxide, carbaryl, carbofuran, 3-hydroxycarbofuran, methomyl, oxamyl and thiodicarb. A US EPA-approved statistical sampling design for collecting the commodities as well as sample preparation procedures (washing apples and tomatoes), peeling (bananas and oranges), coring (apples) reflecting consumer practices were incorporated in the study. The commodities analysed for oxamyl and the residues found are shown in Table 39.

Table 39. US Carbamate Market Basket Study (CMBS), distribution of oxamyl residues detected in single unit crop samples (Carringer, 2000).

Crop	Total no. of analyses	Samples with no residues, ≥ 0.001 mg/kg		Samples with residues ≥ 0.001 mg/kg		Range of residues, mg/kg	US MRL, mg/kg
		no.	%	no.	%		
Apple	400	379	95	21	5	ND – 0.038	2
Tomato	399	388	97	11	3	ND – 0.014	2
Orange	399	399	100	0	0	ND	3
Banana	400	400	100	0	0	ND	0.3

ND: <0.001 mg/kg (LOQ)

The United States Department of Agriculture's Pesticide Data Program (PDP) collects data on pesticide residues in food in or on selected commodities in the USA. The results of the PDP for oxamyl from 1993 to 1998 are shown in Table 40.

Table 40. Distribution of oxamyl residues (United States Department of Agriculture, 1993-1998).

Commodity	Total no. of samples	No. of detections	% of samples with detections	Minimum detected, mg/kg	Maximum detected, mg/kg	US MRL, mg/kg
USDA, 1993						
Apples	650	45	6.9	0.014	0.68	2
Bananas	614	1	0.2	0.065	0.065	0.3
Celery	620	140	22.6	0.014	0.82	3
Green beans	554	4	0.7	0.057	0.22	NT
Grapefruit	624	1	0.2	0.3	0.3	3
Orange	623	9	1.4	0.065	0.31	3
Peaches	358	1	0.3	0.014	0.014	NT
Potatoes	638	1	0.2	0.065	0.065	0.1
USDA, 1994						
Apples	687	23	3.3	0.014	0.32	2
Celery	176	29	16.6	0.014	0.28	3
USDA, 1995						
Apples	692	29	4.2	0.016	0.088	2
Green Beans	598	1	0.2	0.025	0.025	NT
USDA, 1996						
Apple juice	177	0				2
Apples	530	18	3.4	0.015	0.043	2
Carrots	500	0				0.1
Grapes	525	0				NT
Green beans	531	0				NT
Oranges	518	0				0.3
Peaches	325	0				NT
Spinach	517	2	0.4	0.015	0.082	NT
Sweet corn	173	0				NT
Sweet peas	355	0				NT
Sweet potatoes	507	0				0.1
Tomatoes	174	2	1.1	0.021	0.022	2

Commodity	Total no. of samples	No. of detections	% of samples with detections	Minimum detected, mg/kg	Maximum detected, mg/kg	US MRL, mg/kg
USDA, 1997						
Apple juice	693	1	0.1			2
Green beans	707	0		0.017	0.017	NT
Orange juice	692	0				3
Peaches	756	0				NT
Pears	708	6	0.8	0.017	0.12	2
Spinach, fresh	512	0				NT
Spinach, canned	168	0				NT
Sweet potatoes	695	0				0.1
Tomatoes	722	6	0.8			2
W Squash, fresh	440	0				2
W Squash, frozen	221	0		0.015	0.043	2
USDA, 1998						
Apple juice	694	0				2
Cantaloupe	408	1	0.2	0.015	0.015	2
Grape juice	653	0				NT
Green beans	359	0				NT
Orange juice	700	0				3
Pears	712	8	1.1	0.03	0.15	2
Spinach, canned	695	0				NT
Strawberries, fresh	610	0				NT
Strawberries, frozen	47	0				NT
Sweet potatoes	357	0				0.1
Tomatoes	717	4	0.6	0.015	0.058	2
W. Squash, fresh	530	1	0.2	0.9	0.9	2
W. Squash, frozen	149	0				2

NT: No US MRL

NATIONAL MAXIMUM RESIDUE LIMITS

Residue definition: sum of oxamyl and oxamyl oxime, expressed as oxamyl unless otherwise stated.

Country	Commodity	MRL, mg/kg	Residue definition
Australia	Banana	0.2	
	Cereals	0.02*	
	Edible offal (mammalian)	0.02*	
	Eggs	0.02*	
	Meat (mammalian)	0.02*	
	Milks	0.02*	
	Poultry fats	0.02*	
	Poultry meat	0.02*	
	Poultry, edible offal of	0.02*	
	Tomato	0.05	
Austria	Potato	0.05	
	Sugar beet	0.05	
Canada	Potato	0.1	
	Raspberry	0.1	
France	Banana	0.2	
Germany	All products of plant origin	0.05	Oxamyl
Italy	Sugar beet	0.05	
Japan	Apple	2	
	Artichoke	0.1	

Country	Commodity	MRL, mg/kg	Residue definition
	Banana	0.2	
	Barley	0.02	
	Bell pepper	2	
	Buckwheat	0.02	
	Burdock	0.1	
	Cabbage	0.02	
	Carrot	0.2	
	Celery	5	
	Chicory	0.1	
	Coffee beans	0.1	
	Corn	0.05	
	Cotton seed	0.2	
	Cucumber	2	
	Egg plant	2	
	Endive	0.5	
	Garlic	0.1	
	Ginger	0.1	
	Grapefruit	5	
	Horseradish	0.1	
	Japanese large orange	5	
	Japanese pear	2	
	Kidney bean, immature	0.2	
	Lemon	5	
	Lettuce	0.5	
	Lime	5	
	Mandarin orange	3	
	Orange	5	
	Melon	2	
	Onion	0.05	
	Other alium vegetables	0.1	
	Other citrus fruits	5	
	Other curcurbit vegetables	1	
	Other edible chrysanthemums	1	
	Other grains	0.02	
	Other potatoes	0.1	
	Parsley	0.1	
	Parsnip	0.1	
	Peanuts	0.1	
	Pear	2	
	Pineapple	1	
	Potato	0.1	
	Pumpkin	2	
	Radish, leaf	1	
	Radish, root	0.5	
	Raspberry	0.1	
	Rice	0.02	
	Rye	0.02	
	Salsify	0.1	
	Soya	0.1	
	Strawberry	0.02	
	Sugar beet	0.1	
	Sugar cane	0.05	
	Sweet potato	0.1	
	Taros	0.1	
	Tomato	2	
	Turnip, leaf	1	
	Turnip, root	0.1	
	Watermelon	2	
	Wheat	0.02	
	Yams	0.1	
Macedonia	Tomato	0.1	
Poland	Celery	0.05	
	Cucumber	0.05	
	Onion	0.05	
	Paprika	0.05	

Country	Commodity	MRL, mg/kg	Residue definition
	Potato	0.05	
	Tomato	0.05	
South Africa	Banana	0.05	
	Groundnuts	0.05	
	Pineapple	0.05	
	Potato	0.05	
	Sugar cane	0.05	
	Tobacco	0.1	
Spain	Beans	0.05	
	Brassica leafy vegetables	0.05	
	Bulb vegetables	0.05	
	Cereals	0.05	
	Citrus	3	
	Cucurbit vegetables	2	
	Edible seeds	0.05	
	Forage and straw	0.05	
	Fresh beans	2	
	Leafy vegetable	0.05	
	Mushrooms and fungi	0.05	
	Nuts	0.05	
	Other fruits	0.2	
	Pineapple	1	
	Pome fruit	0.05	
	Potatoes	0.1	
	Root and tuber vegetables	0.05	
	Seeds	0.1	
	Small fruit and berries	0.05	
	Solanacea vegetables	2	
	Stem and stalk vegetables	0.05	
	Stone fruit	0.05	
	Sugar beet	0.1	
	Sugar cane	0.1	
	Tobacco	1	
Taiwan	Bulb vegetables	2	
	Citrus	0.5	
	Cucurbits	2	
	Leafy vegetable	2	
	Legume vegetables	2	
	Melon	0.5	
Turkey	Peanuts	0.2	
	Root and tuber vegetables	0.5	
	Tomato	2	
	Leafy vegetable	0.1	
	Onion	0.1	
	Potato	0.1	
USA	Apple	2	
	Banana	0.3	
	Cantaloupe	2	
	Celery	3	
	Citrus fruits	3	
	Cotton seed	0.2	
	Cucumbers	2	
	Egg plants	2	
	Honeydews	2	
	Peanut, forage	2	
	Peanut, hay	2	
	Peanuts	0.2	
	Pears	2	
	Peppermint, hay	10	
	Peppers, bell	3	
	Peppers, non-bell	5	
	Pineapples	1	
	Pineapples, forage	10	
	Potatoes	0.1	
	Pumpkins	2	

Country	Commodity	MRL, mg/kg	Residue definition
	Root crop vegetables	0.1	
	Soya bean straw	0.2	
	Soya beans	0.2	
	Spearmint, hay	10	
	Summer squash	2	
	Tomatoes	2	
	Watermelon	2	
	Winter squash	2	

* MRL at the LOQ

APPRAISAL

Oxamyl was first evaluated in 1980 for toxicology and residues. The latest evaluation was in 1986 for residues. The compound was listed by the 1997 CCPR (29th Session, ALINORM 97/24A) for Periodic Re-evaluation for residues by the 2002 JMPR.

The manufacturer provided information to the Meeting on metabolism in animals and plants, environmental fate in soil and water, methods of residue analysis and stability of residues in stored analytical samples, uses, residue supervised trials and processing data as well as national MRLs. Information on national GAP data was provided by the governments of Australia, Germany and The Netherlands. Germany and The Netherlands indicated that oxamyl is no longer authorized for use. National MRLs were provided by the governments of Australia, Germany, Poland and The Netherlands.

Pure oxamyl is a white crystalline solid with a melting point of 99.8°C and low volatility. It has medium-high solubility in water and high solubility in certain organic solvents. The log P_{OW} of 0.36 suggests that the compound is not fat soluble.

Metabolic products

The parent, metabolites and degradation products are identified by code numbers as shown below.

Code	Chemical name, Short name	
DPX-D1410	*N,N*-dimethyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide	Oxamyl
IN-A2213	2-hydroxyimino-*N,N*-dimethyl-2-(methylthio)acetamide	Oxamyl oxime
IN-N0079	*N,N*-dimethyl-2-nitriloacetamide	DMCF
IN-D2708	dimethylamino(oxo)acetic acid,	DMOA
IN-L2953	**2-hydroxyimino-*N*-methyl-2-(methylthio)acetamide**	**N-demethyloxime, NDMO**
IN-KP532	methylamino(oxo)acetic acid	
IN-D1409	*N*-methyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide	*N*-demethyl-oxamyl
IN-T2921	*N,N*-dimethyloxamide	DMEA (also DMO)

Animal metabolism

Absorption, distribution, metabolism and excretion of ^{14}C-oxamyl were studied in rats, goats and hens. The metabolism of rats dosed with ^{14}C-oxamyl-oxime (the less toxic, principal hydrolysis product) was also studied to obtain sufficient metabolites to establish the metabolic pathway for oxamyl in rats. Oxamyl was rapidly and extensively metabolised in both livestock (goats and poultry) and laboratory animals (rats) and its metabolic products were mainly excreted in the urine or excreta. The initial steps in the metabolic pathway for oxamyl in goats and hens is similar to that described for rats. The proposed pathway involves oxamyl hydrolysis to oxamyl-oxime (IN-A2213), or Beckmann type rearrangement to IN-N0079 (IN-N0079 could also be formed directly from oxamyl-oxime). IN-N0079 is then converted to IN-D2708 and ultimately incorporated into natural

products. Minor metabolites (IN-L2953, IN-KP532 and IN-D1409) resulting from demethylation reactions were also observed. In livestock studies, the detoxification of IN-N0079 as thiocyanate (through cyanide displacement) was a major part of the pathway. The major metabolite found in lactating goats and laying hens was thiocyanate and radioactivity resulting from incorporation into natural products (such as lactose). In rats, IN-N0079 conversion to thiocyanate was not observed, however conjugates of the principal metabolites were found.

Plant metabolism

Based on the metabolism studies conducted with ^{14}C-oxamyl *via* direct foliage, fruit or soil applications in potatoes, peanuts, tobacco, tomatoes, oranges, and apples, IN-A2213 and IN-L2953 were identified as major breakdown products of oxamyl. The metabolic pathway of oxamyl was similar in the various crops. Metabolism of oxamyl in plant tissues included hydrolysis of the methylcarbamoyl group to yield oxamyl oxime (IN-A2213). IN-A2213 was demethylated before or after glucose conjugation to give IN-L2953 and/or its glucose conjugate. Conjugation of the glucosides of IN-A2213 and IN-L2953 with additional sugar residues was also observed. IN-A2213 (or oxamyl) may also be metabolised to IN-N0079, which is metabolised to IN-D2708 and ultimately incorporated into plant natural products. The only residue of toxicological concern in any plant tissue is oxamyl.

Environmental fate

Laboratory soil degradation studies were conducted in a variety of differing soils. In these studies, IN-A2213 and IN-D2708 were the major (>10%) degradation products of oxamyl consistently observed in soil. The only other major products consistently observed were non-extractable residues and carbon dioxide. Carbon dioxide was the predominant degradation product in all cases. IN-N0079 was observed >10% in the soil photolysis study, but was never detected in any of the other topsoil degradation studies. The half live of oxamyl under aerobic and anaerobic conditions was about 20 days.

In three confined rotational crop studies, the presence of oxamyl, oxamyl-oxime and IN-D2708 in crop samples demonstrated that oxamyl, as well as its soil degradates (oxamyl-oxime and IN-D2708), were taken up by the succeeding crop. The identification of these components and the characterisation of several tentatively identified metabolites (IN-KP532, IN-T2921, IN-L2953 and IN-N0079) further support the metabolic profile in the rotated crop. The proposed metabolic pathway of oxamyl in rotated crops is consistent with the metabolic pathway observed in oxamyl plant metabolism studies. Oxamyl was hydrolysed to oxamyl-oxime, which was ultimately metabolised to IN-D2708 and other polar metabolites. The conversion of oxamyl-oxime to IN-D2708 has been reported to proceed through IN-N0079 and IN-T2921. Oxamyl-oxime can also be demethylated to give IN-L2953, which can be metabolised to IN-KP532. A major component in rotated crops (specifically barley) is proposed to be the glucose conjugate of oxamyl-oxime, a major plant metabolite. The field rotational crop study confirms that succeeding crops take up oxamyl equivalents (oxamyl and/or oxamyl-oxime) when planted 30 days after oxamyl application. However, in the human-edible portion of crops planted 120 days after oxamyl application, no significant oxamyl residues were detected.

IN-A2213 was observed as the only major degradation product in hydrolysis and aqueous photolysis studies. IN-A2213, IN-D2708, IN-N0079, and IN-T2921 were each observed as major degradation products in the water phase of the water/sediment study. Only IN-D2708 was observed exceeding 10% (10.4% to 12.1%) in the sediment phase of the water sediment study.

Methods of analysis

Oxamyl is considered the only relevant analyte in the total toxic residue. However, earlier GLC methods convert oxamyl to oxamyl-oxime and report the total residues of the two analytes in oxamyl

equivalents. Oxamyl (including oxamyl-oxime) residues in plants are detected by initial extraction with ethyl acetate, followed by liquid-liquid partitioning cleanup, and alkaline hydrolysis to the more volatile oximino fragment (oxamyl-oxime, IN-A2213), and final determination by GLC with sulphur-sensitive flame photometric detection. LOQ is 0.02 mg/kg for dry and watery crops. The method can be used for animal products (LOQ fat, meat, kidney, liver 0.04 mg/kg; milk 0.02 mg/kg). Additional clean-up using GPC followed by conversion of oxamyl to oxamyl-oxime and analysis by GLC/MS improved the LOQ for animal products (0.01 mg/kg).

HPLC methods are able to analyse oxamyl only. Oxamyl is extracted with ethyl acetate using a homogeniser. Water is added to the extract and then the ethyl acetate is evaporated under vacuum. Cleanup is performed with liquid-liquid extractions. Reversed phase HPLC/UV under isocratic conditions is used to determine quantitatively oxamyl *per se*. Other methods performed extraction by accelerated solvent extraction rather than traditional mechanical extraction in ethyl acetate. The samples are extracted using an accelerated solvent extractor (ASE) and acetone. The acetone extract is passed through an SPE cartridge to remove pigments and other interfering molecules. The whole SPE eluate is then concentrated to about 0.5 ml by evaporation. The extract is dissolved in a mixture of 10% acetone in cyclohexane (v:v) and applied to a Silica Mega Bond Elute SPE cartridge to complete the clean-up. A HPLC/UV equipped with column switching valve is used.-The LOQ of oxamyl *per se* is 0.02 mg/kg.

Stability of residues in stored analytical samples

Oxamyl and oxamyl-oxime (IN-A2213) are stable in representative crop matrices (watery, starchy, oily, and dry crops) stored frozen for extended periods. These stability data support the magnitude of residue and residue decline supervised trials. In addition, oxamyl is shown to be stable in water and soil matrices when stored frozen.

Definition of residue

Oxamyl is rapidly metabolized in animals (rats, goats, hens) and has not been isolated intact in animal products. None of oxamyl's metabolites contain the carbamate moiety. The major metabolite identified in milk, eggs and tissues was thiocyanate.

Based on the metabolism studies conducted in potatoes, peanuts, tobacco, tomatoes, oranges, and apples, oxamyl-oxime (IN-A2213) and N-demetyloxime (IN-L2953) were identified as major breakdown products of oxamyl. None of the metabolites contain the carbamate moiety responsible for cholinesterase inhibition. The Meeting concluded that the only residue of toxicological concern in any plant tissue is oxamyl. However, most of residue supervised trials samples were analyzed by a GLC method which converts oxamyl to oxamyl-oxime and reports the total residues of the two analytes in oxamyl equivalents.

Due to the nature of the residues determined in supervised residue trials in plants submitted, the Meeting recommended that the definition of the residue for compliance with MRLs should be the sum of oxamyl and oxamyl-oxime expressed as oxamyl.

For the estimation of dietary intake the residue definition should be oxamyl per se. Because the estimated STMRs and HRs are based on the sum of oxamyl and oxamyl-oxime, the Meeting noted that an overestimate of the dietary intake calculations cannot be excluded.

This residue definition applies for both plant and animal commodities.

Results of supervised trials

Oxamyl is used world-wide as a foliar spray or soil treatment in citrus fruits but only US residue supervised trials were submitted. The current US label indicates oxamyl may be applied at either 0.56 - 1.1 kg ai/ha per foliar spray application (not more than 6.7 kg ai/ha per season) or 0.56 - 2.2 kg ai/ha as soil irrigation treatment (not more than 2.2 kg ai/ha in any 30 day period) with a 7-day PHI. No trials were submitted for soil treatment. Three foliar-sprayed grapefruit trials, five orange trials and two lemon trials (1 x 1.1 − 1.5 kg ai/ha, 0.0012 − 0.03 kg ai/hl PHI 7 − 8 days) resulted in residues in whole fruits of 0.13, 1.2 and 2 mg/kg for grapefruit, of 0.14, 0.16, 0.3, 0.34 and 0.8 mg/kg for oranges and of 0.05 and 0.17 mg/kg for lemon. One further trial on oranges treated five times by foliar spraying in a monthly interval (1.1 kg ai/ha, 0.24 kg ai/hl) showed residues lower than the LOQ of 0.04 mg/kg at PHIs of 4 or 8 days. Further trials on tangelo and tangerine did not match the GAP. Combined residue levels of grapefruit, oranges and lemon in rank order (median underlined) were: <0.04, 0.05, 0.13, 0.14, 0.16, 0.17, 0.3, 0.34, 0.8, 1.2 and 2 mg/kg.

Based on residues in whole fruit (no data were available for edible portion), the Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in citrus fruit of 3, 0.17 and 2 mg/kg, respectively. The estimated maximum residue level replaces the current MRL recommendation of 5 mg/kg for citrus fruits.

In the USA, oxamyl is registered for foliar treatment in apples at application rates of 0.56 − 2.2 kg ai/ha (not more than 2.2 kg ai/ha per season) with a PHI of 14 days. Oxamyl levels were after one treatment of 2.2 kg ai/ha (0.42 − 0.5 kg ai/hl) <0.1, 0.18, 0.24, 0.25, 0.26, 0.44, 0.49, 0.52, 0.59, and 0.81 mg/kg. The Meeting noted that a single residue of 1.2 mg/kg was found after two spray treatments at 1.1 kg/ha (interval of 7 days). This value was considered for maximum residue level and HR estimation.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in apples of 2, 0.35 and 1.2 mg/kg, respectively. The current MRL recommendation of 2 mg/kg was confirmed.

The current Australian label indicates oxamyl may be applied at the base of the plant on bananas three times at 1.8 to 3 g ai/plant on a minimum 90-day interval throughout the growing season with an unspecified PHI. Application rates in available Australian trials (1990) according to GAP were 3 x 1.8 or 2.4 g ai/plant (2 trials each). The residues after a PHI of 0 − 28 days were in whole fruit <0.01, 0.01, 0.02, 0.02 mg/kg, in peel <0.01, 0.02, 0.03, 0.03 mg/kg and in pulp <0.01 mg/kg (4). No trial according to the maximum GAP (3 x 3 g ai/plant) was submitted. Further overdosed trials (3 x 4.8 g ai/plant, 6 x 3 g ai/plant, 6 x 6 g ai/plant) show maximum residues in whole fruit, peel and pulp of 0.08, 0.2 and 0.01 mg/kg, respectively. These results indicate that detectable residues may occur in pulp.

The Meeting concluded that there were insufficient data to estimate a maximum residue level for banana, and withdrew the current recommendation of 0.2 mg/kg.

The current USA label indicates oxamyl may be applied on pineapple at 4.5 kg ai/ha at planting followed by applications at 1.1 - 2.2 kg ai/ha (not more than 8.9 kg ai/ha per season) on a minimum 2-week interval throughout the growing season with a 30-day PHI. For purposes of proposing an MRL, oxamyl residue data obtained from US trials with foliar applications of 4 − 5 x 2.2 kg ai/ha (PHI 23 − 35 days) were considered. The residue concentrations were 0.05, 0.17 and 0.59 mg/kg after 4 − 5 treatments with 2.2 kg ai/ha.

The Meeting concluded that there were insufficient data to estimate a maximum residue level for pineapple, and withdrew the current recommendation of 1 mg/kg.

Oxamyl is world-wide registered as foliar spray or soil treatment in onions, but no residue supervised trials data have been received. The Meeting agreed to withdraw the previous recommendation for onion, bulb (0.05* mg/kg).

The current USA label for cucumber and melons indicates oxamyl may be applied to the soil at 4.5 kg ai/ha at planting followed by foliar spray applications at 0.56 - 1.1 kg ai/ha (not more than 6.7 kg ai/ha per season) on a minimum 1-week interval throughout the growing season with a 1-day PHI. For foliar spray use on cucumber, altogether five US outdoor residue trials (1976 - 1978) according to the above named GAP (5 – 7 x 1.1 kg ai/ha) were submitted. Residue levels in rank order were: 0.3, 0.37, 0.38, 0.47 and 0.54 mg/kg. For melons, six US outdoor foliar-sprayed trials according to GAP (5 – 8 x 1.1 kg ai/ha) were submitted and showed residues of 0.16, 0.2, 0.26, 0.26, 0.39, 0.5 mg/kg.

The Meeting noted that the residue data on cucumber and melons were similar and could be combined for mutual support. The combined residues were, in rank order, 0.16, 0.2, 0.26, 0.26, 0.2, 0.37, 0.38, 0.39, 0.47, 0.5 and 0.54 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in cucumber and melons, except watermelons, of 1, 0.37 and 0.54 mg/kg, respectively. The estimated maximum residue level replaces the current recommendations (2 mg/kg).

Oxamyl is registered in the USA in watermelon with soil treatment pre-plant or at planting with 4.5 kg ai/ha followed by foliar spray of 1.1 kg ai/ha (not more than 6.7 kg ai/ha per season) with a 1-day PHI. Only one US trial according to GAP (8 x 1.1 kg ai/ha) was submitted and resulted in a residue of 0.77 mg/kg one day after application.

The Meeting concluded that there were insufficient data to estimate a maximum residue level. The previous MRL recommendation of 2 mg/kg should be withdrawn.

Oxamyl is registered in the USA in summer squash with soil treatment pre-plant or at planting with 2.2 – 4.5 kg ai/ha followed by foliar spray of 0.56 – 1.1 kg ai/ha (not more than 6.7 kg ai/ha per season) with a 1-day PHI. Two overdosed supervised residue trials were submitted (5 x 2.2 mg/kg) but no trials carried out according to GAP were provided. The Meeting recommended to withdraw the previous MRL recommendation of 2 mg/kg.

The current USA label indicates oxamyl may be applied to peppers as a transplant water treatment at a maximum rate of 0.56 kg ai/ha. Following transplant, foliar or soil applications may be at a rate of 0.56 - 1.1 kg ai/ha (not more than 6.7 kg ai/ha) on a minimum 1-week interval through out the growing season with a 7-day PHI. For purposes of proposing an MRL, oxamyl residue data obtained from eight outdoor US trials with a 5 to 7-day PHI and 5 – 8 foliar applications at 1.1 kg ai/ha were considered. No information on variety etc. (bell/long, sweet/hot) was stated in report 9F 2266 (1975/76), but 2 trials were according to GAP resulting in residues of 0.62 and 0.73 mg/kg. In Residues were 0.13, 0.75 and 0.76 mg/kg in sweet pepper and 1.5, 1.8 and 4.3 mg/kg in hot pepper. All residues were in rank order 0.13, 0.62, 0.73, 0.75, 0.76, 1.5, 1.8 and 4.3 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in peppers of 5, 0.755 and 4.3 mg/kg, respectively. The previous recommendation of 2 mg/kg for sweet peppers is withdrawn.

Oxamyl is registered in tomato in the USA for foliar spray use at 0.56 – 1.1 kg ai/ha and for soil drip irrigation at 0.56 – 2.2 kg ai/ha (not more than 8.9 kg ai/ha/season) and a 3-day PHI. Field supervised trials were conducted in 1997 in tomato for both uses in the USA. (foliar spray; drip irrigation 9 - 10 x 1.1 – 2.2 kg ai/ha, PHI 3 days). Residues after 9 - 10 drip irrigation treatments with total 13.3 – 13.5 kg ai/ha/season ranged from 0.12 – 0.72 mg/kg at a 3-day PHI. These values were not included in the evaluation (overdosed). Residues after foliar spray with 8 x 1.1 kg ai/ha (PHI 3 days) were, in rank order, 0.06, 0.27, 0.33, 0.42, 0.48, 0.5, 0.54, 0.55, 0.61, 0.61, 0.69, 0.74, 0.76, 0.82, 0.93, 0.99 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in tomato of 2, 0.58 and 0.99 mg/kg, respectively. The previous recommendation was confirmed.

Oxamyl is registered in Peru and Saudi Arabia for beans but no residue supervised trials data have been received. The Meeting agreed to withdraw the previous recommendations for beans, except broad bean and soya bean of 0.2 mg/kg and for soya bean (dry) of 0.1 mg/kg.

In the USA, oxamyl is registered for soil directed spray use on carrots (3 x 1.1 kg ai/ha, PHI 14 days). Other uses are one soil broadcast treatment at 8.9 kg ai/ha pre-planting or 4.5 kg ai/ha in furrow at planting, all in all not more than 8.9 kg ai/ha per season. In five trials that matched GAP with one pre-emergence/in furrow/pre-plant soil treatment (4.5 – 5.6 kg ai/ha) and three further soil treatments (1.1 kg ai/ha), oxamyl residues, in rank order, were: <0.02, 0.02, 0.03, 0.04 and 0.07 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in carrots of 0.1, 0.03 and 0.07 mg/kg, respectively. The previous recommendation of 0.1 mg/kg for root and tuber vegetables is withdrawn.

The current US label indicates oxamyl may be applied to potatoes once prior to planting or at planting at up to 4.5 kg ai/ha. Post-planting, oxamyl may be applied as a foliar spray up to 6 times at a maximum rate of 1.1 kg ai/ha on a minimum 5-day interval throughout the growing season with a 7-day PHI. For MRL-purposes, oxamyl residue data obtained from 7 trials with 6-to-7-day PHI following one soil treatment with 4.5 kg ai/ha and five to six foliar applications at 1.1 kg ai/ha or five foliar sprays with 1.1 kg ai/ha only were considered. Oxamyl residues, in rank order, were <0.02 (4), 0.03 (2), 0.05 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in potatoes of 0.1, 0.02 and 0.05 mg/kg, respectively. The previous recommendation of 0.1 mg/kg for root and tuber vegetables is withdrawn.

In the USA, oxamyl is registered for use on celery with one treatment pre-planting (2.2 - 4.5 kg ai/ha) plus foliar spray treatments of 0.56 – 2.2 kg ai/ha (not more than 6.7 kg ai/ha) with a 21-day PHI. Eight US trials each carried out with 3 scenarios: (a) pre-planting 1 x 4.5 kg ai/ha plus foliar spraying 2 x 1.1 kg ai/ha, (b) 2 x 1.1 kg ai/ha banded plus 4 x 1.1 kg ai/ha foliar broadcast spray and (c) 6 x 1.1 kg ai/ha foliar broadcast spray (PHI 21 days) were submitted but did not match the maximum GAP (2.2 kg ai/ha, foliar spay).

The Meeting concluded that there were insufficient data according to maximum GAP to estimate a maximum residue level for celery. The previous MRL recommendation of 5 mg/kg should be withdrawn.

Oxamyl is registered in Saudi Arabia for maize but no residue supervised trials data have been received. The Meeting recommended to withdraw the previous MRL recommendation of 0.05* mg/kg for maize.

Oxamyl is registered in Saudi Arabia and Taiwan for sugar cane but no residue supervised trials data were received. The Meeting recommended to withdraw the previous MRL recommendation of 0.05* mg/kg for sugar cane.

The current USA label indicates oxamyl may be applied as a foliar spray on cotton at a maximum rate of 4 x 1.1 kg ai/ha on a minimum 6-day interval and 21 (SL 240) or 14 (SL 420) days PHI. Eight US supervised trials with 3 – 5 treatments of 1.1 kg ai/ha and a 14/15-day PHI showed the following residues in cotton seed: <0.02, <0.02, 0.02, 0.03, 0.04, 0.05, 0.07, 0.08 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in cotton seed of 0.2, 0.035 and 0.08 mg/kg, respectively. The previous recommendation was confirmed.

The current USA label indicates oxamyl may be applied on peanuts once prior to planting or at planting at up to 3.4 kg ai/ha. Following emergence, oxamyl may be applied as a foliar spray twice, with the first application 3 weeks post-emergence and the second application 3 weeks later. The maximum post-emergence rate is 1.1 kg ai/ha. The seasonal maximum usage is 5.6 kg ai/ha. No PHI is specified. Eight trials were conducted in 1988 in the USA side-by-side under each of two application scenarios: (a) a pre- or at-plant application of 3.4 kg ai/ha followed by 2 foliar applications at 1.1 kg ai/ha with a 3-week interval, and (b) a pre- or at-plant application of 6.7 kg ai/ha followed by 2 foliar applications at 2.2 kg ai/ha (overdosed) with a 3-week interval. Oxamyl residues in peanut nutmeat at PHIs from 78 – 118 days were less than the LOQ of 0.02 (8) mg/kg for both scenarios.

Four further trials were conducted in 1975/76 in the USA side-by-side under each of two application scenarios: (c)a pre- or at-plant application of 3.4 kg ai/ha followed by 2 foliar applications at 1.1 kg ai/ha, and (d) a pre- or at-plant application of 5 kg ai/ha followed by 2 foliar applications at 1.1 kg ai/ha. Oxamyl residues in peanut nutmeat were <0.02 mg/kg (2) in the trials (d) with 5 + 1.1 + 1.1 kg ai/ha and <0.02 and 0.03 mg/kg in trials (c) with 3.4 + 1.1 + 1.1 kg ai/ha (PHI 61 and 75 days). All residue values in rank order were: <0.02 (11) and 0.03 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for oxamyl in peanut of 0.05, 0.02 and 0.03 mg/kg, respectively. The estimated maximum residue level replaces the current recommendation (0.1 mg/kg) for peanut.

The corresponding residue values in peanut hay resulting from the above evaluated GAP trials of treatment scenarios (a), (c) and (d) were <0.02 (3), 0.03, 0.04, 0.05, 0.06, 0.07 mg/kg (fresh weight). Allowing for the standard 85% dry matter (FAO Manual), the Meeting estimated a maximum residue level and an STMR value for oxamyl in peanut fodder of 0.2 mg/kg and 0.041 mg/kg (0.035/0.85). The estimated maximum residue level replaces the current recommendation (2 mg/kg) for peanut fodder.

Oxamyl is registered in Central America for coffee but no residue supervised trials data have been received. The Meeting recommended to withdraw the previous MRL recommendation of 0.1 mg/kg for coffee beans.

Fate of residues during storage and processing

Oxamyl residues in tomatoes declined during 3-week storage at 15°C in air and in modified atmospheres. About 50, 40 and 20 % of the initial residue were determined in air, in modified atmosphere 1 (1.5% O_2, 98.5% N_2) and in modified atmosphere 2 (1.5% O_2, 4% CO_2, 79% N_2), respectively.

One hydrolysis study to determine the effects of processing on the nature of residues shows that oxamyl-oxime (IN-A2213) was the only degradation product after simulated pasteurization, baking/boiling and sterilization.

The effect of processing on the concentrations of residues of oxamyl has been studied in oranges, pineapple, tomato, potato, peanut and cotton seed.

Oranges. (RAC residues 0.55 mg/kg) were processed into dry pomace and cold pressed oil with a processing factor of <0.036 for both commodities. Based on the STMR value of 0.17 mg/kg for citrus fruits, the STMR-Ps were 0.006 mg/kg for orange dry pomace and orange oil.

Pineapple. (RAC residues 0.1 mg/kg) were processed into juice and wet skins (pineapple processed residue, wet bran) with processing factors of 1.2 and 1.7. As no maximum residue level could be estimated, no STMR-Ps were calculated.

Tomatoes. (RAC residues 1.5 mg/kg) were processed into canned fruit, juice, paste, catsup and puree with processing factors of 0.073, 0.12, 0.36, 0.24 and 0.16 respectively. Based on the STMR value of 0.58 mg/kg for tomato, the STMR-Ps were 0.042, 0.07, 0.21, 0.14 and 0.093 mg/kg for tomato canned fruit, juice, paste, catsup and puree, respectively.

Potatoes. (RAC residues 0.02 mg/kg) were processed into peels, French fries, chips and granules. No detectable residues were reported in the processed commodities (<0.02 mg/kg) with the exception of peels (0.022 mg/kg). As the concentration of oxamyl residues were near the LOQ in the RAC, no STMR-P value could be estimated.

Peanut nutmeat. (RAC residues 0.12 mg/kg) were processed into meal, crude oil and refined oil. The processing factors were <0.17 for the processed commodities. Based on the STMR value of 0.02 mg/kg for peanut nutmeat, the STMR-Ps were 0.0034 for peanut meal, crude oil and refined oil.

Cotton seed. (RAC residues 2.4 mg/kg) were processed into delinted seeds, hulls, meal, crude oil and refined oil. The processing factors were 0.288, 0.417 and 0.0125 for delinted seeds, hulls and meal and <0.008 for the other processed commodities. Based on the STMR value of 0.035 mg/kg for cotton seed, the STMR-Ps were 0.01 mg/kg for delinted seeds, 0.0146 mg/kg for hulls, 0.0004 mg/kg for meal, 0.0003 mg/kg for crude and refined oil.

Residues in animal commodities

Dietary burden in animals

The Meeting estimated the dietary burden of oxamyl residues in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual. Calculation from MRLs and STMR-P values provides the levels in feed suitable for estimating MRLs for animal commodities, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage of dry matter is taken as 100% when MRLs and STMR values are already expressed as dry weight.

Estimated maximum dietary burden of farm animals

Commodity	Codex Commo-dity Group	Residue (mg/kg)	Basis	% Dry matter	Residue, dry wt (mg/kg)	Choose diets, %			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Orange pomace, dried	AO	0.006	STMR-P	91	0.0066	20	10		0.0013	0.0007	
Cotton seed hulls	AM	0.0146	STMR-P	90	0.016	20	15		0.003	0.0024	
Cotton seed	SO	0.2	MRL	88	0.227	25	25		0.057	0.057	
Cotton seed meal		0.0004	STMR-P	89	0.00045			20			0.00009
Peanut meal		0.0034	STMR-P	85	0.004	10		25	0.0004		0.001
Peanut hay	AL	0.2	MRL	100	0.2	25	50		0.05	0.1	
TOTAL						100	100	45	0.1117	0.1601	0.00109

Estimated STMR dietary burden of farm animals

Commodity	Codex Commodity Group	Residue (mg/kg)	Basis	% Dry matter	Residue, on dry wt (mg/kg)	Choose diets, %			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Orange pomace, dried	AO	0.006	STMR-P	91	0.0066	20	10		0.0013	0.0007	
Cotton seed hulls	AM	0.0146	STMR-P	90	0.016	20	15		0.003	0.0024	
Cotton seed	SO	0.035	STMR	88	0.0398	25	25		0.01	0.01	
Cotton seed meal		0.0004	STMR-P	89	0.00045			20			0.00009
Peanut meal		0.0034	STMR-P	85	0.004	10		25	0.0004		0.001
Peanut hay	AL	0.041	STMR	100	0.041	25	50		0.01025	0.0205	
TOTAL						100	100	45	0.025	0.0336	0.00109

The dietary burdens of oxamyl for estimating MRLs and STMR values for animal commodities (residue concentrations in animal feeds expressed as dry weight) are: 0.11 and 0.025 mg/kg for beef cattle, 0.16 and 0.03 mg/kg for dairy cattle and 0.001 mg/kg each for poultry.

Feeding studies

The Meeting received the information that no residues (<0.02 mg/kg) were detected in tissues (liver, kidney, muscle, fat) and milk (whole milk, milk fat, aqueous fraction) when dairy cows were dosed for 30 days with 2, 10 or 20 mg oxamyl/kg feed.

The Meeting received the information that no residues (<0.02 mg/kg) were detected in tissues and eggs when laying hens were dosed for 4 weeks with 1 and 5 mg oxamyl/kg feed.

The Meeting considered that it is unlikely that any oxamyl residues might occur in animal products as the maximum dietary burden is very low and the feeding studies did not show any residues in tissues, milk and eggs. MRLs at the LOQ of 0.02 mg/kg and STMRs/HRs of 0 were recommended for animal products as eggs, milks, meat of mammals, edible offal (mammalian), poultry meat and poultry, edible offal of.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels, STMR values and HR values shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue
For compliance with MRLs: sum of oxamyl and oxamyl oxime expressed as oxamyl.
For estimation of dietary intake: oxamyl

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg	HT or HR-P, mg/kg
CCN	Name	New	Previous		
FP 0226	Apple	2	2	0.35	1.2
FI 0327	Banana	W	0.2		
VP 0061	Beans, except broad bean and soya bean	W	0.2		
VR 0577	Carrot	0.1	-	0.03	0.07
VS 0624	Celery	W	5		
FC 0001	Citrus fruits	3	5	0.17	2
SB 0716	Coffee beans	W	0.1		
SO 0691	Cotton seed	0.2	0.2	0.035	0.08
OC 0691	Cotton seed oil, crude			0.0003	
OR 0691	Cotton seed oil, edible			0.0003	
VC 0424	Cucumber	1	2	0.37	0.54
MO 0096	Edible offal (Mammalian)	0.02 (*)	-	0	0

	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HT or HR-P, mg/kg
CCN	Name	New	Previous		
PE 0112	Eggs	0.02 (*)	-	0	0
GC0645	Maize	W	0.05 (*)		
MM 0095	Meat (from mammals other than marine mammals)	0.02 (*)		0	0
VC 0046	Melons, except Watermelon	1	2	0.37	0.54
ML 0106	Milks	0.02 (*)		0	
VA 0385	Onion, Bulb	W	0.05 (*)		
	Orange oil			0.006	
SO 0697	Peanut	0.05	0.1	0.02	0.03
AL 0697	Peanut fodder	0.2 (dry weight)	2	0.041	
OC 0697	Peanut oil, crude			0.0034	
OR 0697	Peanut oil, edible			0.0034	
VO 0051	Peppers	5	-	0.755	4.3
VO 0445	Peppers, Sweet	W	2		
FI 0353	Pineapple	W	1		
VR 0589	Potato	0.1	-	0.02	0.05
PM 0110	Poultry meat	0.02 (*)	-	0	0
PO 0111	Poultry, Edible offal of	0.02 (*)	-	0	0
VR 0075	Root and tuber vegetables	W	0.1		
VD 0541	Soya bean (dry)	W	0.1		
VC 0431	Squash, Summer	W	2		
GS 0659	Sugar cane	W	0.05 (*)		
VO 0448	Tomato	2	2	0.58	0.99
	Tomato canned fruit			0.042	
	Tomato ketchup			0.14	
JF 0448	Tomato juice			0.07	
	Tomato paste			0.21	
	Tomato purée			0.093	
VC 0432	Watermelon	W	2		

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes of oxamyl, based on the STMRs estimated for 19 commodities, for the five GEMS/Food regional diets were in range of 2 -– 10 % of the ADI (Annex 3). The Meeting concluded that the long-term intake of residues of oxamyl resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short term Intake (IESTI) for oxamyl was calculated for 20 food commodities for which maximum residue levels were estimated and for which consumption data were available. These results are shown in Annex 4.

The IESTI represented 0 – 610 % of the acute RfD for the general population and 0–1600 % of the acute RfD for children. The values 190, 190, 200, 300, 390, 390, 430, 610 and 610 % represent the estimated short-term intake for tomato, cucumber, lemon, melons, mandarins, oranges, apple, peppers and grapefruit respectively for the total population. The values 400, 650, 660, 730, 1100, 1100, 1300, 1400 and 1600 % represent the estimated short-term intake for cucumber, melons, tomato, lemon, peppers, grapefruit, apple, mandarins and oranges respectively for children.

The Meeting concluded that the short term intake of residues of oxamyl from uses, other than these 9 commodities, that have been considered by the JMPR is unlikely to present a public health concern.

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