

## OXAMYL (126)

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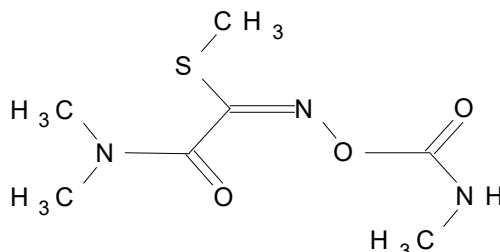
### EXPLANATION

Oxamyl is a carbamate insecticide that acts by inhibiting acetylcholinesterase activity. It was first evaluated by JMPR in 1980 (T, R) and periodic re-evaluation conducted in 2002 (T, R). It was included in the Priority List under the Periodic Re-Evaluation Program at the 48th Session of the CCPR (2016) for evaluation by the 2017 JMPR.

The Meeting received information on physical and chemical properties, animal and plant metabolism, rotational crop studies, environmental fate, analytical methods, GAP information, storage stability, processing and residue trial data on Brussels sprouts, cucumber, courgette, melon, tomato, pepper, eggplant, carrot, sugar beet and potato.

### IDENTITY

Common name:	Oxamyl
Chemical name	
IUPAC:	<i>N,N</i> -dimethyl-2-methylcarbamoyloxyimino-2-(methylthio) acetamide
CAS:	Methyl 2-(dimethylamino)- <i>N</i> -[[[(methylamino)carbonyl]oxy]-2-oxoethanimidothioate
CAS Registry No:	23135-22-0
CIPAC No:	342
Synonyms:	DPX-D1410
Structural formula:	



Molecular formula:	C <sub>7</sub> H <sub>13</sub> N <sub>3</sub> O <sub>3</sub> S
Molecular weight:	219.3

### PHYSICAL AND CHEMICAL PROPERTIES

#### *Pure active ingredient*

Property	Results	Reference
Appearance (color, physical state, odor)	White / Crystalline solid / Sulphurous (100.0% purity)	Tuffey, 2000 DuPont-2129
Vapour pressure	1.80 × 10 <sup>-5</sup> Pa at 20 °C 1.06 × 10 <sup>-3</sup> Pa at 40 °C (99.3% purity)	Harsh, 2009 DuPont-26259

Property	Results	Reference
Henry's law constant (pH 5 buffer)	$2.7 \times 10^{-8}$ Pa m <sup>3</sup> /mol at 20 °C	Malin, 2014 DuPont-38270
Melting point	98.5–100 °C (99.9% purity)	Craig, 2003 DuPont-13350
	99.2 °C (98.0% purity)	Jackson, 2004 DuPont-14983
Boiling point	Oxamyl decomposes at temperatures above approximately 165 °C without boiling (98.0% purity)	
Octanol/water partition coefficient	log Pow = -0.43 (99.1% purity)	Birnschein, 2014 DuPont-39338
Solubility in water (pH 5 buffer)	148.1 g/L at 20 ± 0.5 °C (98.3% purity)	Craig, 2003 DuPont-13351
Solubility in organic solvent (100.0% purity)	n-Heptane	10.5 mg/L at 20 °C
	o-Xylene	3.14 g/L at 20 °C
	Ethyl acetate	41.3 g/L at 20 °C
	Acetone	>250 g/kg at 20 °C
	Methanol	>250 g/kg at 20 °C
	Dichlorethane	>250 g/kg at 20 °C
Relative density	1.313 ± 0.001 g/cm <sup>3</sup> at 23.6 ± 0.1 °C (100.0% purity)	Tuffy, 2000 DuPont-2165
Hydrolysis	Oxamyl was shown to be hydrolytically stable at acidic pH 4. DT <sub>50</sub> = 21.1 days at pH7 at 20 °C DT <sub>50</sub> = 0.20 days at pH9 at 20 °C (98% purity) The rate of degradation was temperature dependent and increased with increasing temperature.	Clark, 2014 DuPont-39015
Photolysis	DT <sub>50</sub> = 3.5 experimental days at pH 5 at 25.3 ± 0.4 °C (99.9% purity) Environmental half-lives: 4.1 days in Phoenix, Arizona (USA, 33.3 °N) 5.8 days in Edmonton, Alberta (Canada, 53.3 °N) 6.3 days in Athens, Greece (EU, 38.0 °N) 7.9 days in London, Great Britain (EU, 51.3 °N) 8.7 days in Tokyo (Japan, 35.1 °N)	Hall, 2014 DuPont-38008
Dissociation constant	Oxamyl is non dissociative under the test condition of pH values between 2.4 and 11.6 at 20 ± 0.5 °C (98.0% purity)	Harsha, 2009 DuPont-26918

### Technical material

Property	Results	Reference
Appearance	no information	
Density		
Purity		
Melting range		
Thermal stability		
Stability		

### Formulations

Oxamyl is registered as a granule (GR) formulation containing 50 g/kg and 100 g/kg. It is also registered as soluble concentrate (SL) formulation containing 100 g/L.

### METABOLISM AND ENVIRONMENTAL FATE

The metabolism of oxamyl has been investigated in plants and animals. The fate and behaviour of oxamyl in plants, animals and the environment was investigated using the [<sup>14</sup>C] labelled test materials shown in Figures 1.

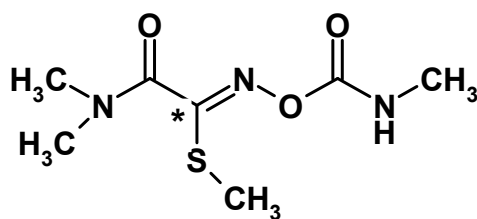


Figure 1 [ $^{14}\text{C}$ ]-Labelled test materials used in plants, animals metabolism studies, and the environmental fate studies[1- $^{14}\text{C}$ ]-oxamyl

The chemical structures of the major degradation compounds from the metabolism of oxamyl are provided below.

Compound name	Structure	Found in metabolism studies
IN-A2213 Oxamyl-oxime	Methyl 2-(dimethylamino)- <i>N</i> -hydroxy-2-oxoethanimidothioate 	Plants, Livestock, Soil
IN-QKT34 IN-A2213 (oxamyl-oxime) glucoside	[(Hexopyranosyloxy)imino]- <i>N,N</i> -dimethyl-2-(methylthio)acetamide 	Plants
IN-D2708 (Dimethylamino)oxoacetic acid		Plants, Livestock, Soil
IN-N0079 DMCF	Dimethylcarboncyanidic amide 	Plants, Livestock,
IN-F3905 Methyl ( <i>E</i> ) 2-(dimethylamino)- <i>N</i> -hydroxy-2-oxoethanimidothioate		Plants, Livestock,
IN-L2953 Methyl <i>N</i> -hydroxy-2-(methylamino)-2-oxoethanimidothioate		Plants, Livestock

Compound name	Structure	Found in metabolism studies
IN-KP532 (Methylamino)oxoacetic acid		Plants, Livestock
IN-KV998 N-Methylethanediamide		Plants, Livestock
IN-T2921 N,N-Dimethylethanediamide DMEA (also DMO)		Plants, Livestock,
IN-D1409 Methyl 2-(methylamino)-N- [[[(methylamino)carbonyl] oxy]-2- N-demethyl-oxamyl oxoethanimidothioate		Livestock
IN-00699 Ethanediamide Oxamide		Livestock
IN-18474 Aminoxyoxoacetic acid Oxamic acid		Livestock
Oxalic acid Ethanedioic acid		Livestock
Thiocyanate ion (shown as sodium salt)	$\text{Na}^+ \text{S}^- \equiv \text{N}$	Livestock

### Plant metabolism

Plant metabolism studies were performed on potatoes and tomatoes with [1-<sup>14</sup>C]-oxamyl. Metabolites were identified using multiple chromatographic systems and authentic standards.

#### Potatoes

The metabolic fate of oxamyl was investigated in potatoes (*Solanum tuberosum* L.) to determine the nature of the terminal <sup>14</sup>C-residues in the potato raw commodity (tubers) (Brown *et al.*, 2001: DuPont-4520).

Seed potatoes (cv. Red Pontiac; 3 seed pieces per pot) were planted in plastic pots containing sandy loam soil in a greenhouse. These pots were immediately treated with a soil single application of

<sup>14</sup>C-oxamyl formulated with inert formulation ingredients to simulate a SL formulation containing 10% w/v oxamyl at a rate of 8 kg ai/ha. Potatoes were maintained and grown to maturity in the greenhouse. At maturity (127 day PHI), the foliage and tubers were collected. The mature foliage was cut just above the soil surface and the tubers were dug up. A representative portion of the treated tubers was rinsed with water, patted dry, then peeled.

The potato peels and the peeled potatoes were separately homogenized and total radioactive residues (TRR) were determined by combustion and LSC analysis. Control potatoes (peels and peeled tubers), and treated and control foliage were also combusted to determine TRR. TRR (combustion analysis) in the peels and peeled potatoes from the <sup>14</sup>C-oxamyl treated plants were 1.02 and 0.78 mg eq/kg, respectively. The TRR in the intact potato was calculated as 0.81 mg eq/kg with the majority (81.1%) of the <sup>14</sup>C-residues in the peeled potatoes. Treated foliage contained 1.25 mg eq/kg.

Treated peels and peeled potatoes were extracted (methanol, 50% aqueous methanol and water) and analysed by HPLC and TLC. TRR in extracted peels (1.11 mg eq/kg) and peeled potatoes (0.86 mg eq/kg) were calculated by adding the extracted and unextracted radioactivity in each sample. The majority (~91%) of the radioactivity was extracted from the peels (1.01 mg eq/kg) and peeled potatoes (0.79 mg eq/kg). The major extractable residue in the peels (68.1% TRR, 0.76 mg eq/kg) and the peeled potato (70.8% TRR, 0.61 mg eq/kg) was <sup>14</sup>C-IN-D2708. No oxamyl or IN-A2213 (oxamyl-oxime) was detected. Other extracted components ranged in concentration from 0.02–0.07 mg eq/kg. The concentration of each of these polar and unknown metabolites would be < 0.04 mg eq/kg in the whole potato (peels plus peeled tuber) and are therefore below the criteria set for metabolite identification.

Unextracted peel and peeled potato residues were treated successively with enzyme (cellulase, pH 5, 37 °C, 96 hr), alkali (0.1N NaOH, 60 °C, 6 hr) and acid (1N HCl, 60 °C, 6 hr). These aqueous extracts each contained < 2% TRR (≤ 0.02 mg eq/kg) and were not analysed further. Matrix-bound residues after exhaustive extraction represented 5.6% (0.06 mg eq/kg) and 6.0% TRR (0.05 mg eq/kg) in the peels and peeled potatoes, respectively.

Treated foliage was also extracted (methanol and 50% aqueous methanol). The majority of the residues were extractable (78.3% TRR, 1.18 mg eq/kg). Minor foliage residues included IN-A2213 (oxamyl-oxime, 5.9% TRR, 0.09 mg eq/kg), oxamyl (1.1% TRR, 0.02 mg eq/kg), and IN-D2708 (1.9% TRR, 0.03 mg eq/kg). The major foliage metabolite (45.7% TRR, 0.69 mg eq/kg) was a water-soluble component that was resistant to enzyme (β-glucosidase) and acid (0.1 M HCl, 90 °C, 6 hours) hydrolysis. This component eluted prior to IN-A2213 and was not present in the potato raw commodity (tuber) fractions. Other foliar metabolites ranged in concentrations of 0.02–0.03 mg eq/kg.

The supplementary studies were conducted to more fully define the nature of the major <sup>14</sup>C-oxamyl derived residue in potato foliage and further substantiate the identity of the IN-QKT34 (IN-A2213 glucoside) isolated from potato plants (Brown *et al.*, 2002 & 2008: DuPont-4520, Supplement No. 1 & 2).

The major foliar metabolite (45.7% TRR, 0.69 mg eq/kg), isolated by successive HPLC and TLC procedures, was resistant to normal glucoside cleavage conditions, enzyme (β- and α-glucosidase) and acid (0.1 N HCl) hydrolysis. HPLC-MS (APCI, positive mode) and high-resolution proton NMR data from the isolated metabolite were consistent with the proposed structure of IN-A2213 glucoside (Supplement No. 1). The chemical structure of IN-A2213 glucoside isolated from <sup>14</sup>C-oxamyl treated potato foliage was confirmed by HPLC-MS/MS and NMR spectroscopy to be identical to the synthetic reference standard IN-QKT34 (Supplement No. 2). The isolate and IN-QKT34 also demonstrated consistent chemical behavior under hydrolytic conditions. Neither IN-QKT34 nor the *in-plant* glucoside were cleaved to any significant extent after 18 hours digestion with hydrolytic enzymes (α- and β-glucosidase) and acid (1N HCl, 60 °C).

Table 1 Nature of the radioactive residues in potatoes

Components	Potato peels		Peeled potatoes		Potato foliage	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Aqueous organic extract	1.01	90.7	0.79	91.0	1.18	78.3

Components	Potato peels		Peeled potatoes		Potato foliage	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Oxamyl	-	-	-	-	0.02	1.1
IN-D2708	0.76	68.1	0.61	70.8	0.03	1.9
IN-A2213	-	-	-	-	0.09	5.9
IN-QKT34	NC	-	NC	-	0.69	45.7
Others	0.08	7.4 (10)	0.07	8.2 (9)	0.02	1.7 (4)
Polar	0.07	6.2	0.05	6.1 (2)	0.20	13.4
Unknowns	0.07	6.4 (2)	-	-	0.09	5.3 (4)
Unextracted	0.10	9.3	0.08	9.0	0.33	21.7
Total hydrolysis <sup>a</sup>	0.04	3.7	0.03	3.0	NC	-
Remaining residues	0.06	5.6	0.05	6.0	0.33	21.7
TRR	1.11	100	0.86	100	1.51	100

NC: Not conducted

<sup>a</sup> Insoluble residues were treated with enzyme (Cellulase), alkali (0.1 N NaOH), and acid (1 N HCl); these fractions each contained < 2% TRR and < 0.02 mg/kg and were not analysed further.

The principal metabolite in the greenhouse-grown potatoes was IN-D2708, a soil degradate and plant metabolite. No oxamyl or IN-A2213 (oxamyl oxime) was detected in the peels or peeled potato; however, minor amounts were observed in the foliage. IN-A2213 is a precursor IN-D2708. IN-N0079 was not detected in either the potato tuber or foliage. The chemical structure of the major metabolite isolated from <sup>14</sup>C-oxamyl treated potato foliage was confirmed by HPLC-MS/MS and NMR spectroscopy to be consistent with the standard IN-QKT34.

#### Tomatoes

The metabolism of oxamyl in tomatoes was investigated with [1-<sup>14</sup>C]-oxamyl, formulated with inert formulation ingredients to simulate a 100 g/L aqueous formulation (Chapleo *et al.*, 2014: Dupont-32188).

This study investigated the metabolism of <sup>14</sup>C-oxamyl following 2 application regimes: (a) multiple foliar and (b) multiple soil applications. The first application occurred immediately after transplant of the tomato plants (*cv.* Red Alert) and was applied at a target application of 2.0 kg ai/ha for both treatment regimes. The 3 subsequent applications for both the foliar and soil experiments were carried out 14 days apart in order to achieve a 21 day PHI; the target rate for each application was 1.0 kg ai/ha. Applications were made to separate groups of plants for each treatment regime.

Samples of immature fruit and foliage were taken at 14DAT3 (14 days after treatment 3; immediately prior to the fourth application; BBCH 74). Ripe fruit and foliage were sampled at 7DAT4 (BBCH 81), 14DAT4 (soil application only; BBCH 81) and 21DAT4 (final harvest, BBCH89). Select tomato fruit and foliage samples were analysed to provide information on oxamyl dissipation and to elucidate the metabolic pathway of oxamyl in tomato plants.

Plant (fruit and foliage) samples receiving the foliar application regime were surface washed in water. The washed samples and samples from the soil application regime were each milled to a powder in dry ice. Portions of milled samples were extracted using methanol followed by methanol/water (1/1, v/v) and water. Extracts were separated from the PES by centrifugation, combined, reduced to incipient dryness under nitrogen and reconstituted in water prior to analysis. Exhaustive extraction of the PES from the 21DAT4 fruit and foliage samples included sequential extractions with water (overnight),  $\alpha$ -amylase (pH 7, 50 °C, 2 × 72 h), a mixture of amyloglucosidase and cellulase (pH 5, 50 °C, 2 × 48 h), NaOH (0.1 N, 60 °C, 2 × 6 h) and HCL (1 N, 60 °C, 2 × 6 h). Levels of radioactivity were determined in each extract by LSC. Terminal unextracted radioactivity in the PES was determined by combustion analysis.

The TRR was determined as the sum of the total extractable (including surface wash, where applicable) residues plus unextracted residues and expressed as mg/kg equivalents of the parent oxamyl. Extracts containing significant radioactivity ( $\geq 0.01$  mg/kg) were analysed by HPLC and

identification of  $^{14}\text{C}$ -residues was accomplished using a variety of contrasting chromatographic systems (HPLC and TLC) with reference to authenticated reference standards.

Table 2 Summary of radioactive residues in tomato fruit following foliar applications of  $^{14}\text{C}$ -oxamyl

Components	14DAT3		7DAT4		21DAT4	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract (Surface wash)	1.38 (0.511)	96.0 (35.6)	0.672 (0.223)	93.9 (31.2)	0.950 (0.013)	95.8 (1.3)
<i>Oxamyl</i>	0.523	36.4	0.223	31.2	0.027	2.9
<i>IN-N0079</i>	0.103	7.2	0.090	12.5	0.088	9.0
<i>IN-QKT34</i>	0.050	3.5	0.033	4.5	0.077	7.7
<i>IN-A2213</i>	0.081	5.7	0.038	5.3	0.096	9.7
<i>IN-F3905</i>	0.114	7.9	0.033	4.6	0.035	3.5
<i>IN-KP532</i>	NC		NC		0.038	3.9
<i>IN-D2708</i>	NC		NC		0.206	20.8
<i>IN-KV998/ IN-T2921</i>	NC		NC		0.007	0.7
<i>Unknowns</i>	0.504	35.2	0.006-0.227	0.8-31.7 (3)	0.001-0.087	0.1-8.8 (13)
<i>NaOH extraction</i>	NC		NC		0.011	1.1
<i>HCl extraction</i>	NC		NC		0.003	0.3
Unextracted	0.057	4.0	0.044	6.2	0.028	2.8
TRR	1.43	100	0.716	100	0.990	100

NC: Not conducted

Table 3 Summary of radioactive residues in tomato foliage following foliar applications of  $^{14}\text{C}$ -oxamyl

Components	14DAT3		7DAT4		21DAT4	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract (Surface wash)	4.49 (1.11)	94.1 (23.2)	9.57 (3.63)	96.8 (36.7)	39.2 (8.74)	98.3 (21.9)
<i>Oxamyl</i>	2.064	43.3	7.219	73.1	31.297	78.4
<i>IN-L2953</i>	-	-	0.081	0.8	-	-
<i>IN-N0079</i>	0.127	2.6	0.393	3.9	0.633	1.5
<i>IN-QKT34</i>	1.673	35.0	1.240	12.6	4.241	10.6
<i>IN-A2213</i>	0.125	2.6	0.193	2.0	0.882	2.2
<i>IN-F3905</i>	0.082	1.7	-	-	-	-
<i>Unknowns</i>	0.025-0.364	0.5-7.6 (3)	0.042-0.396	0.4-4.0 (2)	0.006-1.031	< 0.1-2.6 (9)
<i>NaOH extraction</i>	NC		NC		0.279	0.7
<i>HCl extraction</i>	NC		NC		0.279	0.7
Unextracted	0.277	5.8	0.316	3.2	0.199	0.5
TRR	4.78	100	9.88	100	39.9	100

NC: Not conducted

Table 4 Summary of radioactive residues in tomato fruit following soil applications of  $^{14}\text{C}$ -oxamyl

Components	14DAT3		7DAT4		14DAT4		21DAT4	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	0.661	91.7	0.728	90.4	0.262	79.1	0.626	95.4
<i>Oxamyl</i>	0.028	3.8	0.047	5.9	-	-	-	-
<i>IN-L2953</i>	-	-	0.023	2.9	-	-	-	-
<i>IN-N0079</i>	0.013	1.8	-	-	0.073	21.9	0.015	2.3
<i>IN-QKT34</i>	0.070	9.6	0.068	8.4	0.016	4.8	0.071	10.7
<i>IN-A2213</i>	0.083	11.5	0.089	11.0	0.031	9.3	0.055	8.4
<i>IN-F3905</i>	0.056	7.8	0.058	7.2	0.007	2.2	0.029	4.4
<i>IN-KP532</i>	NC		NC		NC		0.034	5.2
<i>IN-D2708</i>	NC		NC		NC		0.139	21.3
<i>IN-KV998/ IN-T2921</i>	NC		NC		NC		0.003	0.4

Components	14DAT3		7DAT4		14DAT4		21DAT4	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Unknowns	0.022-0.172	3.1-23.9 (4)	0.051-0.228	6.3-28.4 (4)	0.003-0.081	0.8-24.2 (4)	0.001-0.082	0.1-12.4 (9)
NaOH extraction	NC		NC		NC		0.008	1.2
HCl extraction	NC		NC		NC		0.003	0.4
Unextracted	0.060	8.3	0.076	9.5	0.051	15.3	0.020	3.1
TRR	0.721	100	0.805	100	0.332	100	0.655	100

NC: Not conducted

Table 5 Summary of radioactive residues in tomato foliage following soil applications of  $^{14}\text{C}$ -oxamyl

Components	14DAT3		7DAT4		14DAT4		21DAT4	
	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR
Extract	10.6	94.0	5.02	92.0	6.54	91.5	10.7	94.0
Oxamyl	1.527	13.5	1.050	19.3	0.749	10.5	0.730	6.3
IN-L2953	-	-	0.088	1.6	0.250	3.5	-	-
IN-N0079	0.341	3.0	0.154	2.8	-	-	0.499	4.4
IN-QKT34	7.212	63.7	1.918	35.2	3.967	55.6	7.144	62.6
IN-A2213	0.189	1.7	0.416	7.6	0.348	4.9	0.447	3.8
IN-F3905	0.549	4.9	0.176	3.2	0.202	2.8	0.154	1.3
Unknowns	0.073-0.674	0.7-6.0 (3)	0.014-0.986	0.3-18.1 (6)	0.057-0.894	0.8-12.6 (3)	0.005-1.268	< 0.1-11.1 (12)
NaOH extraction	NC		NC		NC		0.251	2.2
HCl extraction	NC		NC		NC		0.251	2.2
Unextracted	0.679	6.0	0.436	8.0	0.614	8.6	0.171	1.5
TRR	11.3	100	5.45	100	7.14	100	11.4	100

NC: Not conducted

Following one 2 kg ai/ha and three 1 kg ai/ha foliar applications of [ $^{14}\text{C}$ ]-oxamyl, TRRs ranged from 0.716–1.43 mg eq/kg in fruit and from 4.78–39.9 mg eq/kg in foliage. Following the same regime of soil applications, fruit TRRs ranged from 0.332–0.805 mg eq/kg and foliage TRRs from 5.45–11.4 mg eq/kg.

Oxamyl concentrations in fruit rapidly declined from 31.2% TRR (0.223 mg/kg), at seven days following four foliar applications (7DAT4) to 2.9% TRR (0.027 mg/kg) at final harvest (21DAT4). Numerous known plant metabolites including IN-A2213, IN-L2953, IN-QKT34, IN-N0079, IN-F3905, IN-D2708, IN-KP532 and IN-KV998/IN-T2920 were detected in fruit and foliage from both treatment regimes. Several (at least three) components more polar than IN-KP532 and IN-D2708 were detected in the fruit and foliage at each time point in each treatment regime.

Efforts to further characterise the unidentified water soluble polar fruit metabolites included chromatographic, deconjugation, hydrolysis, derivatisation and mass spectroscopic techniques, however results were inconclusive. TLC analysis of polar components from an immature fruit sample (14DAT3 foliar regime) indicated the presence of low levels of  $^{14}\text{C}$ -glucose suggesting the possible reincorporation of low levels of radioactivity into plant natural products.

The metabolic pathway of oxamyl in tomato fruit and foliage tissues included hydrolysis of the methylcarbamoyl group to yield the non-insecticidal oxamyloximes (IN-A2213 and IN-F3905). IN-A2213 was conjugated with glucose to yield IN-QKT74. IN-A2213 was demethylated to give IN-L2953. IN-A2213 (or oxamyl) was also metabolised to IN-N0079, which was further metabolised (*via* IN-T2921) to IN-D2708. A similar conversion of IN-L2953 to IN-KP532 (*via* IN-KV998) was observed. Highly polar components possibly resulting from reincorporation (*in part*) of the radiolabel and/or polysaccharide conjugates were also observed.

#### Summary of plant metabolism

Metabolism of  $^{14}\text{C}$  labelled oxamyl has been studied in potatoes and tomatoes, which are suitable to cover the crop groups of root crops and fruiting vegetables. Oxamyl is primarily metabolised in plants



by hydrolysis of the methylcarbamoyl group. The following metabolic pathways were postulated for the available plant metabolism studies.

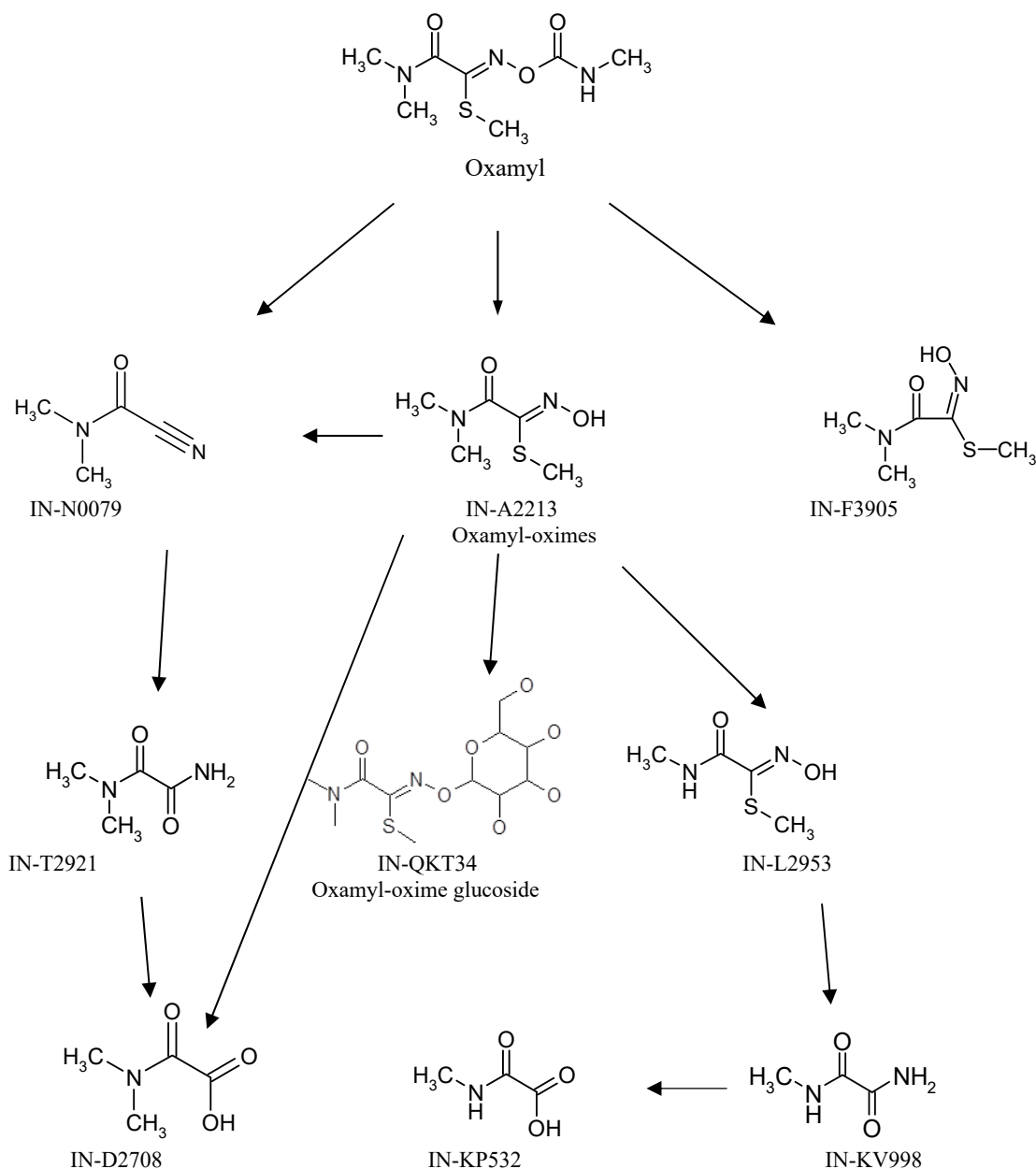


Figure 2 Metabolic Pathway of Oxamyl in Plants (potatoes and tomatoes)

### Farm animal metabolism

The Meeting received studies on the metabolism of oxamyl in lactating goat and laying hens.

#### Lactating goat

The metabolism, excretion and distribution profile of  $^{14}\text{C}$ -oxamyl were studied in the lactating goat (Li, 1994: AMR 2578-92). A capsule containing 59.3 mg  $[1-^{14}\text{C}]$ -oxamyl was administered orally daily to a non-pregnant lactating goat (body weight 37 kg) for five consecutive days. The dose was equivalent to a mean daily dose of 31 ppm in the diet based on the actual average feed consumption of 1.9 kg/day, approximately three times the highest calculated intake. Urine, faeces, cage rinse and milk were collected daily and tissues were collected approximately 21 hours after the last dose. Volatile

gases expired from the goat were monitored daily for radioactivity. Total  $^{14}\text{C}$  residues were determined in milk, urine, faeces, tissues and expired air. There were no apparent toxic effects of the test substance as determined by clinical observations and by measurement of body weight, food consumption and milk production.

Milk samples (0–24 hr, 48–72 hr and 96–120 hr, am/pm sample combined) were sequentially extracted with chloroform and methanol/water (2/1, v/v). Approximately 2–3% of the total radioactivity in milk was found in the chloroform fraction, 67–73% was in methanol/water extract and about 25–30% of the total radioactivity in milk remained in the pellet (unextracted). Most of the radioactivity ( $\geq 90\%$ ) in the unextracted milk pellet was released into supernatant by protease. Oxamyl and IN-A2213 were not detected in any of the fractions ( $\text{LOD} \leq 0.006 \text{ mg eq/kg}$ ). Radioactive thiocyanate was the major component found in the methanol/water extract and in the supernatant of the milk pellet after protease digestion. At least another nine radioactive components were detected in the polar fraction of the methanol/water extract and each of them was less than 10% of the TRR in milk. The total concentrations (following solvent extraction and protease digestion) of radioactive thiocyanate calculated as oxamyl equivalents in milk were: 0–24 hr milk, 0.52 mg/L; 48–72 hr milk, 0.92 mg/L; 96–120 hr milk, 2.0 mg/L.

Liver, kidney, muscle and fat were extracted sequentially by hexane, methylene chloride, ethyl acetate and methanol/water (2/1, v/v). The radioactivity found in hexane was negligible ( $\leq 0.2\%$  TRR,  $\leq 0.01 \text{ mg eq/kg}$ ) in all tissues. Less than 2% of the radioactivity partitioned into the methylene chloride and ethyl acetate fractions. The majority of the extracted radioactivity (30–67%) was found in methanol/water extract. Oxamyl was not detected in any of the organosoluble or aqueous fractions ( $\text{LOD} \leq 0.01 \text{ mg eq/kg}$ ). The chromatographic profiles for all tissue extracts were similar. Radioactive thiocyanate was detected in the methanol/water fraction of all tissues. The concentrations of thiocyanate calculated as oxamyl equivalents were: liver, 0.24 mg/kg; kidney, 0.43 mg/kg; muscle, 0.14 mg/kg; fat, 0.19 mg/kg.

Most of the radioactivity in tissues that was not solvent extracted was released into supernatant by protease digestion. Analysis of the supernatant by HPLC showed that the major radioactive components were more polar than oxamyl and closely related metabolites. Chromatographic characterization showed that the radioactive components were similar in samples from different tissues.

No measured residues of oxamyl were found in any of analysed tissues, urine or milk fractions. Oxamyl was degraded extensively into small molecular weight compounds such as thiocyanate, carbon dioxide and oxamide derivatives found in urine.

Table 6 Recovery of radioactivity in lactating goats following oral administration of [ $^{14}\text{C}$ ]-oxamyl

Sample	% of administered dose (mg/kg oxamyl equivalents)					
	Day 1	Day 2	Day 3	Day 4	Day 5	Total
Urine	6.9 (6.08)	7.2 (4.89)	9.6 (5.75)	10.7 (7.63)	10.3 (7.87)	44.7
Faeces	1.6 (1.49)	1.0 (1.92)	1.5 (2.60)	1.3 (2.67)	1.8 (3.13)	7.2
Cage rinse	0.05 (0.190)	0.09 (0.367)	0.11 (0.302)	0.14 (0.351)	0.17 (0.120)	0.56
Milk	1.12 (1.66)	1.88 (2.84)	1.81 (3.07)	2.46 (3.83)	2.93 (4.63)	10.2
Liver	-	-	-	-	-	1.9 (8.39)
Kidney	-	-	-	-	-	0.2 (4.57)
Muscle	-	-	-	-	-	3.4 (1.29)
Fat	-	-	-	-	-	1.2 (0.642)
Blood	-	-	-	-	-	5.2 (5.90)
Stomach contents	-	-	-	-	-	1.8 (1.66)
Intestinal contents	-	-	-	-	-	1.8 (1.94)
Methanol traps	-	-	-	-	-	< 0.002
Moisture trap	-	-	-	-	-	0.15
NaOH traps	0.52 (0.06)	0.32 (0.04)	0.40 (0.05)	0.33 (0.04)	0.37 (0.04)	1.9
Total	-	-	-	-	-	80.2

Table 7 Distribution of radioactivity in milk of lactating goats

Component	24 hr		72 hr		120 hr	
	mg/L eq <sup>a</sup>	%TRR	mg/L eq <sup>a</sup>	%TRR	mg/L eq <sup>a</sup>	%TRR
Chloroform	0.03	2.17	0.06	1.98	0.09	2.13
Methanol/water	0.97	67.1	1.94	67.6	2.99	72.5
Unextracted	0.45	30.7	0.87	30.5	1.04	25.3
Total	1.45	100	2.87	100	4.12	100

<sup>a</sup> expressed as oxamyl equivalent

Table 8 Composition of <sup>14</sup>C-residue in methanol/water extract in milk of lactating goats

Fraction	24 hr		72 hr		120 hr	
	mg/L eq <sup>a</sup>	%TRR	mg/L eq <sup>a</sup>	%TRR	mg/L eq <sup>a</sup>	%TRR
Methanol/water	0.97	67.1	1.94	67.6	2.99	72.5
Polar Fraction A		58.9		55.2		58.0
Fraction 1 (Thiocyanate)	0.35	23.9	0.65	22.5	1.49	36.1
Fraction 2	0.11	7.30	0.21	7.26	0.15	3.63
Fraction 3	0.02	1.11	0.06	2.23	0.05	1.13
Fraction 4	0.04	2.58	0.06	1.93		
Fraction 5	0.12	8.04	0.16	5.56	0.20	4.93
Fraction 6	0.05	3.46	0.12	4.35	0.11	2.78
Fraction 7	0.04	2.81	0.08	2.92	0.08	1.84
Fraction 8	0.01	0.52	0.02	0.63	0.02	0.46
Fraction 9	0.01	0.55	0.02	0.71	0.02	0.48
Fraction 10	0.02	1.38	0.02	0.63	0.01	0.36
Fraction B					0.02	0.57
Fraction C	0.01	0.38	0.01	0.29	0.01	0.34
Fraction D	0.01	0.38		0	0.03	0.79
Fraction E	0.02	1.24	0.03	0.99	0.05	1.32

<sup>a</sup>: expressed as oxamyl equivalent

Table 9 Composition of <sup>14</sup>C-residue in unextracted pellet in milk of lactating goats

Fraction	24 hr		72 hr		120 hr	
	mg/L eq <sup>a</sup>	%TRR	mg/L eq <sup>a</sup>	%TRR	mg/L eq <sup>a</sup>	%TRR
Unextracted	0.45	30.7	0.87	30.5	1.04	25.3
Supernatant <sup>b</sup>		29.4		27.6		23.3
Polar Fraction		27.3		24.8		21.2
Fraction 1 (Thiocyanate)	0.17	11.5	0.27	9.37	0.51	12.4
Fraction 2	0.01	0.48	0.01	0.48	0.01	0.29
Fraction 3	0.05	3.62	0.11	3.87	0.10	2.48
Fraction 4	0.03	1.85	0.07	2.48	0.05	1.21
Fraction 5	0.04	2.66	0.08	2.65	0.06	1.47
Fraction 6	0.02	1.43	0.02	0.74	0.03	0.65
Fraction 7	0.01	0.65	0.04	1.33	0.03	0.70
Fraction 8	0.03	2.04	0.04	1.51	0.02	0.54
Fraction 9	0.03	1.87	0.03	0.92	0.03	0.64
Lactose	0.07	4.49	0.16	5.50	0.16	3.96

<sup>a</sup>: expressed as oxamyl equivalent

<sup>b</sup> Supernatant contained the released radioactivity from protease digestion of milk pellet.

Table 10 Distribution of radioactivity in tissues of lactating goats

Component	Liver		Kidney		Muscle		Fat	
	mg/kg eq <sup>a</sup>	%TRR	mg/kg eq <sup>a</sup>	%TRR	mg/kg eq <sup>a</sup>	%TRR	mg/kg eq <sup>a</sup>	%TRR
Hexane	0.002	0.02	0.002	0.04	0.001	0.08	0.001	0.20
Methylene chloride	0.02	0.21	0.04	0.87	0.003	0.23	0.005	0.86
Ethyl acetate	0.04	0.45	0.05	1.09	0.01	1.21	0.005	0.74
Methanol/water	2.34	29.7	1.70	36.1	0.63	55.6	0.41	66.9

Component	Liver		Kidney		Muscle		Fat	
	mg/kg eq <sup>a</sup>	%TRR	mg/kg eq <sup>a</sup>	%TRR	mg/kg eq <sup>a</sup>	%TRR	mg/kg eq <sup>a</sup>	%TRR
Unextracted	5.49	69.6	2.91	61.9	0.48	42.9	0.19	31.3
Total	7.89	100	4.70	100	1.13	100	0.61	100

<sup>a</sup> expressed as oxamyl equivalent

Table 11 Composition of <sup>14</sup>C-residue in methanol/water extract in liver of lactating goats

Fraction	mg/kg oxamyl equivalent	% TRR
Methanol/water		42.8
Supernatant		17.6
Polar Fraction		15.4
Fraction A		13.3
Fraction 1 (Thiocyanate)	0.24	2.83
Fraction 2	0.43	5.12
other fractions <sup>a</sup>	0.30	3.59
Fraction B	0.03	0.38
Fraction C	0.06	0.75
Fraction D	0.03	0.32

<sup>a</sup> Aggregate total of several minor metabolite fractions and none of which exceeded 0.9% or 0.07 mg/kg oxamyl equivalent.

Table 12 Composition of <sup>14</sup>C-residue in unextracted pellet in liver of lactating goats

Fraction	mg/kg oxamyl equivalent	% TRR
Unextracted 1 <sup>a</sup>		57.2
Supernatant <sup>b</sup>		43.3
Polar Fraction		37.9
Fraction 1	0.02	0.19
Fraction 2	0.05	0.60
Fraction 3	1.22	14.6
Fraction 4	0.35	4.26
Fraction 5	0.50	5.94
Fraction 6	0.22	2.68
Fraction 7	0.47	5.62
Fraction 8	0.10	1.23
Unextracted 2 <sup>a</sup> (Oxalic acid) <sup>b</sup>	(1.41)	24 (16.9)
Supernatant		24
Polar Fraction		21.9
Fraction 1	0.04	0.47
Fraction 2	0.85	10.2
Fraction 3	0.14	1.64
Fraction 4	0.38	4.57
Fraction 5	0.23	2.73
Fraction 6	0.03	0.35

<sup>a</sup>: Unextracted 1 was the remaining solid after methanol/water extraction.

Unextracted 2 was obtained by acetone precipitation of concentrated methanol/water fraction.

<sup>b</sup>: Base hydrolysis of unextracted 2 released oxalic acid.

Table 13 Composition of <sup>14</sup>C-residue in methanol/water extract in kidney of lactating goats

Fraction	mg/kg oxamyl equivalent	% TRR
Methanol/water		36.1
Supernatant <sup>a</sup>		33.3
Polar Fraction		26.6
Fraction 1 (Thiocyanate)	0.43	9.12
Fraction 2	0.01	0.25
Fraction 3	0.07	1.41
Fraction 4	0.38	8.19

Fraction	mg/kg oxamyl equivalent	% TRR
Fraction 5	0.09	1.98
Fraction 6	0.09	1.84
Fraction 7	0.01	0.25
Fraction 8	0.04	0.92

<sup>a</sup> Supernatant was obtained by acetone precipitation of concentrated methanol/water fraction.

Table 14 Composition of <sup>14</sup>C-residue in unextracted pellet in kidney of lactating goats

Fraction	mg/kg oxamyl equivalent	%TRR
Unextracted <sup>a</sup>	2.91	61.9
Supernatant <sup>b</sup>		57.6
Polar Fraction		47.5
Fraction 1	1.01	21.5
Fraction 2	0.15	3.28
Fraction 3	0.40	8.54
Fraction 4	0.11	2.27
Fraction 5	0.06	1.24
Fraction 6	0.11	2.24
Fraction 7	0.10	2.04

<sup>a</sup> Unextracted was the remaining solid after methanol/water extraction.

<sup>b</sup> Supernatant contained the released radioactivity from protease digestion of unextracted pellet.

Table 15 Composition of <sup>14</sup>C-residue in methanol/water extract in muscle of lactating goats

Fraction	mg/kg oxamyl equivalent	%TRR
Methanol/water		55.6
Supernatant <sup>a</sup>		45.8
Polar Fraction		43.2
Fraction 1 (Thiocyanate)	0.14	12.4
Fraction 2	0.02	1.84
Fraction 3	0.03	2.53
Fraction 4	0.13	11.8
Fraction 5	0.02	1.90
Fraction 6	0.04	3.58
Fraction 7	0.01	1.26
Fraction 8	0.03	2.88

<sup>a</sup>: Supernatant was obtained by acetone precipitation of concentrated methanol/water fraction.

Table 16 Composition of <sup>14</sup>C-residue in unextracted pellet in muscle of lactating goats

Fraction	mg/kg oxamyl equivalent	%TRR
Unextracted <sup>a</sup>	0.49	42.9
Supernatant <sup>b</sup>		39.4
Polar Fraction		24.8
Fraction 1	0.14	12.5
Fraction 2	0.02	1.88
Fraction 3	0.01	0.88
Fraction 4	0.05	4.52
Fraction 5	0.01	0.96
Fraction 6	0.02	1.80
Acid Hydrolysis		33.9
Polar Fraction		30.7
Fraction 1	0.12	10.4
Fraction 2	0.01	0.84
Fraction 3	0.17	15.1
Fraction 4	0.01	1.01
Fraction 5	0.01	0.89
Base Hydrolysis		36.6
Polar Fraction		26

Fraction	mg/kg oxamyl equivalent	%TRR
Fraction 1	0.16	14.4
Fraction 2	0.03	3.06

<sup>a</sup> Unextracted was the remaining solid after methanol/water extraction.

<sup>b</sup> Supernatant contained the released radioactivity from protease digestion of unextracted pellet and this supernatant was further subjected to acid and base hydrolysis.

Table 17 Composition of <sup>14</sup>C-residue in methanol/water extract in fat of lactating goats

Fraction	mg/kg oxamyl equivalent	%TRR
Methanol/water	0.41	66.9
Supernatant <sup>a</sup>		57.0
Polar Fraction		51.0
Fraction 1 (Thiocyanate)	0.19	31
Fraction 2	0.01	1.63
Fraction 3	0.003	0.48
Fraction 4	0.01	1.39
Fraction 5	0.05	7.43
Fraction 6	0.02	2.79
Fraction 7	0.004	0.65

<sup>a</sup> Supernatant was obtained by acetone precipitation of concentrated methanol/water fraction.

Table 18 Composition of <sup>14</sup>C-residue in unextracted pellet in fat of lactating goats

Fraction	mg/kg oxamyl equivalent	%TRR
Unextracted <sup>a</sup>	0.19	31.3
Supernatant <sup>b</sup>		30.6
Polar Fraction		26.5
Fraction 1	0.06	9.94
Fraction 2	0.01	1.71
Fraction 3	0.04	6.52
Fraction 4	0.01	0.96
Fraction 5	< 0.01	0.62
Fraction 6	< 0.01	0.66
Fraction 7	< 0.01	0.70

<sup>a</sup> Unextracted was the remaining solid after solvent extraction.

<sup>b</sup> Supernatant contained the released radioactivity from protease digestion of unextracted pellet.

Chromatographic methods were developed to screen tissues and milk for the reference standards of oxamyl, oxamyl sulfone, oxamyl sulfoxide, oxime, oxime sulfoxide, oxime sulfone, N-methyloxime, N-dimethylcyanoformamide. None of the above compounds were detected in the organosoluble fractions of milk and tissue samples (LOD  $\leq$  0.007 mg eq/kg). The major radioactive components were more polar than oxamyl or closely related metabolites.

Most extracted radioactivity was found in methanol/water fractions. None of the above reference standards were found in muscle, kidney and fat methanol/water extracts (LOD  $\leq$  0.004 mg eq/kg). In liver methanol/water extract, oxamyl and oxamyl sulfone were not detected (LOD  $\leq$  0.01 mg eq/kg). Broad peaks eluted at the retention time of the other standards in this extract and the amount of any individual component must be less than 0.06 mg eq/kg. A broad peak at the retention time of oxime sulfoxide was observed in the milk methanol/water extract, but definitive identification could not be obtained.

Thiocyanate was the major metabolite found in milk and it was also detected in all tissue extracts. Thiocyanate was the major product in milk, not only in the methanol/water extract, but also in the protease digested milk solutions. Lactose was not found in the milk methanol/water extract, but a small amount may have been present in the supernatant of unextracted milk pellet after protease digestion.

About 6.7% (14.9 mg eq/kg) of the administered dose was found in liver, kidney, muscle and fat and approximately 30–70% of this radioactivity was extracted. The most extracted radioactivity was found in methanol/water fractions and thiocyanate was detected in these fractions of all tissue samples. In liver, about half of the extracted radioactivity was precipitated by acetone after concentration and the radioactivity precipitated was released after treatment with protease. Further acid hydrolysis of the supernatant resulted in products different than those obtained from hydrolyzing oxamyl which determines the different chemical nature of these metabolites. Further base hydrolysis of the same supernatant found that the final product was oxalic acid which was also different from those generated by hydrolyzing oxamyl under the same conditions.

Approximately 30–70% of the radioactivity was not extracted with methanol/water in tissues. Most of this radioactivity was released by protease digestion. Chromatographic analysis revealed that the major radioactive components were the same in all tissue residues. However, it has been demonstrated that the products were not closely related to oxamyl by hydrolyzing oxamyl under the same conditions. Derivatization of amino acids in liver did not give evidence for the incorporation of  $^{14}\text{C}$  into amino acids.

### *Rumen fluid*

Rumen fluid experiments were conducted *in vitro* with [ $1\text{-}^{14}\text{C}$ ]-oxamyl, [ $1\text{-}^{14}\text{C}$ ]-IN-N0079, and [ $1\text{-}^{14}\text{C}$ ]-IN-A2213 glucoside (Belasco *et al.*, 1980: AMR-09-80). The rumen fluid (9 flasks, 50 mL per flask) taken from a rumen fistulated Holstein cow was incubated at  $38\pm 0.1^\circ\text{C}$  with nutrients and aqueous solutions of  $^{14}\text{C}$ -oxamyl,  $^{14}\text{C}$ -IN-N0079, and  $^{14}\text{C}$ -IN-A2213 glucoside. Three flasks were incubated with the rumen fluid and 10 mL of an aqueous solution of 0.1%  $^{14}\text{C}$ -oxamyl. Three flasks with 10 mL of an aqueous solution of 0.15%  $^{14}\text{C}$ -IN-N0079 and one flask with the glucose conjugate of  $^{14}\text{C}$ -IN-A2213. Each system was purged with nitrogen (10–20 ml/min) to maintain anaerobic conditions and volatiles ( $^{14}\text{CO}_2$  and radioactive organic compounds) were trapped with 1 N NaOH.

One flask each of the  $^{14}\text{C}$ -oxamyl and  $^{14}\text{C}$ -IN-N0079 treatments was removed at 1, 6, and 24 hours. The single flask with  $^{14}\text{C}$ -IN-A2213 glucoside was incubated for 24 hours. The contents of all flasks were frozen at  $-20^\circ\text{C}$  for brief periods until analysed to prevent further metabolism.

Aliquots (25 mL) from each flask treated with  $^{14}\text{C}$ -oxamyl and  $^{14}\text{C}$ -IN-N0079 were separately centrifuged and the residues were washed twice with 10 mL water. The supernatant and washes from each sample were combined and extracted with ethyl acetate. The extracts were concentrated and analysed by LSC and TLC (silica plates; ethyl acetate). The aqueous fraction (after ethyl acetate extraction) was concentrated and analysed by LSC and TLC (cellulose plates; methanol/acetic acid, 4/1, v/v). Radioactive bands were scraped from the TLC plates, eluted with the respective developing solvent, and submitted to LSC, GC-MS and GC. The washed residues, consisting mainly of microbial cells and solid nutrients, were analysed by combustion and LSC. The NaOH trap solutions were analysed by LSC, then treated with  $\text{BaCl}_2$  solution, filtered, and reanalysed by LSC.

The entire incubation mixture from the flask treated with radiolabeled IN-A2213 glucoside was centrifuged and the residues washed with water. The extracted solids were submitted to combustion and LSC analysis. The supernatant and wash were combined, lyophilised, and the resulting dried residue washed with ethyl acetate, methanol, and water. The ethyl acetate wash was concentrated and analysed by TLC (silica gel plates/ethyl acetate). The methanol wash was purified by gel filtration chromatography (Sephadex LH-20/methanol) and the major radiolabeled peak chromatographed (Porasil A/THF, Permaphase AAX, and Aminex A-6 [ $\text{Ca}^{+2}$ ]) using conditions previously developed for oxamyl metabolite identification. The lyophilisation sublimate was extracted with ethyl acetate and the ethyl acetate phase analysed by LSC and HPLC (Porasil A column with ethyl acetate).

Table 19 Radioactive recoveries from rumen fluid treated with  $^{14}\text{C}$ -oxamyl,  $^{14}\text{C}$ -IN-N0079 and  $^{14}\text{C}$ -IN-A2213 glucoside

Treatment	Incubation time (hours)	Rumen fluid supernatant	% of administered dose		Total
			Rumen fluid solids	Trap solutions	

Oxamyl	0	100	-	-	-
	1	99.5	0.33	0	100
	6	95.6	0.72	0.33	97
	24	83.9	0.79	5.5	90
IN-N0079	0	100	-	-	-
	1	97.6	0	0	98
	6	95.7	1.92	0.96	99
	24	72.1	1.92	12.98	87
IN-A2213 glucoside	0	100	-	-	-
	24	92.5	-	0.62	93

Table 20 The distribution and identification of radioactivity in rumen fluid following incubation with  $^{14}\text{C}$ -oxamyl

Compound	%TRR		
	1 hour	6 hour	24 hour
Oxamyl	58.8	1.2	1.1
IN-A2213	14.0	42.5	66.9
IN-L2953	ND	1.9	1.4
IN-D2708	0.4	0.9	4.6
IN-KP532	0.2	0.2	1.6
IN-N0079	26.6	51.8	12.8
IN-D1409	ND	0.7	1.2
IN-T2921	ND	0.8	10.4
Total	100	100	100

ND: not detected

The total radioactivity of the supernatant solutions from all treatments decreased with time of incubation. This suggested losses due to radiolabelled volatiles and/or the reincorporation of metabolic  $^{14}\text{C}$  into microbial cellular materials.

The major metabolites of oxamyl after 1 hour of incubation were IN-A2213 and IN-N0079, according for 14.0% and 26.6% of the TRR, respectively. Residual oxamyl accounted for 58.8% of the TRR. After 6 hours, the amount of residual oxamyl decreased to only 1.2% of the TRR, while IN-A2213 and IN-N0079, accounted for 42.5% and 51.8% TRR, respectively. At the end of the experimental period (24 hours) the residual oxamyl was present at a level of approximately 1% of the TRR while IN-A2213 and IN-N0079 accounted for 66.9% and 12.8% TRR, respectively. At this point, the concentration of IN-D2708 and IN-T2921 accounted for 4.6% and 10.4% of the TRR, respectively. The minor metabolites, IN-D1409, IN-L2953 and IN-KP532 all increased in concentration over the 24-hour incubation period, but each accounted for only 1–2% of the TRR.

The low specific radioactivity of IN-N0079 used as a substrate in this experiment limited the ability to isolate its metabolites in the complex rumen system. Nevertheless, TLC and radioassay data demonstrated the biodegradation of IN-N0079 to IN-T2921, IN-D2708 and IN-KP532.

Following incubation of  $^{14}\text{C}$ -IN-A2213 glucoside with the rumen fluid, approximately 70% of the radioactivity was converted to IN-N0079, with less than 1% remaining as the original glucoside. The remaining radioactivity was either non-ionic or very weakly acidic and separated into a number of components, none of which were present in large enough quantity for further identification.

#### Laying hens

The metabolism of  $[1-^{14}\text{C}]$ -oxamyl were studied in laying hens (Behmke *et al.*, 1994: AMR 2546-92). White leghorn laying hens were dosed orally for three consecutive days with approximately 3.6 mg  $[1-^{14}\text{C}]$ -oxamyl/day, equivalent to approximately 36.3 ppm in the diet for Group 1 and 42.5 ppm in the diet for Group 2 (different food consumption). Initially, one group of five hens was dosed (Group 1); a second group of ten hens (Group 2) was dosed due to observance of hepatic hemorrhage in two of the Group 1 hens. The dose level for the Group 2 represents approximately 52 times the concentration of oxamyl residues expected in poultry feed.



Excreta and eggs were collected daily and tissues were obtained 20–23 hours after the last dose for both groups. Volatile gases were analysed for Group 1 hens. Only 1.9% of the total administered dose was collected in the volatile traps for Group 1 hens; therefore, volatiles were not analysed for Group 2 hens. Total  $^{14}\text{C}$  residues were determined in eggs, excreta and tissues. For Group 1 hens, composite samples of tissues and eggs were used for analysis; for Group 2 hens, tissues from each of the ten hens were analysed separately ( $n=10$ ). Egg samples were pooled by cage ( $n=5$ ) for Group 2 hens.

For Group 1 hens, no abnormal clinical signs were observed during the dosing phase but, as was mentioned above, at necropsy, a hemorrhage was observed in one of the treated hens and small, dark red foci were observed another treated hen. These livers were separated from the others and were not pooled with the others for analysis. Body weight, food consumption and egg production were not significantly affected by dosing for either group of hens.

Tissues, eggs, and excreta samples were homogenised and total radioactivity determined by LSC following combustion. Tissues from Group 2 hens were sequentially extracted using hexane, methylene chloride, ethyl acetate, and methanol/water. Extracts were analysed using HPLC. Extracted liver was lyophilised and the resulting powder suspended in 0.1 N phosphate buffer (pH 5), then incubated with protease for 24 hours at 37 °C. The mixture was centrifuged and the supernatant analysed by HPLC. Metabolites (0–24 hour excreta) were isolated by preparative HPLC, dissolved in acetonitrile, derivatised with silylating agents and tentatively identified by GC-MS. Thiocyanate was confirmed by precipitation of  $^{14}\text{C}$ -silver thiocyanate.

The total mean recovery of administered radioactivity was 76.2% for Group 1 and 79.0% for Group 2 hens. Group 1 hens excreted a mean of 67.4% of the total dose while Group 2 hens excreted an average of 71.4%. Tissues (muscle, fat, kidney, skin and liver) accounted for a mean of 2.9% and 3.3% of the total dose for Group 1 and Group 2 hens, respectively. Eggs accounted for 1.2% and 0.8% (means) of the total administered dose for Group 1 and Group 2 hens, respectively. The TRRs calculated as oxamyl equivalents for Group 1 hens were: liver: 1.53 mg/kg; kidney: 1.43 mg/kg; breast muscle (light meat): 0.464 mg/kg; thigh muscle (dark meat): 0.590 mg/kg; and fat: 0.035 mg/kg. For Group 2 hens, TRRs expressed as oxamyl equivalents were: liver:  $2.01 \pm 0.30$  mg/kg; kidney:  $1.72 \pm 0.29$  mg/kg; breast muscle:  $0.442 \pm 0.098$  mg/kg; thigh muscle:  $0.675 \pm 0.126$  mg/kg; and fat:  $0.064 \pm 0.030$  mg/kg. Eggs collected following the last dose, 48–72 hour (Day 3) samples, contained 0.771 and 1.05 mg eq/kg in the yolk and white, respectively in Group 1 hens while Group 2 eggs (Day 3) contained  $1.06 \pm 0.17$  mg eq/kg and  $1.16 \pm 0.07$  mg eq/kg, respectively, for the yolk and white.

Table 21 Mean recovery of radioactivity in Group 2 hens dosed with [ $1\text{-}^{14}\text{C}$ ]-oxamyl

Sample	Mean concentration (mg/kg oxamyl equivalents)	Mean recovery (% of administered dose)
Excreta	-	71.4
Liver	2.01	0.7
Kidneys	1.72	0.2
Blood	2.02	2.0
Breast muscle	0.442	0.9
Thigh muscle	0.675	1.3
Skin	0.714	0.1
Fat	0.064	0.1
G. I. Tract	0.933	0.9
G. I. Contents	0.784	0.6
Egg yolk-Day 1	0.105	0.02
Egg yolk-Day 2	0.487	0.06
Egg yolk-Day 3	1.06	0.13
Egg white-Day 1	0.321	0.06
Egg white-Day 2	0.910	0.23
Egg white-Day 3	1.16	0.30

Liver, breast muscle, thigh muscle, egg white and egg yolk samples from Group 2 hens were characterized in terms of extraction into hexane, methylene chloride, ethyl acetate and

methanol/water. In general, most of the radioactivity was extracted into methanol/water for each tissue indicating the presence of more polar metabolites. In fact, only the ethyl acetate extract of liver contained more than 10% of the TRR (24.3%, 0.488 mg/kg oxamyl equivalents) and was equivalent to the percent found in the methanol/water extract (21.6% of the TRR, 0.434 mg/kg oxamyl equivalents).

Isolation of residues was conducted primarily with the methanol/water extracts of tissues, egg samples and excreta (from Group 2 hens only) as these samples contained the greatest concentration of  $^{14}\text{C}$ -residues. HPLC analyses indicated that no oxamyl was present in any tissue sample or in the excreta; nor was there any carbamate-containing  $^{14}\text{C}$  residue that was a known degradate: oxamyl sulfoxide or oxamyl sulfone. The major metabolite in all tissues was identified as thiocyanate. It represented 13.6% of the TRR in liver (0.273 mg/kg oxamyl equivalents or 0.072 mg/kg thiocyanate equivalents); 26.0% of the TRR (0.301 mg/kg oxamyl equivalents and 0.080 mg/kg thiocyanate equivalents) in the 48–72 hour egg white sample; and 33.3% of the TRR (0.353 mg/kg oxamyl equivalents and 0.093 mg/kg thiocyanate equivalents) in the 48–72 hour egg yolk sample. Oxime sulfoxide, oxalic acid, oxamic acid, urea and the anti-isomer of oxamyl oxime were also tentatively identified as minor components in the excreta.

Table 22 Distribution of radioactivity in tissues of Group 2 hens (oxamyl equivalents)

Fraction	Liver		Breast muscle		Thigh muscle	
	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR
Hexane	0.005	0.23	0.001	0.028	< 0.001	0.071
Methylene chloride	0.009	0.43	0.006	1.38	0.014	2.04
Ethyl acetate	0.488	24.3	0.018	4.26	0.029	4.27
Methanol/water	0.434	21.6	0.360	81.6	0.425	63.2
Protease supernatant	0.655	32.6	NA	NA	NA	NA
Unextracted	0.350	17.4	0.057	12.9	0.110	16.3
Total	1.941	96.6	0.442	100.1	0.578	85.9

NA: not applicable

Table 23 Distribution of radioactivity expressed as mg oxamyl equivalents/kg (%TRR) in egg whites and yolks of Group 2 hens

Fraction	Egg white			Egg yolk		
	Day 1	Day 2	Day 3	Day 1	Day 2	Day 3
Hexane	< 0.001 (0.15)	< 0.001 (0.07)	< 0.001 (0.08)	< 0.001 (0.02)	< 0.001 (< 0.001)	0.006 (0.55)
Methylene chloride	0.019 (5.83)	0.042 (4.66)	0.033 (2.81)	0.009 (8.86)	0.026 (5.34)	0.024 (2.25)
Ethyl acetate	0.005 (1.52)	0.027 (2.99)	0.037 (3.15)	0.007 (6.63)	0.019 (3.98)	0.083 (7.85)
Methanol/water	0.462 (114)	0.870 (95.6)	0.976 (84.2)	0.137 (131)	0.261 (53.7)	0.961 (90.7)
Unextracted	0.028 (8.72)	0.069 (7.80)	0.125 (10.8)	0.012 (11.3)	0.058 (11.9)	0.137 (12.9)
Total	0.514 (130)	1.01 (111)	1.17 (101)	0.165 (158)	0.364 (74.9)	1.21 (114)

Table 24 Concentration of thiocyanate in tissue and egg samples

Sample	mg oxamyl equivalents/kg	mg thiocyanate equivalents/kg	%TRR
Liver	0.273	0.072	13.6
Breast muscle	0.018	0.005	4.1
Thigh muscle	0.070	0.019	10.3
Egg yolk-Day 1	0.035	0.009	33.5
Egg yolk-Day 2	0.086	0.023	17.6
Egg yolk-Day 3	0.353	0.093	33.3
Egg white-Day 1	0.149	0.039	46.5

Sample	mg oxamyl equivalents/kg	mg thiocyanate equivalents/kg	%TRR
Egg white-Day 2	0.330	0.087	36.3
Egg white-Day 3	0.301	0.080	26.0

### Summary of animal metabolism

The metabolism of [1-<sup>14</sup>C]-oxamyl has been studied in lactating goat and laying hens. In both studies, oxamyl was degraded extensively into small molecular weight compounds such as thiocyanate, CO<sub>2</sub> and oxamide derivatives found in urine.

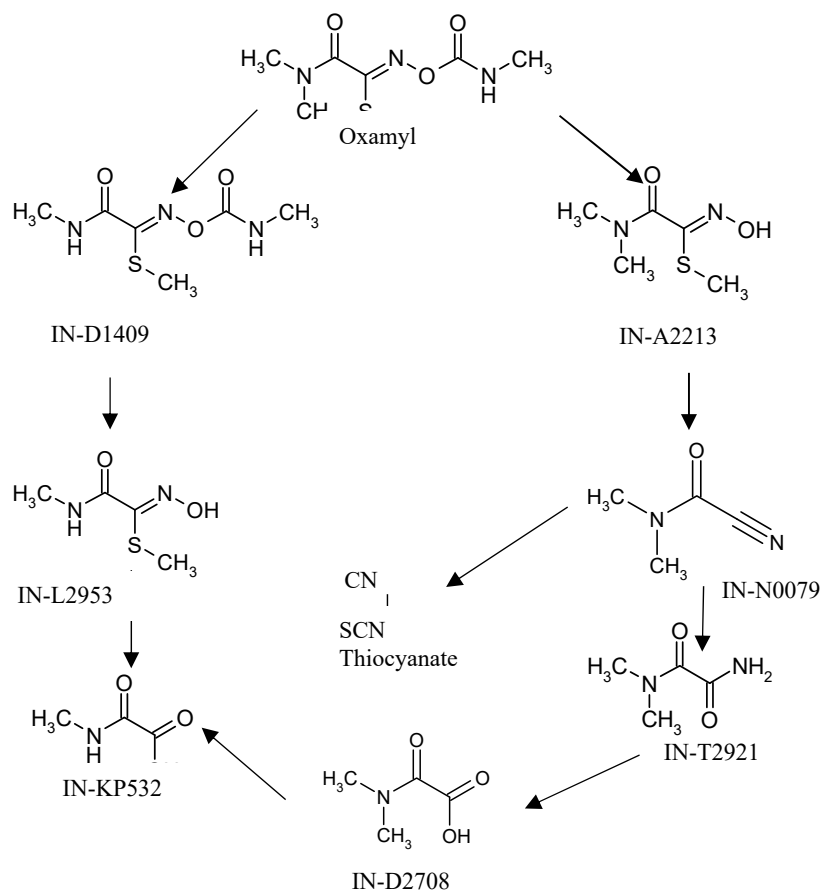


Figure 3 Metabolic Pathway of Oxamyl in Animals (lactating goat, rumen fluid and laying hen)

### Rotational crop studies

#### Confined rotational crop studies

##### Study 1

The study examined the nature and amount of uptake and the potential for accumulation of [1-<sup>14</sup>C]-oxamyl and its soil degradates in barley after a 30-day rotational interval (Brown *et al.*, 2001 & 2002: DuPont-4518 & Supplement No. 1). <sup>14</sup>C-Oxamyl was applied as a single soil application at a rate of 8 kg ai/ha. A solution of <sup>14</sup>C-oxamyl containing inert formulation ingredients to stimulate an SL formulation was applied to sandy loam soil. The pots were maintained in the field under ambient conditions for 30 days, then moved to a greenhouse and planted with spring barley (cv. Harrington). The barley was grown to maturity in the greenhouse.

Soil samples were taken on the day of treatment (Day 0), day of planting (Day 30), and at immature (hay) and mature crop sampling points. Crop samples included barley forage (collected 20 days after planting), hay (collected 63 days after planting) and straw and grain (final harvest, 136 days after planting). At each sampling point the aerial portion of the barley plants (forage, hay and straw) were cut just above the soil surface. At maturity (136 days after planting), the seed heads were removed from the straw using shears. The grain was removed from the chaff by hand. Treated samples, collected at each sampling point, were separately homogenized and TRRs determined by combustion and LSC analysis.

Analysis of soil samples, collected at treatment (Day 0), at planting (Day 30), hay sampling (93 days after treatment) and final harvest (166 days after treatment) showed a steady decline in the concentration of solvent extracted soil residues. Thirty days after treatment, only 32.9% TRR was solvent extracted with 14.8 %TRR (0.10 mg eq/kg) present as oxamyl. At harvest, only 7.0% of the soil residues were extracted (1.0 %TRR, 0.01 mg eq/kg). Other extracted soil residues included IN-D2708 and IN-A2213.

Table 25 Total radioactive residues (TRRs) in aqueous organic soil extracts

	Day 0		Day 30		Day 93		Day 166	
	mg/kg eq*	%TRR	mg/kg eq*	%TRR	mg/kg eq*	%TRR	mg/kg eq*	%TRR
TRR	5.51		0.65		0.35		0.54	
Aqueous organic extracted residues	5.22	94.8	0.21	32.9	0.06	16.2	0.04	7.0
Oxamyl	4.34	78.8	0.10	14.8	0.03	8.8	0.01	1.0
IN-A2213	0.08	1.5	0.02	2.3	< 0.01	0.8	< 0.01	0.5
IN-D2708	0.62	11.2	0.02	3.2	0.01	1.7	0.01	0.8
Others	0.15	2.7	0.06	8.4	0.01	3.8	0.02	4.1

\*Expressed as oxamyl equivalents

The TRRs (combustion analysis) in the treated forage, hay, straw and grain were 7.17, 1.42, 1.79 and 0.26 mg eq/kg, respectively.

Treated barley samples were extracted (methanol, 50% aqueous methanol and water) and analysed by HPLC and/or TLC. Total radioactivity (mg equiv/kg) in extracted samples was calculated by adding the extracted and unextracted radioactivity in each sample. The majority of the radioactivity was extracted from the forage (88.8% TRR, 5.96 mg eq/kg), hay (84.3% TRR, 1.00 mg eq/kg), straw (71.7% TRR, 1.13 mg eq/kg), and grain (60.3% TRR, 0.19 mg eq/kg).

Unextracted hay, straw, and grain residues were treated successively with enzyme (Cellulase, pH 5, 37 °C, hr), alkali (0.1 N NaOH, 60 °C, 6 hr) and acid (1 N HCl, 60 °C, 6 hr). These aqueous extracts each contained ≤ 8% TRR (0.01–0.09 mg eq/kg) and were not analysed further with the exception of straw (enzyme, alkali and acid) and hay (alkali) samples. Matrix bound residues after exhaustive extraction represented 5.5% TRR (0.07 mg eq/kg), 12.5% TRR (0.20 mg eq/kg), 11.2% TRR (0.75 mg eq/kg), and 26.7% TRR (0.09 mg eq/kg) in hay, straw, forage and grain, respectively.

The major extracted residue in the grain (51.3% TRR, 0.16 mg eq/kg) was IN-D2708. No oxamyl or IN-A2213 (oxamyl-oxime) was detected in the grain. Other grain components were present at 4.0% TRR (0.01 mg eq/kg) and were polar.

Table 26 Total radioactive residues (TRRs) in barley grain

Grain			Characterization and identification		
Fraction	mg/kg eq*	%TRR	Analyte	mg/kg eq*	%TRR
Aqueous organic (MeOH/H <sub>2</sub> O) extract	0.19	60.3	<i>IN-D2708</i>	<i>0.16</i>	<i>51.3</i>
			<i>Polar</i>	<i>0.01</i>	<i>4.0</i>
Extract	0.19	60.3	Total characterized/identified	<i>0.17</i>	<i>55.3</i>
Unextracted	0.13	39.7			
Total hydrolysis	0.04	13.0	Enzyme, alkali and acid treatment		
Bound residues	0.09	26.7	Extract pellet analysed by combustion analysis to determine the level of bound radiolabelled residues		

TRR	0.32	100	
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\*Expressed as oxamyl equivalents

Barley forage contained IN-D2708 (3.4% TRR, 0.23 mg eq/kg), IN-A2213 (13.4% TRR, 0.90 mg eq/kg) and oxamyl (24.0% TRR, 1.61 mg eq/kg). Forage components were also tentatively identified on the basis of their chromatographic behavior as IN-KP532 (0.8% TRR, 0.06 mg eq/kg), IN-L2953 (1.4% TRR, 0.09 mg eq/kg) and IN-N0079 (0.6% TRR, 0.04 mg eq/kg). Unknown components were typically present at  $\leq 2\%$  TRR ( $\leq 0.14$  mg eq/kg). However, the major forage component was a polar, water-soluble component (24.4% TRR, 1.64 mg eq/kg) which was also a major component in the barley hay and straw. This metabolite was resistant to enzyme ( $\beta$ -glucosidase) and acid (0.1 M HCl) hydrolysis, eluted prior to IN-A2213 and was not present in barley grain.

Table 27 Total radioactive residues (TRRs) in barley forage

Forage			Characterization and identification		
Fraction	mg/kg eq*	%TRR	Analyte	mg/kg eq*	%TRR
Aqueous organic (MeOH/H <sub>2</sub> O) extract	5.96	88.8	<i>Oxamyl</i>	1.61	24.0
			<i>IN-KP532</i>	0.06	0.8
			<i>IN-D2708</i>	0.23	3.4
			<i>IN-L2953</i>	0.09	1.4
			<i>IN-N0079</i>	0.04	0.6
			<i>IN-A2213</i>	0.90	13.4
			<i>IN-A2213 glucoside</i>	1.64	24.4
			<i>Polar</i>	0.09	1.4
			<i>Unknowns (5)</i>	0.08-0.14	1.1-2.2
Extract	5.96	88.8	Total characterized/identified	0.17	55.3
Unextracted	0.75	11.2			
Total hydrolysis	NC	NC			
Bound residues	0.75	11.2	Extract pellet analysed by combustion analysis to determine the level of bound radiolabelled residues		
TRR	6.71	100			

\*Expressed as oxamyl equivalents

Barley hay contained several components which were identified as IN-D2708 (8.2% TRR, 0.10 mg eq/kg), IN-A2213 (4.6% TRR, 0.06 mg eq/kg) and oxamyl (5.9% TRR, 0.07 mg eq/kg). Hay components were also tentatively identified as IN-KP532 (2.2%TRR, 0.03 mg eq/kg), IN-T2921 (1.7%TRR, 0.02 mg eq/kg), IN-L2953 (6.2% TRR, 0.07 mg eq/kg) and IN-N0079 (2.0% TRR, 0.02 mg eq/kg). Several minor unknown components were present at  $\leq 4\%$  TRR ( $\leq 0.04$  mg eq/kg) each. The major water soluble hay component (40.4% TRR, 0.48 mg eq/kg) was the same as the major forage unknown.

Table 28 Total radioactive residues (TRRs) in barley hay

Hay			Characterization and identification		
Fraction	mg/kg eq*	%TRR	Analyte	mg/kg eq*	%TRR
Aqueous organic (MeOH/H <sub>2</sub> O) extract	1.00	84.3	<i>Oxamyl</i>	0.07	5.9
			<i>IN-KP532</i>	0.03	2.2
			<i>IN-D2708</i>	0.10	8.2
			<i>IN-T2921</i>	0.02	1.7
			<i>IN-L2953</i>	0.07	6.2
			<i>IN-N0079</i>	0.02	2.0
			<i>IN-A2213</i>	0.06	4.6
			<i>IN-A2213 glucoside</i>	0.48	40.4
			<i>Polar</i>	0.04	3.1
			<i>Unknowns (2)</i>	0.02, 0.02	1.3, 1.5
			<i>Others (5 components)</i>	0.04	3.5
Extract	1.00	84.3	Total characterized/identified	0.97	80.6
Unextracted	0.19	15.7			
Total hydrolysis	0.12	10.2	Enzyme, alkali and acid treatment		

Bound residues	0.07	5.5	Extract pellet analysed by combustion analysis to determine the level of bound radiolabelled residues
TRR	1.19	100	

\*Expressed as oxamyl equivalents

Barley straw contained several components which were identified as IN-D2708 (2.9% TRR, 0.05 mg eq/kg), IN-A2213 (6.3% TRR, 0.10 mg eq/kg) and oxamyl (6.0% TRR, 0.09 mg eq/kg). Straw components were also tentatively identified as IN-KP532 (1.0% TRR, 0.02 mg eq/kg), IN-T2921 (1.0% TRR, 0.02 mg eq/kg), and IN-N0079 (13.1% TRR, 0.21 mg eq/kg). Several minor unknown components were present at  $\leq 1\%$  TRR ( $< 0.02$  mg eq/kg), however the major straw component was present at 28.3% TRR (0.45 mg eq/kg), eluted before IN-A2213 and was the major residue seen in forage and hay.

Table 29 Total radioactive residues (TRRs) in barley straw

Straw			Characterization and identification		
Fraction	mg/kg eq*	%TRR	Analyte	mg/kg eq*	%TRR
Aqueous organic (MeOH/H <sub>2</sub> O) extract	1.13	71.7	<i>Oxamyl</i>	0.09	6.0
			<i>IN-KP532</i>	0.02	1.0
			<i>IN-D2708</i>	0.05	2.9
			<i>IN-T2921</i>	0.02	1.0
			<i>IN-N0079</i>	0.21	13.1
			<i>IN-A2213</i>	0.10	6.3
			<i>IN-A2213 glucoside</i>	0.45	28.3
			<i>Polar</i>	0.02	1.2
			<i>Other (1 component)</i>	0.01	0.6
Extract	1.13	71.7	Total characterized/identified	0.97	60.4
Unextracted	0.44	28.3			
Total hydrolysis	0.24	15.7	Enzyme, alkali and acid treatment		
Bound residues	0.20	12.5	Extract pellet analysed by combustion analysis to determine the level of bound radiolabelled residues		
TRR	1.57	100			

\*Expressed as oxamyl equivalents

The major metabolite present in barley forage, hay and straw was a component which eluted prior to IN-A2213 and was resistant to enzyme ( $\beta$ -glucosidase) and acid hydrolysis. To further characterize the major barley foliage metabolite, the barley forage extract was cochromatographed with a potato foliage isolate (from potato plants grown from seed potatoes treated with a single soil application of oxamyl at planting; DuPont-4520) containing IN-A2213 glucoside (Brown *et al.*, 2002: DuPont-4518, Supplement No. 1). The identity of IN-A2213 glucoside in the potato isolate was supported by HPLC-MS and <sup>1</sup>H-NMR spectroscopic data (DuPont-4520, Supplement No 1). Radioanalysis (HPLC and TLC) of the barley forage extract and the potato isolate confirmed that the major barley foliage metabolite was IN-A2213 glucoside.

### Study 2

A sandy loam soil in containers was treated on the surface with [1-<sup>14</sup>C]-oxamyl at 8.96 kg ai/ha, and aged in the greenhouse. Thirty and 120 days after treatment, cabbage (cv. Golden Acre), red beets (cv. Detroit Dark Red) and sorghum (cv. Hybrid G 522 Grain Sorghum) seeds were planted in the containers and the crops grown to maturity. The crops were analysed for total <sup>14</sup>C-residue and, where levels permitted, for oxamyl and oximino compound residue level (Harvey, 1978: O/ME 34).

Both the 30 day and 120 day soil samples were extracted exhaustively with methanol and water, and the extracts analysed by TLC developed with ethyl acetate. At maturity the beet foliage, beet roots, sorghum fodder, sorghum grain and cabbage were harvested and analysed for total radioactivity by combustion and LSC analyses. After exhaustive extraction of the crop with methanol, the combined extracts were concentrated until the concentrate consisted of a clear aqueous phase and a deposit collected on the walls of the concentration flask. The deposit was dissolved by the addition

of hexane equal in volume to that of the aqueous solution. After equilibration the phases were separated and the aqueous phase extracted twice more with hexane and three times with ethyl acetate. Radioactivity in the liquid phases was determined by LSC, and in the unextracted tissue after extraction by combustion. The ethyl acetate soluble fraction from portions of three crops grown on soil aged 30 days contained sufficient radioactivity to permit concentration and analysis by TLC.

In soil, intact  $^{14}\text{C}$ -oxamyl remaining after 30 days amounted to 19% of the applied radioactivity, whereas only a trace (0.3%) was recovered after 120 days ageing. Small amount of IN-A2213 and a polar fraction were present at 30 days but had nearly disappeared after 120 days ageing. Most of the radioactivity was lost from the soil (52% after 30 days; 88% after 120 days), presumably as  $^{14}\text{CO}_2$ .

Table 30 Recovery of radioactivity from soil treated with  $^{14}\text{C}$ -oxamyl

Days after treatment	Recovery of radioactivity (% of applied dose)*				Total
	Oxamyl	IN-A2213	Polar fraction	Unextracted	
30 days	19	1.1	17	11	48
120 days	0.3	0.1	2	9	12

\* Composition based on analysis of the 0-10.3 cm soil segment (88-96% total  $^{14}\text{C}$ )

Crops planted into soil which had been aged 30 days exhibited TRR equivalent to 0.6–4.2 mg/kg. However, the ethyl acetate soluble portion was much lower (0.02–0.47 mg eq/kg) in several instances. Beet leaves (0.47 mg eq/kg) and sorghum fodder (0.18 mg eq/kg) had sufficiently high levels of ethyl acetate extracted radioactivity that TLC analyses could be carried out on the concentrated extracts. A parallel analysis was also carried out on the low level (0.04 mg eq/kg) ethyl acetate extracted from cabbage. In each case the total amount of radioactivity that could be ascribed to oxamyl and/or IN-A2213 was about 25% of the ethyl acetate level. No radioactivity (< 0.5%) was found in that region of the TLC plates that would contain IN-N0079.

Table 31 Distribution and characterization of radioactivity from crops grown in soil treated with  $^{14}\text{C}$ -oxamyl (mg oxamyl equivalents/kg)

Sample	Crops planted 30 days after treatment			Crops planted 120 days after treatment			
	TRR	Ethyl acetate extracted	Oxamyl + IN-A2213	TRR	Ethyl acetate extracted	Oxamyl + IN-A2213	
Beet	leaves	4.2	0.47	0.12	0.15	0.02	NA
	roots	0.6	0.02	NA	0.07	< 0.01	NA
Cabbage	head	0.6	0.04	0.01	0.15	0.01	NA
Sorghum	fodder	2.2	0.18	0.06	0.18	0.02	NA
	seed	1.1	0.02	NA	0.15	0.01	NA

NA = not analysed. Only samples containing greater than 0.02 mg eq/kg were analysed by TLC.

### Study 3

A confined accumulation study of [1- $^{14}\text{C}$ ]-oxamyl has been conducted (Hawkins *et al.*, 1990: AMR 1190-88). [1- $^{14}\text{C}$ ]-oxamyl was applied to the surface of a sandy loam soil contained in containers at a nominal rate of 20.2 kg ai/ha. After nominal ageing periods in a plant growth room of 30, 120 and 363 days, the three rotational crops (lettuce, beetroot and barley) were planted in separate containers and grown to maturity. During growth, the crops were maintained in the growth room with watering. The temperature range was typically 21–25 °C.

Soil samples were collected on the days of treatment (0 day), the time of sowing (30, 120 or 363 days after application), and the time of immature (barley forage) and mature crop harvest. Barley forage samples were also taken and analysed when the barley crops were thinned. Mature plant samples collected at harvest were separated into edible and non-edible fractions. Plants and soil were extracted and the nature of radioactive components investigated. Representative aliquots of each plant fraction were analysed by combustion/LSC to determine the total concentration of radioactive

residues; samples containing significant residues were further analysed to determine the nature of the radioactive components. The chemical nature of the extracted residues was characterised by TLC.

Metabolites were identified by comparison to TLC  $R_f$  values of the reference standards: Oxamyl, IN-A2213, IN-L2953, IN-D2708 and IN-N0079. Plant extracts were also incubated with  $\beta$ -glucosidase (pH 5) and barley straw was submitted to acid hydrolysis (0.1 M HCl/MeOH, 18 hr, 37 °C). Unextracted plant residues were further processed by enzyme (cellulase/hemicellulase, 48 hr, pH 5, 37 °C) treatments.

The concentration of oxamyl in soil declined from 16 mg/kg at zero time to about 0.01 mg/kg at 363 days with a degradation half-life of 34 days. IN-A2213 in soil increased from 0.07 mg eq/kg at zero time to a maximum of 1.3 mg eq/kg at 120 days and then declined with a degradation half-life of 36 days. The TRR in soil was 18 mg eq/kg at the time of application (zero time) and declined with a degradation half-life of 76 days.

Table 32 Characterisation of radioactivity in the soil at various time intervals after application of  $^{14}\text{C}$ -oxamyl

Component	0 day (zero time)		30 days		120 days		363 days	
	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR
Extract	17.6	97.0	11.4	89.5	4.0	57.4	0.02	3.9
Oxamyl	16.0	87.3	8.3	63.9	1.7	24.3	< 0.01	< 0.04
IN-A2213	0.07	0.4	0.87	6.7	1.3	18.5	0.01	1.2
IN-D2708	< 0.16	< 0.9	1.6	12.2	< 0.04	< 0.6	< 0.01	< 0.04
TLC origin	0.68	3.8	< 0.12	< 0.9	0.20	2.8	< 0.01	< 0.04
7 unknowns	< 0.16	< 0.9	< 0.12	< 0.9	< 0.04-0.28	< 0.6-3.9	< 0.01-0.01	< 0.04-1.5
Others	0.85	4.7	0.68	5.2	0.19	2.7	< 0.01	0.2
Unextracted	0.58	3.2	1.4	10.5	3.2	43.9	0.62	96.1
TRR	18	100	13	100	7.2	100	0.64	100

\* Expressed as oxamyl equivalents

TRR for crops sown 30 days after application ranged from 3.1 mg eq/kg in lettuce to 38 mg eq/kg in barley straw. TRR for crops sown 120 days after application ranged from 0.27 mg eq/kg in lettuce to 6.8 mg eq/kg in mature beet foliage. TRR for crops sown 363 days after application ranged from 0.03 mg eq/kg in lettuce to 0.29 mg eq/kg in barley straw.

Table 33 Concentration of radioactivity in barley, beet and lettuce after sown at different intervals in soil treated with  $^{14}\text{C}$ -oxamyl (mg oxamyl equivalents/kg)

Sample		30 days		120 days		363 days	
		Treated	Control	Treated	Control	Treated	Control
Barley	Forage	21	1.3	1.7	< 0.09	0.05	< 0.02
	Straw	38	2.9	5.2	0.73	0.29	0.10
	Chaff	24	3.7	3.6	0.62	0.19	0.10
	Grain	7.2	3.0	1.3	0.37	0.11	0.06
Beetroot	Foliage	24	0.5	6.8	0.08	0.08	< 0.04
	Root	7.1	2.0*	0.86	0.13	0.04	< 0.04
	Peel	14		2.3	0.16	0.24	< 0.07
Lettuce		3.1	0.50	0.27	0.11	0.03	< 0.03

\* Insufficient sample for individual analysis of root and peel

The concentration of oxamyl was highest in crops grown in 30 days aged soil. In crops grown in 120 days aged soil oxamyl was either not detected (beetroot and lettuce) or had concentrations at least 10-fold lower (barley). In crops grown in soil aged 120 days, IN-A2213 concentrations had either decreased about 10-fold in barley (forage, straw and chaff) and beetroot (root) or was not detected.

Oxamyl and IN-A2213 were significant residues in barley forage from both the 30-day and the 120-day soil ageing intervals. These two components were present at much lower levels in the



straw and chaff, and were not detectable in barley grain. IN-D2708, a plant and soil degradate of oxamyl, was only present at low levels (< 1% TRR). The remainder of the radioactive residues in the barley RACs was designated as polar unknowns. Three polar unknowns were significant components of the TRR either approaching or exceeding 10% TRR in the barley straw and chaff from the 30-day and the 120-day soil ageing intervals.

Table 34 Distribution of radioactivity in barley sown 30 and 120 days after application of <sup>14</sup>C-oxamyl

Components	30 days after application							
	Forage		Straw		Chaff		Grain	
	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR
Extract	18.8	85.1	30.9	81.1	15.2	63.2	3.0	42.9
<i>Oxamyl</i>	12.0	57.6	5.9	15.5	1.5	6.1	< 0.03	< 0.4
<i>IN-A2213</i>	2.2	10.7	0.99	2.6	0.89	3.7	< 0.03	< 0.4
<i>IN-D2708</i>	< 0.19	< 0.9	< 0.30	< 0.8	< 0.14	< 0.6	< 0.03	< 0.4
<i>TLC origin</i>	3.5	11.8	< 0.30	< 0.8	< 0.14	< 0.6	2.5	35.2
7 unknowns	< 0.19-1.1	< 0.9-5.0	< 0.30-12.3	< 0.8-32.4	< 0.14-5.5	< 0.6-22.9	< 0.03-0.17	< 0.4-2.4
<i>Others</i>	< 0.19	< 0.9	5.1	13.4	1.2	4.8	0.24	3.4
Enzyme treatment	NC	NC	NC	2.4	NC	2.4	NC	5.8
Unextracted	3.1	14.8	7.2+	16.6	8.9+	34.5	4.1+	51.3
TRR	21	100	38	100	24	100	7.2	100
Components	120 days after application							
	Forage		Straw		Chaff		Grain	
	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR
Extract	1.7	100	2.1	40.6	1.9	53.9	0.8	60.6
<i>Oxamyl</i>	0.53	31.0	0.16	3.1	0.08	2.1	< 0.01	< 0.6
<i>IN-A2213</i>	0.30	17.6	0.08	1.5	0.08	2.3	< 0.01	< 0.6
<i>IN-D2708</i>	< 0.02	< 1.0	< 0.02	< 0.4	< 0.02	< 0.5	< 0.01	< 0.6
<i>TLC origin</i>	< 0.02	< 1.0	< 0.02	< 0.4	< 0.02	< 0.5	< 0.01	< 0.6
7 unknowns	< 0.02-0.87	< 1.0-51.4	< 0.02-0.82	< 0.4-15.8	< 0.02-1.2	< 0.5-34.5	< 0.01-0.76	< 0.6-58.4
<i>Others</i>	< 0.02	< 1.0	0.22	4.3	0.08	2.2	0.03	2.2
Unextracted	< 0.02	< 1.0	3.1	59.4	1.7	46.3	0.51	39.3
TRR	1.7	100	5.2	100	3.6	100	1.3	100

\* Expressed as oxamyl equivalents

+ Included aqueous after enzyme treatment

NC = not conducted

Oxamyl and IN-A2213 were detected in beet root and foliage, and lettuce, at the 30-day soil ageing interval, but in general never exceeded 11% TRR. After soil ageing for 120 days, IN-A2213 was only detected (4.3% TRR) in beet roots; no oxamyl was detected (< 1% TRR) in any of the beet RAC. Neither oxamyl nor IN-A2213 was detected in lettuce from the 120-day soil ageing interval.

Table 35 Distribution of radioactivity in beet and lettuce sown 30 and 120 days after application of <sup>14</sup>C-oxamyl

Components	30 days after application							
	Beet foliage		Beet root		Beet root peel		Lettuce	
	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR
Extract	22.8	97.4	5.9	82.2	11.4	81.7	2.0	63.5
<i>Oxamyl</i>	2.5	10.6	0.13	1.8	< 0.11	< 0.8	0.18	5.7
<i>IN-A2213</i>	1.4	5.7	0.33	4.6	0.29	2.1	0.12	3.8
<i>IN-D2708</i>	< 0.24	< 1	< 0.06	< 0.8	< 0.11	< 0.8	< 0.02	< 0.6
<i>TLC origin</i>	2.0	8.5	< 0.06	< 0.8	0.91	6.5	0.26	8.4
7 unknowns	< 0.24-11	< 1-45.2	< 0.06-2.7	< 0.8-37.9	< 0.11-5.4	< 0.8-38.8	< 0.02-0.73	< 0.6-23.5
<i>Others</i>	0.43	1.8	< 0.06	< 0.8	< 0.11	< 0.8	< 0.02	< 0.6
Enzyme treatment	NC	NC	NC	6.3	NC	2.7	NC	1.0
Unextracted	0.91	3.8	1.2+	11.5	2.9+	17.7	1.1+	35.7

Components	30 days after application							
	Beet foliage		Beet root		Beet root peel		Lettuce	
	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR
TRR	24	100	7.1	100	14	102	3.1	100
Components	120 days after application							
	Beet foliage		Beet root		Beet root peel		Lettuce	
	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR	mg/kg eq*	% TRR
Extract	6.0	88.1	0.61	70.9	0.7	30.9	0.2	93.3
<i>Oxamyl</i>	< 0.06	< 0.9	< 0.01	< 0.1	< 0.01	< 0.3	< 0.01	< 0.9
<i>IN-A2213</i>	< 0.06	< 0.9	0.04	4.3	< 0.01	< 0.3	< 0.01	< 0.9
<i>IN-D2708</i>	< 0.06	< 0.9	} 0.57 }	} 66.6 }	< 0.01	< 0.3	< 0.01	< 0.9
<i>TLC origin</i>	< 0.06	< 0.9			< 0.01	< 0.3	< 0.01	< 0.9
<i>7 unknowns</i>	< 0.06-4.3	< 0.9-63.2			< 0.01-0.36	< 0.3-15.8	< 0.01-0.22	< 0.9-81.7
<i>Others</i>	0.27	4.0	NC	< 0.1	< 0.01	< 0.3	< 0.01	< 0.9
Enzyme treatment	NC	NC	NC	<11.6	NC	5.8	NC	<6.7
Unextracted	0.82	12.1	0.25+	29.1	1.6+	63.5	0.02+	6.7
TRR	6.8	100	0.86	100	2.3	100	0.27	100

\* Expressed as oxamyl equivalents

+ Included aqueous after enzyme treatment

NC = not conducted

A large proportion of the radioactivity grown in soil aged 363 days has been attributed to uptake of  $^{14}\text{CO}_2$  liberated by mineralization of  $^{14}\text{C}$ -oxamyl in the soil. A substantial proportion of the radioactive components in these samples was likely to be derived from uptake of  $^{14}\text{CO}_2$ . As concentration of radioactivity were low in the barley grown in 363 day aged soil and most of the components in samples taken at earlier times were polar or unextracted, the residues in barley were not investigated further.

The beet root and lettuce grown in 363 day aged soil was not investigated further as concentrations of radioactivity were low and most radioactivity in earlier samples was polar or unextracted.

#### *Summary of metabolism in rotational crops*

The major barley foliage metabolite was IN-A2213 glucoside (IN-QKT34). Oxamyl, IN-A2213, IN-N0079 and IN-D2708 were also present in the barley foliage. The nature of the residue derived from  $^{14}\text{C}$ -oxamyl in barley foliage (planted and grown 30 days after soil treatment) is consistent with plant and animal (livestock and rat) metabolism studies.

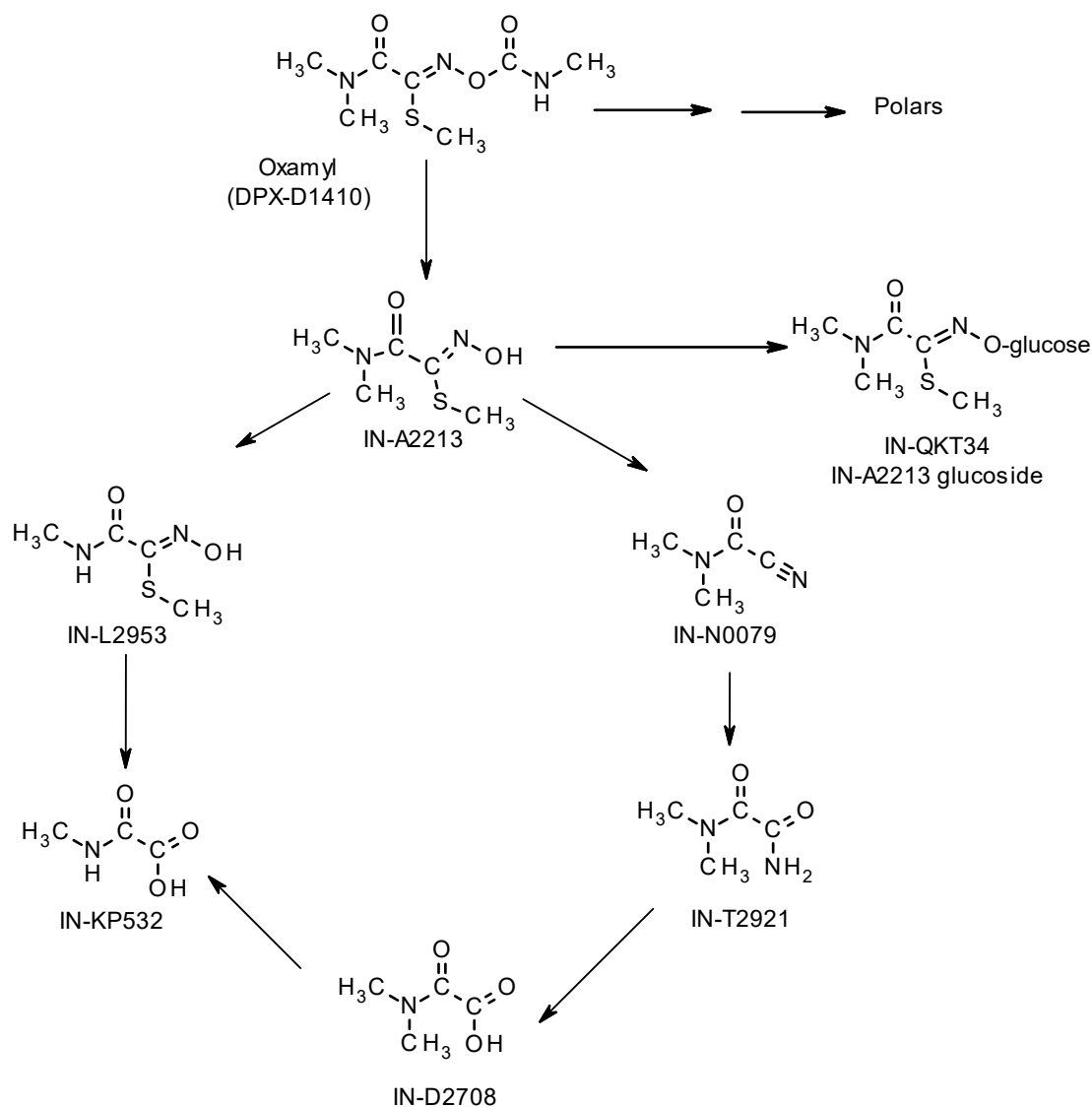


Figure 4 Metabolic Pathway of Oxamyl in rotational crops following soil application

### Field rotational crop studies

#### Study 1

The study was conducted to determine the magnitude of residues of oxamyl in succeeding crops (leafy vegetables, root crops and cereals) planted in fields in northern Europe previously treated with oxamyl (Anderson *et al.*, 2007; DuPont-16669). The 100 g/kg oxamyl GR formulation was applied by granular applicator at planting of potatoes (growth stage BBCH 03) to treated plot. Each application was at a target rate of 55 kg formulated product/ha, for a target application rate of 5.5 kg ai/ha. For all trials, potatoes were removed at 80 or 120 days after the application to allow planting of succeeding crops (lettuce, carrot, winter barley and winter wheat) at the targeted plantback intervals (PBIs).

Field specimens of the succeeding crops were collected at maturity (lettuce, carrots – roots and tops, cereal straw and cereal grain). In addition, a specimen of cereal hay was collected from each plot at the appropriate growth stage. One control specimen and one treated specimen per commodity and sampling interval were submitted for analysis.

Specimens were analysed for residues of oxamyl by HPLC-MS following procedures described in analytical method No. 0259 which was validated by DuPont-11125. The determined LOQ in crop matrices was 0.010 mg/kg. The LOD was 0.007 mg/kg.

The mean recoveries per fortification level were 73% (0.01 mg/kg, n=2) and 79% (0.1 mg/kg, n=2) for lettuce, 72±13% (0.01 mg/kg, n=6) and 79 ± 3% (0.1 mg/kg, n=6) for carrot root, 83 ± 12% (0.01 mg/kg, n=4) and 79 ± 6% (0.1 mg/kg, n=4) for carrot tops, 109% (0.01 mg/kg, n=2) and 97% (0.1 mg/kg, n=2) for cereal grain, 102% (0.01 mg/kg, n=2) and 94% (0.1 mg/kg, n=2) for cereal hay, and 91% (0.01 mg/kg, n=2) and 81% (0.1 mg/kg, n=2) for cereal straw.

Treated lettuce, carrot roots and tops, cereal grain, hay, and straw samples were stored at -18 ± 5 °C for less than 18 months between sampling and analysis.

Table 36 Residues of oxamyl in succeeding crop commodities

Country, year Location, Region	Application		Succeeding crop		DAP <sup>a</sup> Days	PBI <sup>b</sup> Days	Residues, mg/kg			
	Form	kg ai/ha	Crop (variety)	Portion analysed						
UK, 2006 Aberlady, East Lothan	GR	5.3	Lettuce, head (Diamond)	Heads	39 47	81 120	< 0.01 (<LOD) < 0.01 (<LOD)			
				Carrot (Nairobi F1)	Root	86 138	81 120	< 0.01 (<LOD) < 0.01 (<LOD)		
			Tops		86 128	81 120	< 0.01 (<LOD) < 0.01 (<LOD)			
			UK, 2006 Ramsey, Harwich, Essex	GR	5.3	Lettuce, leaf (Majesty)	Leaves	90 50	80 120	< 0.01 (<LOD) < 0.01 (<LOD)
Carrot (Bangor F1)	Root	90 114					80 120	< 0.01 (<LOD) < 0.01 (<LOD)		
	Tops	90 114				80 120	< 0.01 (<LOD) < 0.01 (<LOD)			
UK, 2006 Monikie, Dundee, Angus	GR	4.9				Winter barley (Siberia)	Hay	305 226	80 119	< 0.01 (<LOD) < 0.01 (<LOD)
			Grain	349 310	80 119		< 0.01 (<LOD) < 0.01 (<LOD)			
				Straw	349 310		80 119	< 0.01 (<LOD) < 0.01 (<LOD)		
			UK, 2006 Ramsey, Harwich, Essex	GR	5.5	Winter wheat (Einstein)	Hay	299 259	80 120	< 0.01 (<LOD) < 0.01 (<LOD)
							Grain	324 284	80 120	< 0.01 (<LOD) < 0.01 (<LOD)
								Starw	324 284	80 120

<sup>a</sup> Days after planting: number of days between sowing and harvesting of succeeding crop commodities

<sup>b</sup> Plantback interval: number of days between last application to treated crop and sowing of succeeding crops

Oxamyl residues in succeeding crops (lettuce, carrot roots and tops, and cereal grain, hay, and straw) planted at 80 and 120 days after application of oxamyl at a nominal rate of 5.5 kg ai/ha and harvested at maturity were <LOD (0.007 mg/kg).

### Study 2

The study was conducted to determine the magnitude of residues of oxamyl in lettuces and radish which were planted in protected situations in Southern Europe after the harvest of melons which had previously been treated with oxamyl (Old *et al.*, 2009: DuPont-16693). The 100 g/L oxamyl SL formulation was applied by simulated drip irrigation immediately after transplanting melons and four additional times with the last application being 21 days before melon harvest. Applications were made on a 10-day retreatment interval. First application was made at a target rate of 20 L formulated product/ha, for a target application rate of 2 kg ai/ha and other applications were made at a target rate of 10 L formulated product/ha, for a target application rate of 1 kg ai/ha. For all trials, melons were

removed after the final application to allow the succeeding crops to be planted at the targeted PBIs of 30, 60, 90 or 120 days. The succeeding crops were lettuce and radish grown under protected conditions.

Specimens of peel and flesh of melons were collected at 21 and 28 DALA. Two control specimens and two treated specimens collected 21 DALA and two treated specimens collected 28 DALA were submitted for analysis. Field specimens of the succeeding crops (lettuce and radish) were collected at maturity for each PBI. Radishes were separated into tops and roots. Control and treated specimens were collected at 30 and 90 day PBI and treated specimens at 60 and 120 day PBI.

Specimens were analysed for residues of oxamyl following procedures described in analytical method No. 0259 which was validated by DuPont-11125. The determined LOQ in radish roots, radish tops and lettuce heads was 0.010 mg/kg with a LOD of 0.007 mg/kg in these crops. For melon flesh and melon peel the LOQ in was 0.0050 mg/kg and the LOD was 0.003 mg/kg.

The mean recoveries per fortification level were 90% (0.005 mg/kg) and 88% (0.1 mg/kg) for melon peel, 90% (0.005 mg/kg) and 88% (0.1 mg/kg) for melon flesh, 93% (0.01 mg/kg, n=2) and 66% (0.1 mg/kg, n=2) for lettuce heads, 65% (0.01 mg/kg, n=2) and 82% (0.1 mg/kg, n=2) for radish root, and 86% (0.01 mg/kg, n=2) and 88% (0.1 mg/kg, n=2) for radish tops.

Treated lettuce, radish roots, radish tops, melon flesh and melon peel samples were stored at  $-18 \pm 5$  °C for less than 12 months between sampling and analysis.

Table 37 Residues of oxamyl in succeeding crop commodities

Country, year Location, Region	Application		Succeeding crop		DAP <sup>a</sup> Days	PBI <sup>b</sup> Days	Residues, mg/kg
	Form	kg ai/ha	Crop (variety)	Portion analysed			
Spain, 2007 Los Palacios, Andalucia	SL	6.0	Lettuce (Filipu)	Heads	63	30	< 0.01 (<LOD)
				Heads	52	60	< 0.01 (<LOD)
				Heads	43	93	< 0.01 (<LOD)
				Heads	45	120	< 0.01 (<LOD)
			Radish (Largo Comun)	Root	63	30	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	
				Root	52	60	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	
				Root	43	93	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	
				Root	45	120	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	
Italy, 2007 Roncoferraro, Lombardia	SL	6.0	Lettuce (Justine)	Heads	38	30	< 0.01 (<LOD)
				Heads	39	60	< 0.01 (<LOD)
				Heads	45	92	< 0.01 (<LOD)
				Heads	49	124	< 0.01 (<LOD)
			Radish (National)	Root	58	30	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	
				Root	53	60	< 0.01 (<LOD)
					Tops	Not sampled	
				Root	45	92	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	
				Root	49	124	< 0.01 (<LOD)
					Tops	< 0.01 (<LOD)	

<sup>a</sup> Days after planting: number of days between sowing and harvesting of succeeding crop commodities

<sup>b</sup> Plantback interval: number of days between last application to treated crop and sowing of succeeding crops

Oxamyl residues in succeeding crops (lettuce and radish roots and radish tops) planted at 30, 60, 90 and 120 days after application at a nominal rate of 6.0 kg ai/ha and harvested at maturity were <LOD (0.007 mg/kg). For primary melon crop; melon flesh oxamyl residues of 0.037 mg/kg and 0.026 mg/kg were detected in the 21 DALA and 28 DALA treated samples, respectively. For melon

peel, oxamyl residues of 0.061 mg/kg and 0.027 mg/kg were detected in the 21 DALA and 28 DALA treated samples, respectively. Calculated whole melon residues from the primary melon crop were 0.047 and 0.026 mg/kg in the 21 DALA and 28 DALA treated samples, respectively.

### *Environmental fate in soil*

The Meeting received information on degradation in aerobic and anaerobic soil, soil photolysis, mobility, adsorption/desorption of metabolites and field dissipation studies. Because oxamyl is intended for use as soil treatment, soil degradation (aerobic), soil photolysis and field dissipation studies relevant to the current evaluations were reported below (FAO Manual Third edition, 2016).

### *Degradation in aerobic soil*

The degradation of [ $1-^{14}\text{C}$ ]-oxamyl in soils with varying chemical and physical properties was studied under aerobic condition. [ $1-^{14}\text{C}$ ]-oxamyl was applied at a rate of 2 mg/kg (dry soil basis) to viable agricultural soils, and its aerobic degradation was monitored. The soils were maintained at 20 °C and 40–50% of 0-bar moisture throughout the incubation period of up to 123 days. Volatilized radioactivity was trapped in ethylene glycol and potassium hydroxide solution. Test soil characteristics are detailed below (Smyser, 2000: DuPont-2957 and DuPont-2958).

Test soil	Commerce	Gross Umstadt	Drummer #6	Nijmegen
Origin location	Greenville, MS, USA	Gross Umstadt, Germany	Rochelle, IL, USA	Nijmegen, The Netherlands
Texture (USDA)	Silt loam	Silt loam	Silty clay loam	Loam
% Sand (2000 - 50 $\mu\text{m}$ )	32.8	5.6	8.4	45.2
% Silt (<50 - 2 $\mu\text{m}$ )	56.4	77.2	60.8	40.8
% Clay (<2 $\mu\text{m}$ )	10.8	17.2	30.8	14.0
pH	7.0	7.8	4.8	7.0
Organic matter (%)	0.4	2.1	4.4	2.4
Cation exchange capacity	6.7	9.6	26.3	10.1
Maximum water-holding capacity (%)	33.3	50.0	49.4	33.3
Microbial biomass (mg C/100 g soil)				
Initial:	5.31	30.73	12.3	18.71
Final:	4.76	29.7	7.33	13.45

The test soils were treated with [ $1-^{14}\text{C}$ ]-oxamyl at a mean concentration of 5.0 mg ai/kg dry weight soil and incubated in darkness at approximately  $20 \pm 2$  °C. The samples were incubated under aerobic conditions in flow-through systems designed to maintain soil moisture at 100% of its 0.1-bar moisture and to trap evolved  $\text{CO}_2$  and volatile organic compounds. The soil characteristics are listed below (Clark, 2015: DuPont-39014).

Test soil	Speyer	Tama	LRA-D	Goch
Origin location	Hanhofen, Germany	Stark County, IL, USA	Derbyshire, England	Northrhine-Westfalia, Germany
Texture <sup>a</sup>	Loamy sand	Light clay	Sandy loam	Sandy loam
% Sand (2000-50 $\mu\text{m}$ )	87	31	79	70
% Silt (<50-2 $\mu\text{m}$ )	8	36	14	21

% Clay (<2 µm)	5	33	7	9
pH (1:2 soil:0.01M CaCl <sub>2</sub> )	6.1	6.7	5.4	5.7
Organic matter (%)	2.7	5.4	5.8	3.5
Cation exchange capacity	6.8	20.9	10.5	8.8
Maximum water-holding capacity (%)	45.4	86.5	57.1	54.2
Microbial biomass (µg C/g soil)				
Initial:	425.4	723.1	613.9	553.3
Final:	328.4	448.8	426.7	264.6

<sup>a</sup> International soil classification system

At each sampling point, soils were extracted with a variety of organic solvents and the extracts were profiled by HPLC for oxamyl and degradation products. Following extraction, the soil pellets were combusted to quantify unextracted, bound residues. Volatile traps were also analysed for identification of possible small organic metabolites and to quantify <sup>14</sup>CO<sub>2</sub> resulting from mineralization.

There were three significant degradation products - IN-A2213, IN-D2708 and <sup>14</sup>CO<sub>2</sub>. No other metabolite was observed at >3.8% of the applied radioactivity. The <sup>14</sup>CO<sub>2</sub>, found in the caustic trap, was the final and most significant degradate. Essentially all the applied radioactivity was converted to <sup>14</sup>CO<sub>2</sub> by the end of the study. A DT<sub>50</sub> (half-life) and DT<sub>90</sub> for oxamyl, IN-A2213 and IN-D2708 in all soils were determined using a nonlinear regression of first-order kinetic equations. IN-D2708 was not present in the Drummer soil.

Table 38 DT<sub>50</sub> and DT<sub>90</sub> for oxamyl, IN-A2213 and IN-D2708 in soils (days)

Soil	Oxamyl		IN-A2213		IN-D2708	
	DT <sub>50</sub>	DT <sub>90</sub>	DT <sub>50</sub>	DT <sub>90</sub>	DT <sub>50</sub>	DT <sub>90</sub>
Commerce (Silt loam)	3.0	9.9	5.9	19.7	3.6	12.1
Gross Umstadt (Silt loam)	4.1	13.6	1.7	5.7	3.4	11.2
Drummer #6 (Silty clay loam)	112	373	17.5	58.2	-	-
Nijmegen (Loam)	8	26	2	6	8	25
Speyer (Loamy sand)	7.23	24.0	NC	NC	NC	NC
Tama (Light clay)	9.76	32.4	NC	NC	NC	NC
LRA-D (Sandy loam)	12	40.2	NC	NC	NC	NC
Goch (Sandy loam)	<4.0	NA	NC	NC	NC	NC

NA: Due to rapid degradation, enough data points were not available to produce robust kinetic fits.

NC: not calculated

Maximum levels of the major soil metabolites (IN-A2213 and IN-D2708) are listed in Table 39.

Table 39 Maximum levels of IN-A2213 and IN-D2708 in soils

Soil	IN-A2213		IN-D2708	
	% Applied radioactivity	Days	% Applied radioactivity	Days
Commerce (Silt loam)	51.0	7	25.7	11
Gross Umstadt (Silt loam)	24.9	2	34.7	10
Drummer #6 (Silty clay loam)	7.6	60	ND	-
Nijmegen (Loam)	13.4	7	29.6	14
Speyer (Loamy sand)	9.1	4	55.5	28
Tama (Light clay)	13.5	4	25.2	14
LRA-D (Sandy loam)	5.8	7	27.4	28
Goch (Sandy loam)	5.0	4	78.0	7

## Oxamyl

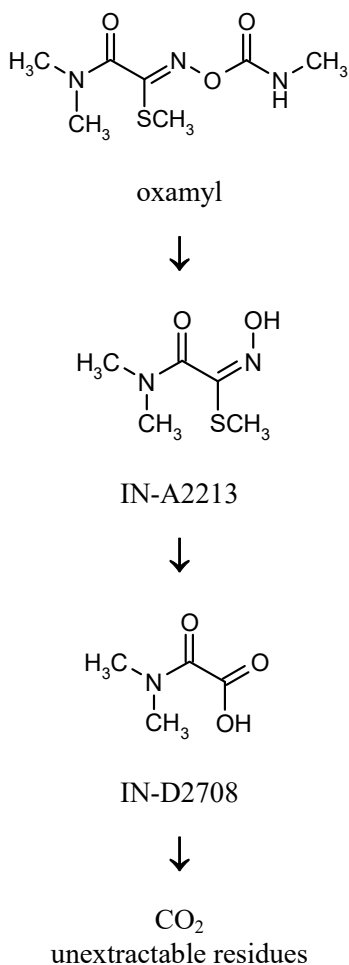


Figure 5 Metabolic Pathway of Oxamyl in aerobic soil

*Soil photolysis*

The photodegradation of [1-<sup>14</sup>C]-oxamyl on non-sterile silty clay loam soil was investigated (Habeeb, 2011: DuPont-31501). Thin layers of soil (2 mm) were treated with oxamyl to obtain a concentration of 5.3 mg ai/kg dry weight soil. The temperature of the irradiated soil samples was maintained at approximately 21 ± 2 °C with continuous irradiation for up to 15 days under simulated natural sunlight produced by a Xenon arc lamp. A set of non-irradiated controls were incubated at approximately 20 ± 2 °C and kept in the dark in the environmental chamber. The soil characteristics are listed below.

Origin location	Stark Country, IL, USA
Texture <sup>a</sup>	Light clay
% Sand (2000-50 μm)	33
% Silt (<50-2 μm)	36
% Clay (<2 μm)	31
pH	6.7
Organic matter (%)	4.1
Cation exchange capacity	17.0
Maximum water-holding capacity (%)	78.3



<sup>a</sup> International soil classification system

Recovery of radioactivity ranged from 87.6% applied radioactivity (AR) to 102.9% AR in all samples. In the irradiated oxamyl samples, the degradation products were IN-D2708, IN-N0079 and IN-A2213, which reached average maximum concentrations of 44.7% AR (Day 15), 8.7% AR (Day 5), and 3.6% AR (Day 3), respectively. In the non-irradiated oxamyl samples, the degradation products were IN-D2708 and IN-A2213, which reached average maximum concentrations of 6.7% AR (Day 11) and 8.0% AR (Day 3), respectively.

The DT<sub>50</sub> and DT<sub>90</sub> values using first order kinetics of oxamyl were 4.7 and 15.7 days in irradiated samples and 24.2 and 80.5 days in non-irradiated samples, respectively.

### Field soil dissipation

#### Study 1

The study was conducted to determine the environmental fate and the rate of decline of oxamyl, its major soil degradates IN-A2213 and IN-D2708 following application of a SL formulation to cropped and non-cropped plots under actual field conditions in Italy (Zietz, 2002: DuPont-4800) and Spain (LeNoir, 2003: Dupont-4719). Oxamyl was applied once to each of the six plots using a drip irrigation technique delivering 1.5 kg ai/ha to the soil surface. The greenhouse conditions were maintained according to local practice. The non-cropped plots were kept in non-cropped condition throughout the study period while cucumbers (*Cucumis sativus*) were grown on the cropped plots.

Soil samples were analysed for residues of oxamyl and IN-A2213 by HPLC-MS/MS. The LOQ for oxamyl and IN-A2213 was 0.005 mg/kg. The analysis of IN-D2708 was performed separately using an HPLC-MS technique operating in the negative ion mode. The LOQ for IN-D2708 was 0.01 mg/kg.

For each sampling time, the residue concentrations of oxamyl, IN-A2213 and IN-D2708 in each depth segment were converted to oxamyl equivalents and expressed on a mass/area basis ( $\mu\text{g}/\text{cm}^2$ ). The resulting mass/area values for each depth segment were summed for the entire sampled soil profile, which provided the total mass of each analyte present in the sampled soil profile at each sampling time. Due to the similarity of the cropped and non-cropped plot data, the average mass/area values (n=6, average of three cropped and three non-cropped plots) were used in the primary kinetic analysis. A non-linear simple first order regression technique was used to determine the half-lives.

Table 40 DT<sub>50</sub> and DT<sub>90</sub> for oxamyl, IN-A2213 and IN-D2708 in greenhouse soils

Location	Oxamyl		IN-A2213	IN-D2708
	DT <sub>50</sub> (days)	DT <sub>90</sub> (days)	DT <sub>50</sub> (days)	DT <sub>50</sub> (days)
Sicily, Italy	5.3	18	5.7	3.2
Vinalesa, Spain	3.3	11	2.1	0.52

#### Study 2

The field soil dissipation study was conducted to determine the dissipation of oxamyl together with its primary soil degradation products IN-A2213 and IN-D2708 following application of the test substance under actual field conditions in the Netherlands (Mol, 2002: DuPont-2815) and England (Zietz, 2002: DuPont-3026). Oxamyl, as a GR formulation, was applied once at 4.0 kg ai/ha (NL) and 5.5 kg ai/ha (UK) to bare ground. The agricultural sites were maintained in a non-cropped condition throughout the study period.

Soil samples were analysed for residues of oxamyl and IN-A2213 by HPLC-MS/MS. The LOQ for oxamyl and IN-A2213 was 0.005 mg/kg. The analysis of the degradate IN-D2708 was performed separately using a specific method basing on HPLC-MS technique operating in the negative ion mode. The LOQ for IN-D2708 was 0.01 mg/kg.

Degradation rates were determined for the individual plots using a nonlinear regression of first-order kinetic equations.

Table 41 DT<sub>50</sub> and DT<sub>90</sub> for oxamyl, IN-A2213 and IN-D2708 in soils

Location	Oxamyl		IN-A2213		IN-D2708	
	DT <sub>50</sub> (days)	DT <sub>90</sub> (days)	DT <sub>50</sub> (days)	DT <sub>90</sub> (days)	DT <sub>50</sub> (days)	DT <sub>90</sub> (days)
Limburg, NL	9.25	30.7	1.70	5.64	6.68	22.2
Lincolnshire, UK	11	36	4.5	15	3.4	11

### Study 3

Three bare field dissipation studies were carried out in the USA at a total of four sites. The locations of these studies were Madera/CA (Lin, 1990: AMR 1824-90, Revision No. 1), Bradenton/FL, Wapato/WA, Madera/CA (Lin, 1991: AMR 1151-88, Revision No. 1), and Greenville/MS (McClory, 1996: AMR 2889-93). In all four field dissipation studies, oxamyl as the SL formulation was applied once at 20.2 kg ai/ha to bare ground via a broadcast sprayer.

The soils were sampled to a depth of 90 cm and analysed for oxamyl and IN-A2213. Oxamyl and IN-A2213 were found to have a low mobility. The estimated first order half-lives of oxamyl in these US dissipation trials ranged from 9–29 days.

Table 42 Estimated half-lives for oxamyl in US field dissipation studies

Study	Location	DT <sub>50</sub> (days)
AMR-1824-90	Madera, CA	12
	Bradenton, FL	18
AMR 1151-88	Wapato, WA	29
	Madera, CA	12
AMR 2889-93	Greenville, MS	9

### Environmental fate in water and water-sediment systems

The Meeting received information on hydrolytic degradation, photochemical degradation and degradation in water sediment systems. Because oxamyl is intended for use as soil treatment, hydrolytic degradation study relevant to the current evaluations were reported below (FAO Manual Third edition, 2016).

#### Hydrolysis

The hydrolysis of [1-<sup>14</sup>C]-oxamyl was studied in sterile aqueous solutions buffered at pH 4 (0.01 M acetate), pH 7 (0.01 M phosphate), and pH 9 (0.01 M borate) at three temperatures ranging from 20 ± 1 °C to 30 ± 1 °C and up to a period of 30 days (Clark, 2014: DuPont-39015). The concentration of the test substances was 0.928–1.04 mg/L.

In the preliminary experiment, oxamyl was shown to be stable at pH 4, so this pH was not run in the definitive experiment. For the definitive experiment, buffer solutions at pH 7 and 9 were fortified with test substance and incubated at temperatures ranging from 20 °C to 30 °C and analysed at various intervals by HPLC with an in-line radiochemical detector and LSC. Radioactivity was quantitatively recovered from each test solution with averages from 97.8 to 100.7 % of applied radioactivity (AR).

At each pH and temperature, hydrolysis products greater than 10% of AR were identified. From the [<sup>14</sup>C]-oxamyl fortification, the major transformation product identified was IN-A2213. The first-order DT<sub>50</sub> values (days) of oxamyl in aqueous solution at various pH and temperature are summarized in the following table.

Table 43 DT<sub>50</sub> and DT<sub>90</sub> for oxamyl in aqueous solution

pH	Temperature	DT <sub>50</sub> (days)	DT <sub>90</sub> (days)
4	-	Stable	Stable
7	20 °C	21	70
	25 °C	9.0	30
	30 °C	4.2	14
9	20 °C	0.20	0.67
	25 °C	0.098	0.32
	30 °C	0.046	0.15

Table 44 The maximum amount in % AR and times in days of the major transformation product, IN-A2213, in buffer solutions

pH	Temperature	% AR	Times (days)
7	20 °C	63.2	29.7
	25 °C	89.0	30.0
	30 °C	91.0	14.8
9	20 °C	68.7	0.34
	25 °C	87.7	0.30
	30 °C	94.9	0.19

Oxamyl was hydrolytically stable at acidic conditions (pH 4), but unstable under neutral condition (pH 7) and alkaline conditions (pH 9). Hydrolysis occurred at a more rapid rate at higher temperatures across the pH range tested. On the basis of these results, oxamyl is considered hydrolytically unstable at temperatures above 20 °C for pH 7, and all temperatures tested for pH 9.

## RESIDUE ANALYSIS

### *Analytical methods*

Descriptions of analytical methods together with validation data for residues of oxamyl in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction with solvent. After column clean-up, the oxamyl residue is prepared for HPLC analysis. Oxamyl residues can be measured by fluorescence or mass spectrometric (MS/MS) detection, to an LOQ of 0.01 mg/kg.

Detailed descriptions of all these analytical methods are presented below.

### *Plant matrices*

Melon, Lettuce, Sugar beet, Potato, Citrus fruits (DuPont-4722)

Analyte: Oxamyl HPLC-PCD/Fluo

LOQ: 0.01 mg/kg

Description Samples (15 g) were extracted with acetone, and partitioned into dichloromethane/petroleum ether (1:1, v/v). An aliquot of extract was evaporated to dryness and cleaned-up with solid phase extraction using aminopropyl-bonded silica cartridges. Oxamyl residues were determined by HPLC with post-column derivatization and fluorescence detection (HPLC-PCD/Fluo).

[Determination was performed by HPLC with post-column hydrolysis of oxamyl, derivatization of the formed methylamine by reaction with o-phthaldehyde reagent, and fluorescence detection of the derivative.]

Melon, Lettuce, Sugar beet, Potato, Citrus fruits (DuPont-3702)

Analyte: Oxamyl, IN-A2213 (oxamyl oxime) HPLC-CS/UV

LOQ: 0.02 mg/kg for both analytes

**Description** Samples (3 g) were extracted by Accelerated Solvent Extraction (ASE), using acetone as the extraction solvent. The whole extract was cleaned-up using ENVI-Carb SPE cartridge to remove pigments and other interfering molecules. The whole SPE eluate was then concentrated to about 0.5 mL by evaporation. The extract was dissolved in a mixture of 10% acetone in cyclohexane (v/v) and applied to a Silica Mega Bond Elut SPE cartridge to complete the clean-up. The eluate from the Silica SPE was evaporated to about 0.5 mL under nitrogen, then the final volume of the extract was brought to 2 mL with a mixture of 8% acetonitrile in water (v/v). The final extract was filtrated and analysed by HPLC with column-switching and UV detection (HPLC-CS/UV).

Potato (DuPont-11125)

**Analyte:** Oxamyl LC-MS No. 0259

**LOQ:** 0.01 mg/kg

**Description** Samples (15 g) were extracted with acetone, and partitioned into dichloromethane/petroleum ether (1:1, v/v). An aliquot of extract was evaporated to dryness and cleaned-up by solid phase extraction using an aminopropyl cartridge. Oxamyl residues were determined by LC-MS.

Wheat grain, Linseed, Cucumber, Orange (DuPont-33191)

**Analyte:** Oxamyl LC-MS/MS Charles River Analytical Procedure No. 1901.01  
(m/z 237→72 for quantification, m/z 237→90 for confirmation)

**LOQ:** 0.01 mg/kg for wheat grain, linseed and cucumber, 0.005 mg/kg for orange

**Description** Samples (15 g) were extracted with acetone, and partitioned into dichloromethane/petroleum ether (1:1, v/v). An aliquot of extract was evaporated to dryness and cleaned-up by solid phase extraction using an aminopropyl cartridge. Oxamyl residues were determined by LC-MS/MS employing positive-ion electrospray ionisation (ESI).

Tabacco leaf (green, dried and fermented) (DuPont-17601)

**Analyte:** Oxamyl LC-MS No.0893

**LOQ:** 0.01 mg/kg

**Description** Samples (15 g) were extracted with acetonitrile, and partitioned into hexane. An aliquot of acetonitrile extract was evaporated to dryness and cleaned-up by solid phase extraction using an aminopropyl cartridge. Oxamyl residues were determined by LC-MS.

Tobacco leaf (dried) , Wheat, Tomato, Avocado, Grape (DuPont-41730)

**Analyte:** Oxamyl LC-MS/MS QuEChERS  
(m/z 237→72 for quantification, m/z 237→90 for confirmation)

**LOQ:** 0.01 mg/kg

**Description** Samples (2.0 g) are weighed into a 50 mL polypropylene centrifuge tube. To each sample, 100 µL of the internal standard Carbofuran-d3 was added. Water (10 mL) and 1% acetic acid in acetonitrile (10 mL) were added to the samples along with 1 g of anhydrous sodium acetate. After the samples had been vortexed for 20 sec, 4 g of anhydrous magnesium sulphate were added to the sample, and the samples were shaken at 1700 rpm for 1 min. The samples were then centrifuged at 3600 rpm for 5 min. The supernatant was pipetted into 15 mL QuEChERS centrifuge tubes that contain 400 mg PSA and 1200 mg MgSO<sub>4</sub>. Following centrifugation at 3600 rpm for 5 min, 5.0 mL of the supernatant was pipetted into a 15 mL polypropylene centrifuge tube and blow down to between 0.2 and 0.3 mL under a stream of nitrogen. This sample was then sequentially diluted with methanol and 0.1 M ammonium acetate (1:1, v/v). The samples were then syringe filtered into amber vial for LC-MS/MS analysis (positive-ion electrospray ionization).

#### *Extraction efficiency*

A study was conducted to compare the quantity of [<sup>14</sup>C]-oxamyl from crop commodities using the extraction methods described in DuPont-41730 (QuEChERS method), DuPont-17601 (Crop Residue Method No. 0893) and Dupont-32188 (Plant Metabolism Method) (Cochrane, 2015: DuPont-44316). The crop samples selected were tomato fruit and tomato foliage which had received four foliar applications of [1-<sup>14</sup>C]-oxamyl. The first application was applied immediately after transplant of the tomato plants at a target rate of 2.0 kg ai/ha followed by three subsequent 1.0 kg ai/ha applications carried out 14 days apart to achieve a 21 day pre harvest interval (PHI).

For the purposes of this study, the residue of interest in crops was oxamyl only. The profile of components extracted from the fruit and foliage samples was compared by HPLC using fraction collection and online radio-detection. The radiochromatograms showed a similar distribution and concentration of oxamyl (the residue of interest) in all samples, regardless of which extraction technique was used; a comparison of this data is presented below.

Table 45 Extraction efficiency of oxamyl residue analytical methods

	% TRR identified as oxamyl			QuEChERS method as % difference of metabolism method	Crop residue method as % difference of metabolism method
	QuEChERS method % TRR (mg/kg)	Crop residue method No.0893 % TRR (mg/kg)	Metabolism method % TRR (mg/kg)		
Tomato fruit	3.9 (0.039)	3.9 (0.038)	3.1 (0.031)	0.8 (0.008)	0.8 (0.007)
Tomato foliage	60.2 (24.0)	79.0 (31.5)	71.5 (28.5)	11.3 (4.5)	7.5 (3.0)

HPLC quantification data showed that the crop residue and the QuEChERS methods are suitable for extracting the residue of interest, parent oxamyl.

Validation data for methods on plant matrices are summarized in Table 46.

Table 46 Summary of Recovery Data for oxamyl fortified into plant matrices

Commodity	Transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Melon, flesh (MV)	-	0.01	5	88 – 98	93	4	DuPont-4722
		0.1	5	67 – 82	73	10	
Melon, peel (MV)	-	0.01	6	81–116	99	13	
		0.1	5	76–111	88	17	
Lettuce, leaf (MV)	-	0.01	5	70–108	92	19	
		0.1	7	57–95	80	20	
Sugar beet, root (MV)	-	0.01	5	96–108	103	6	
		0.1	5	84–112	94	12	
Sugar beet, tops (MV)	-	0.01	5	82–105	95	9	
		0.1	5	62–97	81	19	

Commodity	Transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Potato, tuber (MV)	-	0.01 0.1	5 5	105–112 82–105	108 91	3 10	
Citrus fruit, peel (MV)	-	0.01 0.1	5 5	78–97 75–89	87 82	10 8	
Citrus fruit, pulp (MV)	-	0.01 0.1	5 5	71–111 60–86	86 74	20 13	
Tomato (MV)	-	0.01 0.1	2 2	84, 84 78, 80	84 79	- -	DuPont-4583
Tomato (CR)	-	0.01 0.1	3 3	72–105 65–77	89 74	18 11	
Sugar beet, root (CR)	-	0.01 0.1	3 3	72–79 82–83	75 83	5 1	DuPont-3940
Sugar beet, Leaves with top (CR)	-	0.01 0.1	3 3	71–82 71–79	78 76	8 5	
Sugar beet, root (CR)	-	0.01 0.1	3 3	97–103 89–92	99 90	3 2	DuPont-4582, Revision No. 1
Sugar beet, Leaves with top (CR)	-	0.01 0.1	3 3	99–108 88–92	104 90	5 2	
Potato (CR)	-	0.01 0.1	2 2	74, 76 80, 83	75 81	- -	DuPont-5989
Melon, flesh (MV)	-	0.02 0.2	8 5	59–88 74–84	79 80	12 4	DuPont-3702
	(IN-A2213)	0.02 0.2	5 5	62–96 67–91	84 76	16 12	
Melon, peel (MV)	-	0.02 0.2	7 7	67–106 65–93	86 80	17 16	
	(IN-A2213)	0.02 0.2	7 7	89–112 68–81	98 75	8 6	
Lettuce, leaf (MV)	-	0.02 0.2	8 5	70–104 60–80	90 74	14 11	
	(IN-A2213)	0.02 0.2	5 5	79–110 71–84	96 79	12 7	
Sugar beet, root (MV)	-	0.02 0.2	5 5	63–106 59–95	86 77	19 20	
	(IN-A2213)	0.02 0.2	5 5	83–110 64–94	93 76	12 16	
Sugar beet, tops (MV)	-	0.02 0.2	5 5	68–96 81–95	80 89	16 7	
Potato, tuber (MV)	-	0.02 0.2	5 8	67–101 78–104	82 92	20 11	
Sugar beet, root (CR)	-	0.02 0.2	3 3	70–100 65–80	83 72	18 11	DuPont-2408
Sugar beet, Leaves with top (CR)	-	0.02 0.2	3 3	65–100 70–75	83 73	21 4	
Potato (MV)	-	0.01 0.1	5 5	82–88 86–93	86 90	3 3	DuPont-11125 No. 0259
Brussels sprouts (CR)	-	0.01 0.1	2 2	94, 96 89, 93	95 91	- -	DuPont-14669 No. 0259
Cucumber/Courgette (CR)	-	0.01 0.1	12 12	70–85 70–92	77 81	7 10	DuPont-19518 No.0259
Cucumber (CR)	-	0.01 0.1	4 4	75–89 78–88	81 82	7 6	DuPont-29314 No.0259
Courgette (CR)	-	0.01 0.1	2 2	72, 84 79, 79	78 79	- -	

Commodity	Transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method	
Melon, peel (CR)	-	0.005 0.1	8 8	59-108 79-95	85 87	19 6	DuPont-29316 No. 0259	
Melon, flesh (CR)	-	0.005 0.1	8 7	72-96 71-92	83 84	11 8		
Cherry tomato (CR)	-	0.01 0.1	6 6	64-83 70-86	76 76	8 8	DuPont-19521 No. 0259	
Tomato (CR)	-	0.01 0.1	7 8	70-100 66-94	76 77	14 13	DuPont-19519 No. 0259	
Cherry tomato/Tomato (CR)	-	0.01 0.1	10 10	77-94 77-88	85 83	6 4	DuPont-29313 No. 0259	
Pepper (CR)	-	0.01 0.1	11 11	70-96 57-106	82 84	12 16	DuPont-19522 No. 0259	
Pepper (CR)	-	0.01 0.1	4 4	83-94 83-91	88 88	5 4	DuPont-29315 No. 0259	
Eggplant (CR)	-	0.01 0.1	4 4	75-87 76-88	82 83	7 6	DuPont-19520 No. 0259	
Eggplant (CR)	-	0.01 0.1	4 4	85-93 78-90	89 85	4 7	DuPont-29317 No. 0259	
Carrot (CR)	-	0.01 0.1	4 4	72-88 77-85	80 81	11 5	DuPont-13037 No. 0259	
Carrot (CR)	-	0.01 0.1	2 2	87, 87 87, 91	87 89	- -	DuPont-14668 No. 0259	
Potato (CR)	-	0.005 0.1	4 4	84-93 84-101	87 92	5 9	DuPont-19526 No. 0259	
Potato, whole tubers (CR)	-	0.005 0.1	2 2	81, 85 90, 96	83 93	- -	DuPont-27667 No. 0259	
Potato, baked tubers (CR)	-	0.005 0.1	2 2	85, 91 91, 92	88 91	- -		
Potato, boiled tubers (CR)	-	0.005 0.1	1 2	97 99, 114	97 107	- -		
Potato, microwave baked tubers (CR)	-	0.005 0.1	2 2	92, 114 93, 95	103 94	15 2		
Potato, microwave baked tubers (CR)	-	0.005 0.1	2 2	92, 114 93, 95	103 94	15 2		
Wheat grain (MV)	m/z 237→72 Quantification	0.01 0.1	4 5	73-91 93-115	82 103	10 10	DuPont-33191 Charles River Analytical Procedure No. 1901.01	
	m/z 237→90 Confirmation	0.01 0.1	4 5	87-103 96-116	95 106	8 9		
Linseed (MV)	m/z 237→72 Quantification	0.01 0.1	5 5	88-113 94-104	100 100	10 4		
	m/z 237→90 Confirmation	0.01 0.1	5 5	94-118 94-103	101 100	11 4		
Cucumber (MV)	m/z 237→72 Quantification	0.01 0.1	5 5	71-86 79-86	83 82	8 3		
	m/z 237→90 Confirmation	0.01 0.1	5 5	65-87 74-85	75 81	12 5		
Orange (MV)	m/z 237→72 Quantification	0.005 0.1	5 5	78-87 77-85	83 82	5 4		
	m/z 237→90 Confirmation	0.005 0.1	5 5	68-91 77-85	83 82	11 4		
Cucumber (CR)	m/z 237→72 Quantification	0.01 0.1	2 2	63, 81 86, 87	72 87	- -		DuPont-31505 No. 1901
Courgette (CR)	m/z 237→72 Quantification	0.01 0.1 0.2 0.5	3 3 3 2	71-82 79-86 81-90 89, 96	78 82 86 93	7 4 5 -		
Melon, peel (CR)	m/z 237→72 Quantification	0.005 0.1	5 5	77-117 67-124	90 93	18 28		
Melon, peel (CR)	m/z 237→72 Quantification	0.005 0.1	5 5	77-117 67-124	90 93	18 28		

Commodity	Transition	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Melon, flesh (CR)	m/z 237→72 Quantification	0.005	3	70–85	76	11	
		0.1	3	52–101	82	32	
Cherry tomato/Tomato (CR)	m/z 237→72 Quantification	0.01	4	64–105	91	21	DuPont-31506 No. 1901
		0.1	4	72–86	80	8	
Eggplant (CR)	m/z 237→72 Quantification	0.01	2	76, 87	81	-	DuPont-31509 No. 1901
		0.1	2	83, 89	86	-	
Tobacco, green leaves (MV)	-	0.01	5	92–102	96	4	DuPont-17601 No.0893
		0.1	5	84–98	93	6	
Tobacco, dried leaves (MV)	-	0.01	4	92–105	100	6	
		0.1	5	93–106	97	5	
Tobacco, fermented leaves (MV)	-	0.01	5	86–102	92	7	
		0.1	5	81–84	82	1	
Grape (MV)	m/z 237→72 Quantification	0.01	5	92–96	94	2	DuPont-41730 TOPS-142 (QuEChERS method)
		0.1	5	100–104	102	1	
	m/z 237→90 Confirmation	0.01	5	95–97	96	1	
		0.1	5	98–100	99	1	
Tomato (MV)	m/z 237→72 Quantification	0.01	5	98–102	101	2	
		0.1	5	105–111	107	3	
	m/z 237→90 Confirmation	0.01	5	100–103	101	1	
		0.1	5	99–104	102	2	
Avocado (MV)	m/z 237→72 Quantification	0.01	5	66–96	80	15	
		0.1	5	99–106	103	3	
	m/z 237→90 Confirmation	0.01	5	72–100	85	13	
		0.1	5	96–103	99	3	
Wheat, flour (MV)	m/z 237→72 Quantification	0.01	5	76–85	82	5	
		0.1	5	104–108	106	1	
	m/z 237→90 Confirmation	0.01	5	97–102	99	2	
		0.1	5	101–103	102	1	
Tobacco, dried leaves (MV)	m/z 237→72 Quantification	0.01	5	86–95	92	4	
		0.1	5	94–100	97	3	
	m/z 237→90 Confirmation	0.01	5	87–96	92	4	
		0.1	5	93–98	95	2	
Tomato (ILV)	m/z 237→72 Quantification	0.01	5	79–97	88	9	DuPont-41873 TOPS-142 (QuEChERS method)
		0.1	5	87–114	104	11	
	m/z 237→90 Confirmation	0.01	5	80–95	87	7	
		0.1	5	88–112	104	10	
Grape (ILV)	m/z 237→72 Quantification	0.01	5	76–88	80	6	
		0.1	5	88–98	94	5	
	m/z 237→90 Confirmation	0.01	5	75–87	80	6	
		0.1	5	87–96	92	4	
Tobacco (ILV)	m/z 237→72 Quantification	0.01	5	78–90	84	5	
		0.1	5	77–89	83	6	
	m/z 237→90 Confirmation	0.01	5	77–92	84	8	
		0.1	5	78–90	84	6	

CR: Concurrent Recovery, MV: Method Validation, ILV: Independent Laboratory Validation

### Animal matrices

Milk, Muscle, Liver, Fat, Eggs (DuPont-38597)

Analyte: Oxamyl  
(m/z 237→72 for quantification, m/z 237→90 for confirmation)

LC-  
MS/MS

LOQ: 0.01 mg/kg



**Description** Sample (5 g) was added to 10 mL of 0.1% formic acid in methanol and shaken at 1100 strokes/min for 2 min. Following extraction, the sample was centrifuged at 3000 rpm for 10 min and the extract was decanted. Oily co-extracts were removed by vortexing the samples in the presence of 10 mL of hexane and centrifuging at 3000 rpm for 5 min. The hexane layer was then discarded. The extracts were cleaned up further through the addition of 0.25 g of SAX (Strong Anion Exchanger) sorbent to a small volume of each extract: 400  $\mu$ L for liver, fat, eggs and bovine muscle and 600  $\mu$ L for milk (cream, low fat and whole) in a 15 mL centrifuge tube. The extracts were then brought to a total volume of 10 mL with HPLC grade water and vortexed for 10 sec to disperse the SAX sorbent. The SAX sorbent was removed by centrifuging the samples for 5 min at 3000 rpm. Each of the cleaned extracts were then subjected to LC-MS/MS analysis (positive-ion electrospray ionization).

#### Blood (DuPont-38598)

**Analyte:** Oxamyl LC-MS/MS  
(m/z 237 $\rightarrow$ 72 for quantification, m/z 237 $\rightarrow$ 90 for confirmation)

**LOQ:** 0.01 mg/kg

**Description** 100  $\mu$ L of blood were placed in a 15 mL centrifuge tube. 400  $\mu$ L of 0.1% formic acid in methanol are added to the sample. The sample was then vortexed for 15 sec, inverted several times to ensure homogeneity and vortexed for another 15 sec. The sample was then diluted with 0.5 mL of HPLC grade water and vortexed again for 15 sec. Following extraction, the sample was centrifuged at 3000 rpm for 5 min to separate the precipitated proteins. After centrifugation, 20  $\mu$ L of the extract was added to a 15 mL centrifuge tube containing 50  $\pm$  10 mg of SAX sorbent and 980  $\mu$ L of water. The diluted sample was then vortexed to ensure homogeneity. The SAX sorbent was separated by centrifuging the samples for 5 min at 3000 rpm. Each of the cleaned extracts was then subjected to LC-MS/MS analysis (positive-ion electrospray ionization).

#### Milk, Eggs, Muscle, Liver, Fat (DuPont-41763)

**Analyte:** Oxamyl LC-MS/MS QuEChERS  
(m/z 237 $\rightarrow$ 72 for quantification, m/z 237 $\rightarrow$ 90 for confirmation)

**LOQ:** 0.01 mg/kg

**Description** Sample (2.0 g) were weighed into a 50 mL polypropylene centrifuge tube. To each sample, 100  $\mu$ L of the internal standard Carbofuran-d3 was added. Water (10 mL) and 1% acetic acid in acetonitrile (10 mL) were added to the samples along with 1 g of anhydrous sodium acetate. After the samples had been vortexed for 20 sec, 4 g of anhydrous magnesium sulphate were added to the sample, and the samples were shaken at 1700 rpm for 1 min. The samples were then centrifuged at 3600 rpm for 5 min. The supernatant was pipetted into 15 mL QuEChERS centrifuge tubes that contain 400 mg PSA and 1200 mg MgSO<sub>4</sub>. Following centrifugation at 3600 rpm for 5 min, 5.0 mL of the supernatant was pipetted into a 15 mL polypropylene centrifuge tube and blow down to between 0.2 and 0.3 mL under a stream of nitrogen. This sample was then sequentially diluted with methanol and 0.1 M ammonium acetate (1:1, v/v). The samples were then syringe filtered into amber vial for LC-MS/MS analysis (positive-ion electrospray ionization).

Validation data for methods on animal matrices are summarized in Table 47.

Table 47 Summary of Recovery Data for oxamyl fortified into animal matrices

Commodity	Transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Chicken liver (MV)	m/z 237→72	0.01	5	98–108	102	4	DuPont-38597
	Quantification	0.1	5	82–90	86	4	
	m/z 237→90	0.01	5	94–113	105	7	
	Confirmation	0.1	5	82–100	90	7	
Eggs (MV)	m/z 237→72	0.01	5	94–114	102	7	
	Quantification	0.1	5	86–94	90	4	
	m/z 237→90	0.01	5	85–107	97	9	
	Confirmation	0.1	5	83–103	96	8	
Bovine muscle (MV)	m/z 237→72	0.01	5	92–107	103	6	
	Quantification	0.1	5	88–90	89	1	
	m/z 237→90	0.01	5	95–102	99	3	
	Confirmation	0.1	5	84–89	87	3	
Bovine fat (MV)	m/z 237→72	0.01	5	78–102	88	12	
	Quantification	0.1	5	76–85	82	4	
	m/z 237→90	0.01	5	80–95	86	7	
	Confirmation	0.1	5	73–92	80	11	
Milk (MV)	m/z 237→72	0.01	5	96–112	103	7	
	Quantification	0.1	5	87–108	95	9	
	m/z 237→90	0.01	5	97–113	104	6	
	Confirmation	0.1	5	94–103	99	3	
Low fat milk (MV)	m/z 237→72	0.01	5	93–106	102	5	
	Quantification	0.1	5	86–104	96	8	
	m/z 237→90	0.01	5	91–109	104	7	
	Confirmation	0.1	5	94–103	99	3	
Cream (MV)	m/z 237→72	0.01	5	99–103	104	4	
	Quantification	0.1	5	88–100	92	6	
	m/z 237→90	0.01	5	87–103	98	7	
	Confirmation	0.1	5	81–97	89	7	
Bovine muscle (ILV)	m/z 237→72	0.01	5	77–87	81	5	DuPont-39679
	Quantification	0.1	5	77–90	84	6	
	m/z 237→90	0.01	5	79–92	83	6	
	Confirmation	0.1	5	76–93	84	7	
Eggs (ILV)	m/z 237→72	0.01	5	66–76	70	6	
	Quantification	0.1	5	70–92	80	10	
	m/z 237→90	0.01	5	69–77	72	5	
	Confirmation	0.1	5	70–90	79	10	
Blood (MV)	m/z 237→72	0.05	5	78–111	102	14	DuPont-38598
	Quantification	0.5	5	87–123	99	15	
	m/z 237→90	0.05	5	78–109	99	13	
	Confirmation	0.5	5	87–124	99	15	
Milk (MV)	m/z 237→72	0.01	5	96–102	100	2	DuPont-41763 (QuEChERS method)
	Quantification	0.10	5	104–107	106	1	
	m/z 237→90	0.01	5	99–101	100	1	
	Confirmation	0.10	5	99–105	101	2	
Eggs (MV)	m/z 237→72	0.01	5	96–99	97	1	
	Quantification	0.10	5	97–102	100	2	
	m/z 237→90	0.01	5	91–97	93	2	
	Confirmation	0.10	5	98–101	99	1	
Muscle (MV)	m/z 237→72	0.01	5	93–97	95	2	
	Quantification	0.10	5	101–108	105	3	
	m/z 237→90	0.01	5	93–99	95	2	
	Confirmation	0.10	5	97–102	101	2	
Liver (MV)	m/z 237→72	0.01	5	76–90	83	7	
	Quantification	0.10	5	94–100	97	3	
	m/z 237→90	0.01	5	79–93	86	5	
	Confirmation	0.10	5	91–94	93	2	
Fat (MV)	m/z 237→72	0.01	5	79–92	84	6	
	Quantification	0.10	5	85–93	90	3	

Commodity	Transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference	
	m/z 237→90 Confirmation	0.01 0.10	5 5	81–99 85–90	88 87	7 2		
Muscle (ILV)	m/z 237→72 Quantification	0.01 0.1	5 5	81–90 97–105	86 100	4 3	DuPont-41873 (QuEChERS method)	
	m/z 237→90 Confirmation	0.01 0.1	5 5	81–90 97–104	87 99	4 3		
	Milk (MV)	m/z 237→72 Quantification	0.01 0.1	5 5	92–97 94–97	95 95		2 1
		m/z 237→90 Confirmation	0.01 0.1	5 5	93–96 94–98	94 95		1 2
Eggs (MV)	m/z 237→72 Quantification	0.01 0.1	5 5	83–88 92–95	86 94	2 1		
	m/z 237→90 Confirmation	0.01 0.1	5 5	83–86 92–96	85 94	2 2		

CR: Concurrent Recovery, MV: Method Validation, ILV: Independent Laboratory Validation

### Soil

#### Soil (DuPont-38689)

Analyte: Oxamyl LC-  
(m/z 237→72 for quantification, m/z 237→90 for MS/MS  
confirmation)

LOQ: 0.001 mg/kg

Description Soil (10 g) was added to 10 mL of a 9 to 1 methanol to water solution and shaken at 1100 strokes/min for 2 min. The sample was centrifuged at 3000 rpm for 10 min and the extract was decanted. The procedure was repeated an additional time, yielding a total of 20 mL of soil extract. The extract was syringe filtered and 100 µL was diluted to a final volume of 1.0 mL with water. The sample was then subjected to LC-MS/MS analysis (positive-ion electrospray ionization).

#### Soil (DuPont-2392, Revision No.1)

Analyte: Oxamyl, IN-A2213 LC-MS

LOQ: 0.001 mg/kg

Description Soil (13 g fw) was combined and mixed with silica gel (2/1 w/w). Soil: silica gel mix was loaded into ASE extraction cell. The sample was extracted with 0.01% formic acid in acetonitrile/methanol (80:20, v/v). The extract was added 1 mL of 0.01% formic acid in water and concentrated under nitrogen to 1.5 mL. The concentrate was diluted to the final volume of 10 mL using 0.01% formic acid in water, sonicated and shaken. The sample was filtered and subjected to LC-MS analysis.

#### Soil (DuPont-7191, Revision No.1)

Analyte: Oxamyl LC-  
(m/z 237→72 for quantification, m/z 237→90 for MS/MS  
confirmation)

IN-A2213

(m/z 163→72 for quantification, m/z 163→90 for  
confirmation)

LOQ: 0.005 mg/kg for oxamyl and IN-A2213

**Description** Soil samples were extracted with a preheated (50 °C) solution of 2.5% formic acid in methanol/acetonitrile (25/75, v/v), using mechanical shaking. An aliquot of extract was taken and the solvent was exchanged to methanol/0.1% formic acid (10/90, v/v) in 10 mM ammonium acetate. The sample was then analysed for Oxamyl and IN-A2213 metabolite using reversed phase LC/MS/MS (positive-ion electrospray ionization).

Validation data for methods on soil are summarized in Table 48.

Table 48 Summary of Recovery Data for oxamyl and IN-A2213 fortified into soil

Soil name, Country Soil type	Analyte	Transition	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Speyer, Germany Sandy loam (MV)	Oxamyl	m/z 237→72	0.001	5	84–103	96	8	DuPont-38689
		Quantification	0.01	5	92–99	96	3	
		m/z 237→90	0.001	5	87–103	96	8	
		Confirmation	0.01	5	94–100	97	2	
Nambesheim, France Sandy loam (MV)	Oxamyl	m/z 237→72	0.001	5	80–98	89	8	
		Quantification	0.01	5	86–106	95	8	
		m/z 237→90	0.001	5	85–93	89	4	
		Confirmation	0.01	5	90–97	93	3	
Drummer, USA Clay loam (MV)	Oxamyl	m/z 237→72	0.001	5	80–88	82	4	
		Quantification	0.01	5	82–89	86	4	
		m/z 237→90	0.001	5	72–86	78	7	
		Confirmation	0.01	5	83–88	86	3	
Edgecombe Country, NC/USA Sandy (MV)	Oxamyl	-	0.01	7	76–96	90	7	DuPont-2392, Revision No.1
		-	0.03	3	88–93	90	2	
		-	0.10	3	85–104	95	10	
	IN-A2213	-	0.01	7	78–103	96	7	
		-	0.03	3	88–93	90	3	
		-	0.10	3	81–102	93	11	
Edgecombe Country, NC/USA Sandy (ILV)	Oxamyl	-	0.01	5	80–94	91	7	DuPont-3738
		-	0.10	5	100–113	103	2	
	IN-A2213	-	0.01	5	98–103	100	2	
		-	0.10	5	85–96	89	5	
Limburg, Netherlands Sandy loam (MV)	Oxamyl	m/z 237→72	0.0055	12	82–100	93	6	DuPont-7191, Revision No.1
		Quantification	0.11	6	86–102	93	8	
		-	2.2	6	72–96	90	11	
	IN-A2213	m/z 163→72	0.0056	12	78–103	88	7	
		Quantification	0.11	6	86–100	92	5	
		-	2.2	6	72–98	88	10	
Vinalesa, Spain Loamy sand (MV)	Oxamyl	m/z 237→72	0.0055	8	70–108	96	12	
		Quantification	0.11	4	71–100	90	14	
		-	2.2	4	94–119	103	11	
	IN-A2213	m/z 163→72	0.0056	8	90–114	103	7	
		Quantification	0.11	4	92–100	97	4	
		-	2.2	4	99–125	106	11	

MV: Method Validation, ILV: Independent Laboratory Validation

### ***Stability of pesticide residues in stored analytical samples***

The Meeting received data on the storage stability of oxamyl residue in orange, tomato, lettuce, sugar beet and potato samples for plant commodities stored frozen.

The stability of oxamyl was studied by fortifying homogenized control vegetable and fruit samples of lettuce, tomato, sugar beet root, potato tuber and orange peel with 0.50 mg/kg oxamyl (Dubey *et al.*, 2002: DuPont-4235). The fortified samples were stored in a freezer at approximately -18 °C. The stored samples were analysed at approximate time intervals of day 0, 3, 6, 12, 18 and 24 months of frozen storage. Oxamyl residue concentrations in the vegetable samples were determined by HPLC with column switching and UV detection (HPLC-CS/UV). Oxamyl residue concentrations

in orange peel were determined by HPLC with post-column derivatization followed by fluorescence detection (HPLC-PCD/Fluo). The LOQ was 0.01 mg/kg.

Table 49 Recovery of oxamyl from stored fortified samples of plant matrices

Storage interval (months)	Recovery (%) [0.50 mg/kg fortification]		
	Procedural	% remaining	Mean of % remaining
Potato tuber			
0	83, 83	-	-
3	78, 86	73, 80	77
6	73, 80	73, 82	78
12	70, 79	70, 88	79
18	77, 85	79, 81	80
24	71, 80, 85, 86	61, 65, 75, 79	70
Sugar beet root			
0	70, 77	-	-
3	74, 85	81, 83	82
6	75, 86	83, 85	84
12	71, 74	70, 73	72
18	76, 78	68, 70	69
24	78, 81	66, 84	75
Leaf lettuce			
0	96, 97	-	-
3	78, 84	77, 91	84
6	81, 86	86, 90	88
12	89, 93	86, 90	88
18	83, 86	81, 86	84
24	91, 100	90, 91	91
Tomato			
0	84, 96	-	-
3	78, 88	78, 87	83
6	75, 83	83, 91	87
12	77, 92	76, 85	81
18	73, 83	69, 73	71
24	77, 88	72, 82	77
Orange peel			
0	93, 96	-	-
6	60, 86	87, 95	91
12	93, 93	91, 94	93
18	96, 97	86, 90	88
24	82, 89	78, 84	81

Samples of ground oranges, fortified with oxamyl at a level of 0.10 mg/kg, were stored at approximately -20 °C for a duration of 12 months (Cairns *et al.*, 2013: DuPont-32189). Specimens were analysed for residues of oxamyl using Charles River Analytical Procedure No. 1901.01 described in Report No. DuPont-33191. Residues of oxamyl were extracted from orange samples by homogenising in acetone and then shaking using a mixture of solvent. Following SPE clean-up of the extracts, oxamyl was quantified by LC/MS/MS analysis. The LOQ was 0.005 mg/kg.

Table 50 Recovery of oxamyl from stored fortified samples of orange

Storage interval (months)	Recovery (%) [0.10 mg/kg fortification]		
	Procedural	% remaining	Mean of % remaining
0	87, 93	-	-
3	81, 95	84, 110	97
6	98, 109	101, 109	105
9	80, 88	83, 95	89
12	90, 115	100, 102	101

## USE PATTERN

Oxamyl is registered for use on a variety of crops. The Meeting received labels in Italy, Netherlands, Spain and UK. The information available to Meeting on registered uses of oxamyl is summarized in Table below.

Table 51 Registered uses of oxamyl for crops

Crop	Country	Formulation		Application				PHI, days and/or Application timing
		Type	Conc. of oxamyl	Method	Rate kg ai/ha	No. max	Interval, days	
Tomatoes* Eggplant*	Italy	SL	100 g/L	Soil drip irrigation	1.0-2.0 1.0 for 2nd-4th appl. max 5.0/crop cycle	4	10-14	PHI 28
Tomatoes Eggplant	Spain	SL	100 g/L	Soil irrigation via sprinkler	2.0 for 1st appl. 1.0 for 2nd-4th appl. max 5.0/crop cycle	4	10-14	PHI 28 The 1st treatment at immediately after transplant up to 42 days after the transplant
Tomatoes*	Italy	GR	50 g/kg	Soil broadcast with incorporation	4.5-5.5	1	-	Prior to transplanting
				Soil incorporation in furrow	3.0-3.5			At planting
Peppers*	Italy	SL	100 g/L	Soil drip irrigation	1.0-2.0 1.0 for 2nd-3rd appl max 4.0/crop cycle	3	10-14	PHI 35
Peppers	Spain	SL	100 g/L	Soil irrigation via sprinkler	2.0 for 1st appl. 1.0 for 2nd-3rd appl max 4.0/crop cycle	3	10-14	PHI 35 The 1st treatment at immediately after transplant up to 28 days after the transplant
Cucumber* Zucchini* Melons* Watermelons*	Italy	SL	100 g/L	Soil drip irrigation	1.0-2.0 1.0 for 2nd appl. max 3.0/crop cycle	2	10-14	PHI 50
Melons Watermelons	Spain	SL	100 g/L	Soil irrigation via sprinkler	2.0 for 1st appl. 1.0 for 2nd appl. max 3.0/crop cycle	2	10-14	PHI 50 The 1st treatment at immediately after transplant up to 10-14 days after the transplant
Brussels sprouts	Netherlands	GR	100 g/kg	Soil incorporation	4.0	1	-	Before planting
Sugar beet	Italy	GR	50 g/kg	Soil incorporation in furrow	1.5-2.0	1	-	At planting
Sugar beet	Netherlands	GR	100 g/kg	Soil incorporation in furrow	0.75-2.5	1	-	At drilling
Sugar beet	UK	GR	100 g/kg	Soil incorporation in seed furrow	0.030 g ai/m	1	-	At drilling
Potatoes	Italy	GR	50 g/kg	Soil incorporation in furrow	3.0-3.5 2.0 for early potatoes	1	-	At drilling PHI 90 for early potatoes
Potatoes	Spain	GR	100 g/kg	Soil incorporation	4.0 for all the surface of the ground 0.04 g ai/m for the sowing furrow	1	-	Not applicable
Potatoes	Netherlands	GR	100 g/kg	Soil incorporation	4.0	1	-	Before or at planting
					2.0			Before planting

Crop	Country	Formulation		Application				PHI, days and/or Application timing
		Type	Conc. of oxamyl	Method	Rate kg ai/ha	No. max	Interval, days	
				Soil incorporation in furrow	1.0			At planting
Potatoes	UK	GR	100 g/kg	Soil incorporation	5.5 4.0 for early potatoes	1	-	At planting PHI 80
Carrot	Italy	GR	50 g/kg	Soil incorporation in seed furrow	2.5	1	-	Pre-sowing or at drilling
				Soil incorporation in row	3.0-3.5			Prior to transplanting
Carrot, winter carrot	Netherlands	GR	100 g/kg	Soil incorporation	4.0	1	-	Before or at sowing
				Soil incorporation in seed furrow	1.0			At sowing
Carrot and Parsnips	UK	GR	100 g/kg	Soil incorporation in seed furrow	0.090 g ai/m	1	-	At planting PHI 12 weeks

Rotational crop restrictions:

For 50 g/kg GR in Italy; It is recommended that lettuce and similar crops, head and leaf cabbage, and onions be planted or transplanted no sooner than 120 days after application date.

For 100 g/L SL in Spain; Crops can be planted in rotation 30 days after the last application.

\* only in a protected environment (glasshouses or tunnels)

## RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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

Group	Commodity	Table
Brassica vegetables (except Brassica leafy vegetables)	Brussels sprouts	Table 52
Fruiting vegetables, Cucurbits	Cucumber & Courgette (Squash, Summer)	Table 53
	Melons	Table 54
	Tomato	Table 55, 56
Fruiting vegetables, other than Cucurbits	Peppers	Table 57
	Eggplant	Table 58
	Carrot	Table 59
Root and tuber vegetables	Sugar beet	Table 60
	Potato	Table 61

Oxamyl formulation was applied for soil treatment. Each of the field trial sites generally consisted of untreated control plot and treated plot. Application rates and residue concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the applicators used, plot size, field sample size and sampling date.

*Brassica vegetables (except Brassica leafy vegetables)*

*Brussels sprouts*

The Meeting received 3 trials (at harvest trials) on Brussels sprouts which were conducted in Northern Europe (Foster, 2005: DuPont-14669). In each trial, a GR formulation (100 g/kg oxamyl) was applied once by granular applicator to the treated plot. Each application was made at a target rate of 0.5 g ai/m<sup>2</sup> (5.0 kg ai/ha), for a seasonal application rate of 0.5 g ai/m<sup>2</sup> (5.0 kg ai/ha). The application occurred immediately prior to transplanting the young Brussels sprout plants (BBCH 12-14). For all trials, specimens of Brussels sprouts (sprout buttons) were collected at normal commercial harvest (BBCH 49) on the appropriate date. The specimens were collected 158 days after last application (DALA). One control sprout button specimen and one treated sprout button specimen, from a single sampling interval were submitted for analysis.

Specimens were analysed for residues of oxamyl following analytical method No. 0259 described in Report No. DuPont-11125. The LOQ was 0.01 mg/kg and The LOD was 0.007 mg/kg. The mean recovery for oxamyl from Brussels sprout specimens was 95% at 0.01 mg/kg fortified and 91% at 0.1 mg/kg fortified. Treated Brussels sprout samples were stored at -18 ± 5 °C for less than 2 months between sampling and analysis.

Table 52 Residues of oxamyl on Brussels sprouts from supervised trials in Northern Europe

Brussels sprouts country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH* (transplants)	no.			
<i>GAP, Netherlands</i>	<i>GR</i>	<i>4.0</i>		<i>1</i>	<i>Before planting</i>		
Netherlands, 2004 Moerkapelle (Abbacus)	GR	4.59	12-14	1	158	≤0.01 (<LOD)	DuPont-14669
Netherlands, 2004 Rocanje (Abbacus)	GR	4.59	12-14	1	158	≤0.01 (<LOD)	Sampling to analysis: 49 days
Netherlands, 2004 Stellendam (Abbacus)	GR	4.59	12-14	1	158	≤0.01 (<LOD)	

Portion analysed: sprout buttons

\* Growth stage of transplants, planted into treated soil immediately after test item application and incorporation

*Fruiting vegetables, Cucurbits*

*Subgroup of Fruiting vegetables, Cucurbits – Cucumber and Summer squashes*

*Cucumber and courgette*

The Meeting received 11 trials (decline trials) on protected cucumber and 11 trials (decline trials) on protected courgette which were conducted in Southern Europe. In each trial, a SL formulation (100 g/L) was applied twice either *via* watering can with dribble bar followed by drip irrigation or directly using drip irrigation. The applications were made to the treatment plot immediately after transplanting, followed by application occurring at least 10 ± 1 days after application. First application was made at a target rate of 2.0 kg ai/ha. Second application was made at a target rate of 1.0 kg ai/ha for a seasonal application rate of 3.0 kg ai/ha. For all sites, specimens of cucumber/courgette were collected from -0, to 85 days after last application (DALA).

Specimens were analysed for residues of oxamyl following analytical method No. 0259 described in Report No. DuPont-11125 (Boissinot, 2007: DuPont-19518 and Haigh, 2011: DuPont-



29314). The LOQ was 0.01 mg/kg and the LOD was 0.007 mg/kg. Treated cucumber/courgette samples were stored at  $-18 \pm 5$  °C for less than 9 months between sampling and analysis.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 1901 described in Report No. DuPont-33191 (Haigh, 2012: DuPont-31505). The LOQ was 0.01 mg/kg and the LOD was 0.003 mg/kg. Concurrent recoveries from untreated samples fortified at the LOQ (0.01 mg/kg) to as high as 0.50 mg/kg) ranged from 63–96%. Mean recoveries per fortification level/matrix combination ranged from 72–93% for 2 to 3 fortifications per fortification level/matrix combination. Treated specimens were stored at  $-18 \pm 5$  °C for less than 10 months between sampling and analysis.

Table 53 Residues of oxamyl on protected cucumber and courgette from supervised trials in Southern Europe

Cucumber & Courgette country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
<b>Cucumber</b>							
<i>GAP, Italy</i>	SL	1.0-2.0 kg ai/ha max 3.0 kg ai/ha/season		2	50		
Spain, 2006 Los Palacios, Andalucia (Torres) Indoor (greenhouse)	SL	2.1 1.0	12 71 12 65	2	-0 14 21 28 35	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	DuPont-19518  Mean recovery for oxamyl: 77 ± 7% (n=12, cucumber/ courgette) at 0.01 mg/kg 81 ± 10% (n=12, cucumber/ courgette) at 0.1 mg/kg
Italy, 2006 Triginto di Mediglia, Lombardia (Edona) Indoor (plastic tunnel)	SL	2.1 1.0	13 82-85 13 61-64	2	-0 14 21 28 35	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	
Greece, 2006 Profitis Thessaloniki, Central Macedonia (Luberon) Indoor (greenhouse)	SL	2.0 1.0	12-13 74-76 12-13 27-28	2	-0 14 21 28 35	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	Sampling to analysis: 60-209 days
Italy, 2006 Campofilone Marche, Ascoli Piceno (Jazzer) Indoor (plastic tunnel)	SL	2.0 1.0	15 77 15 74	2	-0 14 22 29 36	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	
Spain, 2009 Venta del Viso, Andalucia (Urano) Indoor (plastic greenhouse)	SL	2.1 1.0	12, 61 12, 27 12, 17 12, 15 12, 13	2	30 45 60 75 85	< 0.01 (<LOD) ≤ 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	DuPont-29314  Mean recovery for oxamyl: 81 ± 7% (n=4) at 0.01 mg/kg 82 ± 6% (n=4) at 0.1 mg/kg
Spain, 2010 La Mojonera, Andalucia (Poseidon) Indoor (plastic greenhouse)	SL	2.1 1.0	12, 68 12, 55 12, 23 12, 18 12, 14	2	28 43 58 73 83	< 0.01 (<LOD) ≤ 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	Sampling to analysis: 81-181 days
Greece, 2010 Nea Magnisia, Thessaloniki (Illas) Indoor (greenhouse)	SL	2.0 2.0 1.0	12 12, 14	1 2	43 34	< 0.01 (<LOD) < 0.01 (<LOD)	
Italy, 2010 Contrada Sughero Torto, Vittoria (Setman) Indoor (greenhouse)	SL	2.1 1.0	11, 65 11, 18/51 11, 15 11, 13 11, 12	2	31 44 57 73 76	< 0.01 (<LOD) <u>0.016</u> < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	
Spain, 2010 La Mojonera, Andalucia	SL	2.1 1.0	12, 69 12, 57	2	28 43	< 0.01 (<LOD) ≤ 0.01 (<LOD)	

Cucumber & Courgette country, year (variety) (Augie)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Indoor (plastic greenhouse)			12, 25		58	< 0.01 (<LOD)	DuPont-31505  Mean recovery for oxamyl: 72% (n=2) at 0.01 mg/kg 87% (n=2) at 0.1 mg/kg
			12, 21		73	< 0.01 (<LOD)	
			12, 14		83	< 0.01 (<LOD)	
Italy, 2010 Contrada Randello, Sicily (Solverde) Indoor (greenhouse)	SL	2.1	12	1	73	< 0.01 (<LOD)	
		1.1	12, 67	2	14	< 0.01 (0.009)	
			12, 64		21	< 0.01 (<LOD)	
			12, 63		28	< 0.01 (<LOD)	
			12, 61		35	< 0.01 (<LOD)	
			12, 16		46	< 0.01 (<LOD)	
12, 13	63	< 0.01 (<LOD)					
Spain, 2010 La Mojonera, Andalucia (Urano) Indoor (plastic greenhouse)	SL	2.1	13-14	1	65	< 0.01 (<LOD)	
		1.1	13-14, 63	2	14	0.11	
			13-14, 55		21	0.037	
			13-14, 51		28	< 0.01 (0.006)	
			13-14, 22		35	< 0.01 (0.004)	
			13-14, 21		45	< 0.01 (<LOD)	
			13-14, 15-16		55	< 0.01 (<LOD)	
Courgette							
<i>GAP, Italy</i>	<i>SL</i>	<i>1.0-2.0 kg ai/ha max 3.0 kg ai/ha/season</i>		<i>2</i>	<i>50</i>		
Spain, 2006 Los Palacios, Andalucia (Helida) Indoor (greenhouse)	SL	2.1	12	2	-0	Not sampled	DuPont-19518  Mean recovery for oxamyl: 77 ± 7% (n=12, cucumber/ courgette) at 0.01 mg/kg 81 ± 10% (n=12, cucumber/ courgette) at 0.1 mg/kg
		1.0	19		14	< 0.01 (0.008)	
			12		21	< 0.01 (<LOD)	
			14		28	< 0.01 (<LOD)	
Italy, 2006 Roncoferraro, Lomabardia (President) Indoor (plastic tunnel)	SL	2.1	14	2	-0	< 0.01 (<LOD)	
		1.0	70		14	< 0.01 (<LOD)	
			14		21	< 0.01 (<LOD)	
			51		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
Greece, 2006 Profitis Thessaloniki, Central Macedonia (Sivas) Indoor (greenhouse)	SL	2.0	12	2	-0	< 0.01 (<LOD)	
		1.0	75		14	< 0.01 (<LOD)	
			12		22	< 0.01 (<LOD)	
			67-71		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
Italy, 2006 Corropoli Abruzzo, Teramo (Gryzine) Indoor (plastic tunnel)	SL	2.0	18	2	-0	< 0.01 (<LOD)	
		1.0	77		14	< 0.01 (<LOD)	
			18		21	< 0.01 (<LOD)	
			74		29	< 0.01 (<LOD)	
					36	< 0.01 (<LOD)	
Spain, 2009 Berja, Andalucia (Sinatra) Indoor (plastic greenhouse)	SL	2.1	11, 65	2	30	0.023	
		1.0	11, 63		45	< 0.01 (<LOD)	
			11, 51		60	< 0.01 (<LOD)	
			11, 19		75	< 0.01 (<LOD)	
			11, 14		85	< 0.01 (<LOD)	
Spain, 2009 La Mojonera, Andalucia (Capea) Indoor (plastic greenhouse)	SL	2.1	12	1	67	< 0.01 (<LOD)	
		1.0	12, 16		2	32	0.014
			12, 15			47	< 0.01 (<LOD)
			12, 14			57	< 0.01 (<LOD)
Greece, 2010 Nea Magnisia, Thessaloniki (ARO-800) Indoor (greenhouse)	SL	2.0	12	1	43	< 0.01 (<LOD)	
		1.0	12, 14	2	34	< 0.01 (<LOD)	
Italy, 2009 Contrada Moglie Dirillo, Acate (Richgreen) Indoor (greenhouse)	SL	1.0	12, 73	2	31	< 0.01 (<LOD)	
			12, 71		43	< 0.01 (<LOD)	
			12, 61		57	< 0.01 (<LOD)	
			12, 19		72	< 0.01 (<LOD)	
			12, 19		77	< 0.01 (<LOD)	

Cucumber & Courgette country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Spain, 2010 El Ejido, Andalucia (Sinatra) Indoor (plastic greenhouse)	SL	2.1	12-13	1	65	< 0.01 (0.004)	DuPont-31505  Mean recovery for oxamyl: 78 ± 7% (n=3) at 0.01 mg/kg 82 ± 4% (n=3) at 0.1 mg/kg 86 ± 5% (n=3) at 0.2 mg/kg 93% (n=2) at 0.5 mg/kg
		2.1 1.1	12-13, 65	2	14	0.44	
			12-13, 61		21	0.11	
			12-13, 51		28	0.35	
			12-13, 22		35	0.071	
			12-13, 19		45	<u>0.022</u>	
			12-13, 15-16		55	< 0.01 (0.009)	
Spain, 2010 Puebla de Vicar, Andalucia (Natura) Indoor (plastic greenhouse)	SL	2.1	12	1	65	< 0.01 (<LOD)	0.1 mg/kg 86 ± 5% (n=3) at 0.2 mg/kg 93% (n=2) at 0.5 mg/kg
		2.1 1.1	12, 63	2	15	0.16	
			12, 52		21	0.079	
			12, 25		28	0.14	
			12, 21		35	< 0.01 (0.007)	
			12, 17		45	< 0.01 (0.005)	
			12, 14-15		55	< 0.01 (<LOD)	
Greece, 2010 Vasilika, Central Macedonia (ARO800) Indoor (greenhouse)	SL	2.1	13	1	49	< 0.01 (<LOD)	Sampling to analysis: 246-274 days
		2.1 1.0	13, 51/61	2	14	0.015	
			13, 19/51		23	< 0.01 (<LOD)	
			13, 18/19		30	< 0.01 (<LOD)	
			13, 16		37	< 0.01 (<LOD)	
			13, 14/16		39	< 0.01 (<LOD)	

Portion analysed: fruit

-0: on the day of the last application, directly before the application took place

### *Subgroup of Fruiting vegetables, Cucurbits – Melons, Pumpkins and Winter squashes*

#### *Melons*

The Meeting received 13 trials (decline trials) on protected melons which were conducted in Southern Europe (Haigh, 2011 and 2012). In each trial, a SL formulation (100 g/L oxamyl) was applied at a target rate of 2.0 kg ai/ha immediately after transplanting with subsequent applications of 1.0 kg ai/ha. All applications were made in acidified water (pH 5–6) *via* drip irrigation systems. There were five applications with a 10 ± 1 day re-treatment interval.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125 (Haigh, 2011: DuPont-29316). The LOQ was 0.005 mg/kg and the LOD was 0.0033 mg/kg. Treated melon fruit samples were stored at -18 ± 5 °C for less than 9 months between sampling and analysis.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 1901 described in Report No. DuPont-33191 (Haigh, 2012: DuPont-31508). The LOQ was 0.005 mg/kg and the LOD was 0.0015 mg/kg. Treated specimens were stored at *ca* -18 °C for less than 8 months between sampling and analysis.

Table 54 Residues of oxamyl on protected melons from supervised trials in Southern Europe

Melons country, year (variety)	Application				DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	BBCH	no.		Peel Flesh	Whole*	
<i>GAP, Italy &amp; Spain</i>	<i>SL</i>	<i>1.0-2.0 kg ai/ha max 3.0 kg ai/ha</i>	<i>2</i>	<i>50</i>				
Spain, 2009 Puebla de Vicar, Andalucia (Monzon) Indoor (plastic greenhouse)	SL	2.1	12	1	112	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	DuPont-29316  Mean recovery for oxamyl: Peel
		2.1 1.0	12, 65	2	52	0.0074 < 0.005 (<LOD)	<u>0.0053</u>	
			12, 55-62		62	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	

Melons country, year (variety)	Application				DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	BBCH	no.		Peel Flesh	Whole*	
		2.1 1.0 × 2	12, 52, 54- 55	3	67	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	85±19% (n=8) at 0.005 mg/kg 87 ± 6% (n=8) at 0.10 mg/kg Flesh 83 ± 11% (n=8) at 0.005 mg/kg 84 ± 8% (n=7) at 0.10 mg/kg
		2.1 1.0 × 4	12, 19, 19, 52-53, 55- 62	5	62	< 0.005 (0.004) < 0.005 (<LOD)	< 0.005 (0.004)	
			12, 15, 19, 19, 52-53		72	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Spain, 2009 La Mojonera, Andalucia (Homer) Indoor (plastic greenhouse)	SL	2.1 1.0	12, 12	2	88	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	Sampling to analysis: 50-133 days
		2.1 1.0 × 2	12, 12, 19	3	78	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 3	12, 12, 19, 24	4	68	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 4	12, 24, 51, 61, 63	5	38	0.026 0.014	0.019	
			12, 19, 22, 25, 53		53	< 0.005 (0.004) < 0.005 (<LOD)	< 0.005 (0.0034)	
			12, 12, 19, 24, 51		58	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Spain, 2009 Las Norias de Daza, Andalucia (Siglo) Indoor (plastic greenhouse)	SL	2.0	13	1	103	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0	13, 68	2	43	0.005 < 0.005 (<LOD)	< 0.005 (0.004)	
			13, 67		53	< 0.005 (0.004) < 0.005 (<LOD)	< 0.005 (0.004)	
		2.1 1.0 × 2	13, 65, 66	3	58	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 4	13, 19-51, 51, 66, 67	5	53	< 0.005 (0.004) < 0.005 (<LOD)	< 0.005 (0.004)	
			13, 14, 19- 51, 51, 66		63	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Spain, 2009 Puebla de Vicar, Andalucia (Cantalup) Indoor (plastic greenhouse)	SL	2.0 1.0	13, 14	2	97	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 2	13, 14, 21	3	87	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 3	13, 14, 21, 29-51	4	77	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 4	13, 29-51, 54, 61, 64	5	47	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			13, 19, 23, 52, 56		62	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			13, 14, 21, 29-51, 54		67	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Spain, 2009 El Ejido, Andalucia (Valverde) Indoor (plastic greenhouse)	SL	2.1 1.0	12, 15	2	82	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 2	12, 15, 22	3	72	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 3	12, 15, 22, 51	4	62	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 ×	12, 51, 64, 68, 69	5	32	0.0068 < 0.005 (<LOD)	0.005	

Melons country, year (variety)	Application				DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	BBCH	no.		Peel Flesh	Whole*	
		4	12, 19, 25, 62, 66		47	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			12, 15, 22, 51, 64		52	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Greece, 2010 Kolchiko, Thessaloniki (Galia F1) Indoor (greenhouse)	SL	2.0	12-13	1	61	0.013 < 0.005 (<LOD)	0.0067	
		2.0 1.0	12-13, 19	2	51	0.061 < 0.005 (<LOD)	< 0.005 (0.004)	
		2.0 1.0 × 2	12-13, 19, 61	3	41	0.018 0.018	0.018	
		2.1 1.0 × 3	12-13, 19, 61, 64	4	31	0.027 0.0080	0.014	
		2.1 1.0 × 4	12-13, 19, 61, 64, 69	5	21	0.005 0.014	0.011	
Greece, 2010 Boutaika, Achaia (Citirex) Indoor (greenhouse)	SL	2.0	12-13	1	63	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.0 1.0	12-13, 19	2	52	0.005 < 0.005 (<LOD)	< 0.005 (0.004)	
		2.0 1.0 × 2	12-13, 19, 61	3	43	0.0083 0.010	0.0094	
		2.0 1.0 × 3	12-13, 19, 61, 65	4	34	0.017 0.014	0.015	
		2.0 1.0 × 4	12-13, 19, 61, 65, 72	5	23	0.019 0.030	0.026	
Italy, 2010 Contrada Chiappa, Acate, Sicily (Solemio) Indoor (greenhouse)	SL	2.1 1.0	12, 13	2	83	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 2	12, 13, 14	3	73	0.0080 < 0.005 (<LOD)	0.0057	
		2.1 1.0 × 3	12, 13, 14, 16	4	63	< 0.005 (0.004) < 0.005 (<LOD)	< 0.005 (0.004)	
		2.1 1.0 × 4	12, 16, 23, 63, 65	5	31	0.098 0.028	0.066	
			12, 14, 14, 18, 61		48	0.046 0.011	0.030	
			12, 13, 14, 16, 23		52	0.0060 0.0057	0.0059	
Italy, 2010 Contrada Dirillo, Acate, Sicily (Cabrero) Indoor (greenhouse)	SL	2.1 1.0	12, 13	2	84	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 2	12, 13, 16	3	74	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 3	12, 13, 16, 61	4	63	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
		2.1 1.0 × 4	12, 61, 63, 65, 66	5	33	0.0093 0.0091	0.0092	
			12, 13, 17, 62, 65		49	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			12, 13, 16, 61, 63		53	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Italy, 2010 Contrada Dirillo, Acate, Sicily (Cabrero) Indoor	SL	2.1 1.1	12, 76	2	20	0.054 0.031	0.044	DuPont-31508
			12, 73		28	0.009 0.005	0.0071	Mean recovery

Melons country, year (variety) (greenhouse)	Application				DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	BBCH	no.		Peel Flesh	Whole*	
			12, 64		38	< 0.005 (0.002) < 0.005 (<LOD)	< 0.005 (0.002)	for oxamyl: Peel 90 ± 18% (n=5) at 0.005 mg/kg 93 ± 28% (n=5) at 0.10 mg/kg Flesh 76 ± 11% (n=3) at 0.005 mg/kg 82 ± 32% (n=3) at 0.10 mg/kg  Sampling to analysis: 186- 226 days
			12, 62		48	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			12, 61		59	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Spain, 2010 El Ejido, Andalucia (Monzon) Indoor (plastic greenhouse)	SL	2.1 1.1	12, 67	2	21	0.006 0.005	0.0057	
			12, 63		28	< 0.005 (0.004) < 0.005 (0.003)	< 0.005 (0.003)	
			12, 53		40	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			12, 51		50	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
			12, 24		60	< 0.005 (<LOD) < 0.005 (<LOD)	< 0.005 (<LOD)	
Spain, 2010 El Ejido, Andalucia (Magenta) Indoor (plastic greenhouse)	SL	2.1 1.1	13, 67	2	21	0.074 0.019	0.040	
			13, 63		28	0.032 0.007	0.017	
			13, 52		40	0.016 < 0.005 (0.003)	0.0078	
			13, 29		50	0.011 < 0.005 (0.002)	<u>0.0054</u>	
			13, 25		60	< 0.005 (0.002) < 0.005 (<LOD)	< 0.005 (0.002)	
Spain, 2011 Puebla de Vicar, Andalucia (Brisa) Indoor (plastic greenhouse)	SL	2.1 1.1	13, 63	2	21	0.025 0.024	0.024	
			13, 55		28	< 0.005 (0.003) < 0.005 (0.003)	< 0.005 (0.003)	
			13, 51		41	< 0.005 (0.002) < 0.005 (<LOD)	< 0.005 (0.002)	
			13, 26		50	< 0.005 (0.003) < 0.005 (<LOD)	< 0.005 (0.002)	
			13, 22		60	< 0.005 (0.003) < 0.005 (<LOD)	< 0.005 (0.002)	

Portion analysed: Peel (up), Flesh (down)

\* For the whole commodity, residues were calculated from flesh and peel residue concentration and fraction weights:  
(mg/kg) whole = [(mg/kg)flesh + [(mg/kg)peel × (kg peel/kg flesh)]] / [1 + (kg peel/kg flesh)]

### Fruiting vegetables, other than Cucurbits

#### Subgroup of Tomatoes

##### Tomato

The Meeting received 12 trials (decline trials) on protected cherry tomatoes and 21 trials (decline trials) on protected tomatoes which were conducted in Southern Europe (Boissinot *et al.*, 2007 and Haigh *et al.*, 2011, 2012). In each trial, a SL formulation (100 g/L oxamyl) was applied three to four times *via* drip irrigation or watering can with dribble bar followed by drip irrigation. The first application was made to each treated plot immediately after transplanting. Treatment plots at each test site received two or three additional applications occurring at *ca.* 10-day intervals triggered by the predicted first commercial harvest. The oxamyl rate of first application was targeted at a rate of 2.0 kg ai/ha. Oxamyl applications 2–4 were applied at a target rate of 1.0 kg ai/ha for a seasonal application rate of 4.0–5.0 kg ai/ha.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125 (Boissinot *et al.*, 2007: DuPont-19521, DuPont-19519 Revision No. 1 and Haigh *et al.*, 2011: DuPont-29313). The LOQ was 0.01 mg/kg and the LOD was 0.007 mg/kg

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 1901 described in Report No. DuPont-33191 (Haigh *et al.*, 2012: DuPont-31506). The LOQ was 0.010 mg/kg and the LOD was 0.003 mg/kg.

Treated protected tomatoes samples were stored at  $-18 \pm 5$  °C for less than 8 months between sampling and analysis.

Table 55 Residues of oxamyl on protected cherry tomatoes and tomatoes from supervised trials in Southern Europe

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
<i>GAP, Italy &amp; Spain</i>	<i>SL</i>	<i>1.0-2.0 kg ai/ha max 5.0 kg ai/ha/season</i>		<i>4</i>	<i>28</i>		
Cherry tomato							
Spain, 2006 Los Palacios, Andalucia (Lupita) Indoor (greenhouse)	SL	2.1 1.0 × 2	12, 71, 81	3	-0	< 0.01 (<LOD)	DuPont-19521  Mean recovery for oxamyl: 76 ± 8% (n=6) at 0.010 mg/kg 76 ± 8% (n=4) at 0.10 mg/kg  Sampling to analysis: 50-176 days
			12, 66, 71		21	< 0.01 (<LOD)	
			12, 63, 66		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
	2.1 1.0 × 3	12, 65, 71, 81	4	-0	0.019	< 0.01 (<LOD)	
				21	< 0.01 (<LOD)		
		12, 63, 66, 71	28	< 0.01 (<LOD)			
			35	< 0.01 (<LOD)			
			42	< 0.01 (<LOD)			
			49	< 0.01 (<LOD)			
Italy, 2006 Via San Martino, Grottammare, Marche, Ascoli Piceno (Carminio de reuter) Indoor (plastic tunnel)	SL	2.0 1.0 × 2	18, 64, 81	3	-0	< 0.01 (<LOD)	
			18, 51, 77		21	< 0.01 (<LOD)	
			18, 25, 54		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
	2.1 1.0 × 3	18, 29, 64, 82	4	-0	< 0.01 (<LOD)		
				21	< 0.01 (<LOD)		
		18, 24, 51, 77	28	< 0.01 (<LOD)			
			35	< 0.01 (<LOD)			
			41	< 0.01 (<LOD)			
			49	< 0.01 (<LOD)			
Greece, 2006 Profitis Thessaloniki, Central Macedonia (Winner) Indoor (greenhouse)	SL	2.0 1.0 × 2	12-13, 66-72, 72-74	3	-0	< 0.01 (<LOD)	
			12-13, 63-65, 67-73		21	< 0.01 (<LOD)	
			12-13, 52-54, 65-71		29	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
	2.0 1.0 × 3	12-13, 60-63, 66-72, 72-74	4	-0	< 0.01 (<LOD)		
				21	< 0.01 (<LOD)		
		12-13, 25-27, 63-65, 67-73	29	< 0.01 (<LOD)			
			36	< 0.01 (<LOD)			
			43	< 0.01 (<LOD)			
			50	< 0.01 (<LOD)			
Greece, 2006 Nea Magnisia, Thessaloniki, Central	SL	2.0 1.0 × 2	12-13, 62-64, 66-73	3	-0	< 0.01 (<LOD)	
					20	< 0.01 (<LOD)	
		12-13, 55-57, 64-72	28		< 0.01 (<LOD)		

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Macedonia (Sanny) Indoor (greenhouse)			12-13, 26-27, 61-64		34 41 49	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	
		2.0 1.0 × 3	12-13, 52-55, 62-64, 66-73	4	-0 20	< 0.01 (<LOD) < 0.01 (<LOD)	
			12-13, 26-27, 55-57, 64-72		28 35 42 49	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	
Spain, 2010 Castell de Ferro, Andalucia (Cupido) Indoor (plastic greenhouse)	SL	2.1, 1.0	14, 15-51	2	87	< 0.01 (<LOD)	DuPont-29313  Mean recovery for oxamyl: 85 ± 6% (n=10, 6 tomato and 4 cherry tomato) at 0.010 mg/kg 83 ± 4% (n=10, 6 tomato and 4 cherry tomato) at 0.1 mg/kg
		2.1 1.0 × 2	14, 15-51, 52	3	77	< 0.01 (<LOD)	
		2.1 1.0 × 3	14, 65, 67, 69	4	37	< 0.01 (<LOD)	
			14, 61, 64, 65 14, 15-51, 52, 62		52 67	< 0.01 (<LOD) < 0.01 (<LOD)	
Spain, 2010 Castell de Ferro, Andalucia (DRC 524) Indoor (plastic house)	SL	2.1 1.0	13, 17-51	2	98	< 0.01 (<LOD)	Sampling to analysis: 61-134 days
		2.1 1.0 × 2	13, 17-51, 54	3	88	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 62, 63, 64	4	48	< 0.01 (<LOD)	
			13, 61, 62, 62 13, 17-51, 54, 62		63 78	< 0.01 (<LOD) < 0.01 (<LOD)	
Spain, 2010 Albuñol. Andalucia (Catalina) Indoor (plastic greenhouse)	SL	2.1 1.0	12, 13	2	108	< 0.01 (<LOD)	
		2.1 1.0 × 2	12, 13, 16	3	98	< 0.01 (<LOD)	
		2.1 1.0 × 3	12, 52, 61, 65	4	58	< 0.01 (<LOD)	
			12, 17, 51, 53 12, 13, 16, 19		73 88	< 0.01 (<LOD) < 0.01 (<LOD)	
Greece, 2010 Nea Magnisia, Thessaloniki (Corbus) Indoor (greenhouse)	SL	2.0	16	1	70	< 0.01 (<LOD)	
		2.0 1.0	16, 52/61	2	55	< 0.01 (<LOD)	
		2.0 1.0 × 2	16, 52/61, 53/61	3	47	< 0.01 (<LOD)	
		2.0 1.0 × 3	16, 53/61, 62, 63 16, 18/51, 53/61, 62	4	28 37	< 0.01 (<LOD) < 0.01 (<LOD)	
Italy, 2010 Contrada, Dirillo, Vittoria, Sicily (Genio) Indoor (greenhouse)	SL	2.1 1.0 × 3	13, 63, 65, 66	4	31	< 0.01 (<LOD)	
			13, 62, 63, 64		48	< 0.01 (<LOD)	
			13, 61, 61, 62		62	< 0.01 (<LOD)	
			13, 14, 18, 61		76	< 0.01 (<LOD) < 0.01 (<LOD)	
Italy, 2011 Contrada Rinazze di Strada, Vittoria, Sicily (Genio) Indoor (greenhouse)	SL	2.1 1.1 × 3	13, 63, 64, 65	4	20	< 0.01 (0.005)	DuPont-31506  Mean recovery for oxamyl: 91 ± 21% (n=4) at 0.010 mg/kg 80 ± 8% (n=4) at 0.10 mg/kg
			13, 63, 63, 64		30	< 0.01 (<LOD)	
			13, 61, 62, 63		43	< 0.01 (<LOD)	
			13, 15, 17, 62		58	< 0.01 (<LOD)	
Spain, 2011 Campohermoso, Andalucia (Genio) Indoor (plastic greenhouse)	SL	2.1 1.1 × 3	13, 66, 69, 72	4	21	< 0.01 (<LOD)	
			13, 62, 66, 69		30	< 0.01 (<LOD)	
			13, 51, 61, 63		45	< 0.01 (<LOD)	
			13, 24, 28, 53		60	< 0.01 (<LOD)	



Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Spain, 2011 Campohermoso, Andalucia (Santawest) Indoor (plastic greenhouse)	SL	2.1	13, 66, 69, 72	4	21	< 0.01 (0.003)	Sampling to analysis: 208-219 days
		1.1 × 3	13, 62, 66, 69		30	< 0.01 (<LOD)	
			13, 51, 61, 63		45	< 0.01 (<LOD)	
			13, 24, 28, 53		60	< 0.01 (<LOD)	
Tomato							
Spain, 2006 Los Palacios, Andalucia (Eldiez) Indoor (greenhouse)	SL	2.1	12, 64, 72	3	-0	< 0.01 (<LOD)	DuPont-19519, Revision No. 1  Mean recovery for oxamyl: 76 ± 14% (n=7) at 0.010 mg/kg 77 ± 13% (n=8) at 0.10 mg/kg
		1.0 × 2	12, 62, 65		21	< 0.01 (<LOD)	
			12, 61, 65		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
			42	< 0.01 (<LOD)			
			49	< 0.01 (<LOD)			
		2.1	12, 61, 64, 72	4	-0	< 0.01 (<LOD)	
	1.0 × 3	12, 61, 62, 65	21		< 0.01 (<LOD)		
		28	< 0.01 (<LOD)				
		35	< 0.01 (<LOD)				
		42	< 0.01 (<LOD)				
		49	< 0.01 (<LOD)				
Italy 2006 Triginto di Mediglia, Lombardia (Oskar) Indoor (plastic tunnel)	SL	2.1	14, 64, 72	3	-0	< 0.01 (0.008)	Sampling to analysis: 49-127 days
		1.0 × 2	14, 63, 64		21	< 0.01 (<LOD)	
			14, 19-51, 63-64		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
			42	< 0.01 (<LOD)			
			49	< 0.01 (<LOD)			
		2.1	14, 62, 64, 72	4	-0	0.031	
	1.0 × 3	14, 19, 63, 64	21		< 0.01 (<LOD)		
		28	< 0.01 (<LOD)				
		35	< 0.01 (<LOD)				
		42	< 0.01 (<LOD)				
		49	< 0.01 (<LOD)				
Italy, 2006 Roconferraro, Lombardia (Carso) Indoor (plastic tunnel)	SL	2.1	14, 61, 63-71	3	-0	< 0.01 (<LOD)	
		1.0 × 2	14, 52, 62		21	< 0.01 (<LOD)	
			14, 52, 61		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
			42	< 0.01 (<LOD)			
			49	< 0.01 (<LOD)			
		2.1	14, 52, 61, 63-71	4	-0	< 0.01 (<LOD)	
	1.0 × 3	14, 21, 52, 62	21		< 0.01 (<LOD)		
		28	< 0.01 (<LOD)				
		35	< 0.01 (<LOD)				
		42	< 0.01 (<LOD)				
		49	< 0.01 (<LOD)				
Spain, 2006 Belcaire d' Urgell, Lleida (Caramba) Indoor (plastic tunnel)	SL	2.1	13-14, 72, 73	3	-0	< 0.01 (<LOD)	
		1.0 × 2	13-14, 51, 72-73		21	< 0.01 (<LOD)	
			13-14, 26-27, 22		28	< 0.01 (<LOD)	
					35	< 0.01 (<LOD)	
			42	< 0.01 (<LOD)			
			49	< 0.01 (<LOD)			
		2.1	13-14, 29-51, 72, 73	4	-0	< 0.01 (<LOD)	
	1.0 × 3	13-14, 19, 51, 72-73	21		< 0.01 (<LOD)		
		28	< 0.01 (<LOD)				
		35	< 0.01 (<LOD)				
		42	< 0.01 (<LOD)				
		49	< 0.01 (<LOD)				
Spain, 2006 Partida foutanet, Lleida (Caramba) Indoor (greenhouse)	SL	2.1	13-14, 71-72, 75	3	-0	< 0.01 (<LOD)	
		1.0 × 2	13-14, 61, 72		21	< 0.01 (<LOD)	
			13-14, 51, 63-64		28	< 0.01 (<LOD)	
			35	< 0.01 (<LOD)			
			42	< 0.01 (<LOD)			
		49	< 0.01 (<LOD)				
	2.1	13-14, 60, 71-72, 75	4	-0	< 0.01 (<LOD)		

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref			
	Form	kg ai/ha	BBCH	no.						
		1.0 × 3	13-14, 51, 61, 72		21	< 0.01 (<LOD)				
					28	< 0.01 (<LOD)				
					35	< 0.01 (<LOD)				
					42	< 0.01 (<LOD)				
					49	< 0.01 (<LOD)				
Greece, 2006 Profitis, Thessaloniki, Central Macedonia (Belladona) Indoor (greenhouse)	SL	2.0 1.0 × 2	12-13, 53-61, 66-72 12-13, 51-52, 64-71 12-13, 16, 53	3	0	< 0.01 (<LOD)				
					21	< 0.01 (<LOD)				
					28	< 0.01 (<LOD)				
					35	< 0.01 (<LOD)				
					42	< 0.01 (<LOD)				
					49	< 0.01 (<LOD)				
	2.0 1.0 × 3	12-13, 51, 53-61, 66-72 12-13, 16, 51-52, 64-71	4	0	< 0.01 (<LOD)					
				21	< 0.01 (<LOD)					
				28	< 0.01 (<LOD)					
				35	< 0.01 (<LOD)					
Greece, 2006 Nea Magnisia, Thessaloniki, Central Macedonia (Alma) Indoor (greenhouse)	SL	2.0 1.0 × 2	12-13, 62-63, 66-72 12-13, 52, 64-71 12-13, 25-26, 60-62	3	0	< 0.01 (<LOD)				
					21	< 0.01 (<LOD)				
					28	< 0.01 (<LOD)				
					35	< 0.01 (<LOD)				
					42	< 0.01 (<LOD)				
					49	< 0.01 (<LOD)				
	2.0 1.0 × 3	12-13, 26-27, 62-63, 66-72 12-13, 25-26, 52, 64-71	4	0	< 0.01 (<LOD)					
				21	< 0.01 (<LOD)					
				28	< 0.01 (<LOD)					
				35	< 0.01 (<LOD)					
Italy, 2006 Contrada Pozzo Bollente, Victria, Sicily (Panarea) Indoor (plastic greenhouse)	SL	2.0 1.0 × 2	12, 64, 67 12, 62, 65 12, 61, 63	3	0	0.010				
					21	< 0.01 (<LOD)				
					28	< 0.01 (<LOD)				
					35	< 0.01 (<LOD)				
					42	< 0.01 (<LOD)				
					49	< 0.01 (<LOD)				
	2.0 1.0 × 3	12, 62, 64, 67 12, 61, 62, 65	4	0	0.012					
				21	< 0.01 (<LOD)					
				28	< 0.01 (<LOD)					
				35	< 0.01 (<LOD)					
Spain, 2009 Las Norias de Daza, Andalucia (Zinac) Indoor (plastic greenhouse)	SL	2.1 1.0	12, 72 12, 53, 53-71 12, 19, 52, 53/71 12, 16, 19, 51 12, 14, 16, 19	2	57	< 0.01 (<LOD)	DuPont-29313  Mean recovery for oxamyl: 85 ± 6% (n=10, 6 tomato and 4 cherry tomato) at 0.010 mg/kg 83 ± 4% (n=10, 6 tomato and 4 cherry tomato) at 0.1 mg/kg  Sampling to analysis: 96-212 days			
					3	62		< 0.01 (<LOD)		
						4		67	< 0.01 (<LOD)	
								82	< 0.01 (<LOD)	
					1.0 × 3	12, 19, 51, 53/71 12, 18, 19, 51 12, 14, 18, 19		4	92	< 0.01 (<LOD)
									2	68
	3	73	< 0.01 (<LOD)							
		4	78	< 0.01 (<LOD)						
	3		93	< 0.01 (<LOD)						
			103	< 0.01 (<LOD)						
Spain, 2009 La Mojonera, Andalucia (Denis)	SL	2.1 1.0	15, 52 15, 51, 52	2	55	< 0.01 (<LOD)				
					3	60	< 0.01 (<LOD)			

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Indoor (plastic greenhouse)		1.0 × 2					
		2.1	15, 19, 19, 51	4	65	< 0.01 (<LOD)	
		1.0 × 3	15, 19, 19, 19		80	< 0.01 (<LOD)	
			15, 17, 19, 19		90	< 0.01 (<LOD)	
Spain, 2009 Puebla de Vicar, Andalucia (Enate) Indoor (plastic greenhouse)	SL	2.1 1.0	13, 72	2	54	< 0.01 (<LOD)	
		2.1 1.0 × 2	13, 52/71, 72	3	59	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 19, 32, 71	4	64	< 0.01 (<LOD)	
			13, 17, 18, 31		79	< 0.01 (<LOD)	
			13, 15, 17, 18		89	< 0.01 (<LOD)	
Spain, 2009 Ruescas, Andalucia (Octydia) Indoor (plastic greenhouse)	SL	2.1	13	1	105	< 0.01 (<LOD)	
		2.1 1.0 × 2	13, 64, 68	3	45	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 51, 59, 65	4	50	< 0.01 (<LOD)	
			13, 16, 19, 53		65	< 0.01 (<LOD)	
			13, 14, 16, 19		75	< 0.01 (<LOD)	
Spain, 2009 Ruescas, Andalucia (Pristila) Indoor (plastic greenhouse)	SL	2.1	13	1	105	< 0.01 (<LOD)	
		2.1 1.0 × 2	13, 65, 68	3	45	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 51, 59, 66	4	50	< 0.01 (<LOD)	
			13, 16, 19, 53		65	< 0.01 (<LOD)	
			13, 14, 16, 19		75	< 0.01 (<LOD)	
Spain, 2009 Ruescas, Andalucia (Tya) Indoor (plastic greenhouse)	SL	2.1	14	1	107	< 0.01 (<LOD)	
		2.1 1.0 × 2	14, 65, 68	3	47	< 0.01 (<LOD)	
		2.1 1.0 × 3	14, 51, 59, 65	4	52	< 0.01 (<LOD)	
			14, 17, 19, 53		67	< 0.01 (<LOD)	
			14, 15, 17, 19		77	< 0.01 (<LOD)	
Greece, 2010 Nea Magnisia, Thessaloniki (Victor) Indoor (greenhouse)	SL	2.0	18	1	70	< 0.01 (<LOD)	
		2.0 1.0	18, 52/61	2	55	< 0.01 (<LOD)	
		2.0 1.0 × 2	18, 52/61, 53/61	3	47	< 0.01 (<LOD)	
		2.0 1.0 × 3	18, 53/61, 62, 63	4	28	< 0.01 (<LOD)	
			18, 19/51, 53/61, 62		37	< 0.01 (<LOD)	
Italy, 2009 Contrada Bosco Rotondo, Sicily (Rovente) Indoor (greenhouse)	SL	2.1 1.0 × 3	12, 61, 63, 64	4	31	< 0.01 (<LOD)	
			12, 61, 61, 62		46	< 0.01 (<LOD)	
			12, 16, 19, 61		60	< 0.01 (<LOD)	
			12, 14, 15, 18		74	< 0.01 (<LOD)	
Spain, 2010-2011 El Ejido, Andalucia (Zinac) Indoor (plastic greenhouse)	SL	2.1	13, 66, 69, 72	4	21	< 0.01 (0.003)	DuPont-31506  Mean recovery for oxamyl: 91 ± 21% (n=4) at 0.010 mg/kg 80 ± 8% (n=4) at 0.10 mg/kg
		1.1 × 3	13, 64, 66, 69		31	< 0.01 (<LOD)	
			13, 51, 62, 65		45	< 0.01 (<LOD)	
			13, 25, 29, 53		60	< 0.01 (<LOD)	
Spain, 2010-2011 Venta del Viso, Andalucia (Mayoral)	SL	2.1	12-13, 66, 69, 72	4	21	< 0.01 (<LOD)	
		1.1 × 3	12-13, 62, 68, 69		30	< 0.01 (<LOD)	
			12-13, 51, 58, 65		45	< 0.01 (<LOD)	

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Indoor (plastic greenhouse)			12-13, 29, 29, 55		60	< 0.01 (<LOD)	Sampling to analysis: 200-232 days
Spain, 2011 La Mojonera, Andalucia (Realeza)	SL	2.1	14, 67, 69, 72	4	21	< 0.01 (0.006)	
Indoor (plastic greenhouse)	1.1 × 3	14, 61, 67, 69	30		≤ 0.01 (0.004)		
		14, 51, 56, 64	44		< 0.01 (<LOD)		
		14, 22, 28, 54	60		< 0.01 (<LOD)		
Greece, 2010 Almyros, Central Greece (Belladonna)	SL	2.1	12, 16/51, 52/61	3	84	< 0.01 (<LOD)	
Indoor (greenhouse)	1.1 × 2	12, 52-53/61, 53-54/63, 54-55/64	4		45	< 0.01 (<LOD)	
		12, 52-53/61, 52-53/62, 53-54/63			54	< 0.01 (<LOD)	
		12, 19/61, 52/61, 52-53/61			69	< 0.01 (<LOD)	

Portion analysed: fruit

-0: on the day of the last application, directly before the application took place

The Meeting received eight trials (decline trials) on protected tomatoes which were conducted in Southern Europe (Françon *et al.*, 2001: DuPont-4583). One application of oxamyl (as 50 g/kg GR formulation) was performed at planting, at rates of either 3 kg ai/ha (in-furrow application) or 5.5 kg ai/ha (broadcast application). The plots were sampled at earliest normal harvest.

Oxamyl residue concentrations were determined in tomato fruits by HPLC with post-column derivatization followed by fluorescence detection (HPLC-PCD/Fluo) according to the method validated in DuPont-4722. The LOQ was 0.010 mg/kg and the LOD was 0.005 mg/kg.

Table 56 Residues of oxamyl on protected tomatoes from supervised trials in Southern Europe

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
<i>GAP, Italy</i>	<i>GR</i>	<i>4.5-5.5 (Soil broadcast)</i> <i>3.0-3.5 (Soil in furrow)</i>		<i>1</i>	<i>Before transplanting</i>		
Greece, 2000 Agios Spyridonas, Arta (Baya)	GR	3.0	16	1	99	< 0.01 (<LOD) ≤ 0.01 (<LOD)	DuPont-4583  Mean recovery for oxamyl: 89 ± 18% (n=3) at 0.01 mg/kg 74 ± 11% (n=3) at 0.1 mg/kg
Greece, 2000 Profitis, Thessaloniki (Victor)	GR	5.5	16	1	78	< 0.01 (<LOD) ≤ 0.01 (<LOD)	
Italy, 2001 Contrada Pozzo Bollente, Vittoria, Sicily (Red Wing)	GR	3.2	pre-planting	1	114	< 0.01 (<LOD) ≤ 0.01 (<LOD)	Sampling to analysis: 41-102 days
Italy, 2001 Contrada Sughero Torto, Vittoria, Sicily (Mirò)	GR	5.9	pre-planting	1	101	< 0.01 (<LOD) ≤ 0.01 (<LOD)	
Italy, 2000 Contrada Cervi, Vittoria, Sicily (Camelia)	GR	3.3	pre-planting	1	88	< 0.01 (<LOD) ≤ 0.01 (<LOD)	

Tomato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Italy 2000 Zapponeta (Naxos) Indoor (greenhouse)	GR	5.8	Before transplanting	1	77	< 0.01 (0.008) < 0.01 (<LOD)	
Italy 2000 Zapponeta (Naxos) Indoor (greenhouse)	GR	3.2	Before transplanting	1	77	< 0.01 (<LOD) < 0.01 (<LOD)	
Italy, 2000 Ceriale (Cencara) Indoor (greenhouse)	GR	5.9	Before transplanting	1	105	< 0.01 (<LOD) < 0.01 (<LOD)	

Portion analysed: fruit

### Subgroup of Peppers

#### Pepper

The Meeting received 16 trials (decline trials) on protected peppers which were conducted in Southern Europe (Boissinot, 2007: DuPont-19522, Revision No. 1 and Haigh, 2011: DuPont-29315). In each trial, a SL formulation (100 g/L oxamyl) was applied three-four times *via* drip irrigation or acidified water (pH 5–6) *via* drip irrigation systems.. The first application was made to each treated plot immediately after transplanting. The additional applications (2<sup>nd</sup> to 4<sup>th</sup>) occurred at 10 ± 1 day intervals triggered by the predicted first commercial harvest. First application was made at a target rate of 2.0 kg ai/ha. All other applications were made at a target rate of 1.0 kg ai/ha for a seasonal rate of 4.0 or 5.0 kg ai/ha, respectively.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125. The LOQ was 0.01 mg/kg and the LOD was 0.007 mg/kg. Treated protected peppers samples were stored at -18 ± 5 °C for less than 6 months between sampling and analysis.

Table 57 Residues of oxamyl on protected peppers from supervised trials in Southern Europe

Pepper country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
<i>GAP, Italy &amp; Spain</i>	SL	1.0-2.0 kg ai/ha max 4.0 kg ai/ha/season		3	35		
Spain, 2006 Los Palacios, Andalucia (Andarax) Sweet pepper Indoor (greenhouse)	SL	2.1 1.0 × 2	12, 73, 74	3	-0	0.013	DuPont-19522, Revision No. 1  Mean recovery for oxamyl: 82 ± 12% (n=11) at 0.01 mg/kg 84 ± 16% (n=11) at 0.1 mg/kg
			12, 72, 74		14	< 0.01 (0.009)	
			12, 65, 74		21	0.014	
			12, 63, 71		21	< 0.01 (<LOD)	
	SL	2.1 1.0 × 3	12, 71, 73, 74	4	-0	0.023	
			12, 68, 72, 73		14	< 0.01 (<LOD)	
			12, 61, 68, 73		28	< 0.01 (<LOD)	
					28	< 0.01 (<LOD)	
Spain, 2006 Utrera, Andalucia (Aran) Sweet pepper Indoor (greenhouse)	SL	2.0 1.0 × 2	12, 68, 71	3	-0	< 0.01 (<LOD)	Sampling to analysis: 38-168 days
			12, 66, 68		14	0.045	
			12, 63, 68		21	< 0.01 (0.009)	
			12, 55, 65		21	< 0.01 (<LOD)	
	SL	2.1 1.0 × 3	12, 65, 68, 71	4	-0	0.035	
			12, 63, 66, 66		14	< 0.01 (<LOD)	
			12, 52, 63, 66		28	< 0.01 (<LOD)	
					28	0.022	
Italy, 2006 Poirino, Piemonte	SL	2.0 1.0 × 2	15/16, 70, 70	3	-0	< 0.01 (<LOD)	
					13	< 0.01 (<LOD)	

Pepper country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
(Quadrato di Camagnola) Sweet pepper Indoor (Plastic tunnel)			15/16, 69, 70		21	< 0.01 (<LOD)	
			15/16, 62, 69-70		28	< 0.01 (<LOD)	
			15/16, 62, 64		34	< 0.01 (<LOD)	
	2.0 1.0 × 3	15/16, 64, 70, 70	4	-0	< 0.01 (<LOD)		
		15/16, 62, 69, 70		13	< 0.01 (<LOD)		
		15/16, 61/62, 62, 69-70		21	< 0.01 (<LOD)		
			28	< 0.01 (<LOD)			
Spain, 2006 Partida Quatre Pils, Lleida (Italiano) Chili pepper Indoor (Plastic tunnel)	SL	2.0 1.0 × 2	17-18, 75-76, 78	3	-0	< 0.01 (<LOD)	
			17-18, 72-74, 78		14	< 0.01 (<LOD)	
			17-18, 61-71, 74-75		21	< 0.01 (<LOD)	
			17-18, 51, 63-73		28	< 0.01 (<LOD)	
	2.0 1.0 × 3	17-18, 63-74, 75-76, 78-79	4	-0	< 0.01 (<LOD)		
		17-18, 61, 72-74, 78		14	< 0.01 (<LOD)		
		17-18, 22, 61-71, 74-75		21	< 0.01 (<LOD)		
				28	< 0.01 (<LOD)		
Spain, 2006 Bellcaire d' Urgell, Lleida (Italianos) Chili pepper Indoor (plastic tunnel)	SL	2.1 1.0 × 2	13-14, 76, 79	3	-0	< 0.01 (<LOD)	
			13-14, 72, 77		14	< 0.01 (<LOD)	
			13-14, 51-53, 73		21	< 0.01 (<LOD)	
			13-14, 26-27, 62		28	< 0.01 (<LOD)	
	2.1 1.0 × 3	13-14, 62, 76, 79	4	-0	< 0.01 (<LOD)		
		13-14, 29-51, 72, 77		14	< 0.01 (<LOD)		
		13-14, 19, 51-53, 73		21	< 0.01 (<LOD)		
				28	< 0.01 (<LOD)		
Greece, 2006 Nea Magnisia, Thessaloniki, Central Macedonia (Yanka) Hot pepper Indoor (greenhouse)	SL	2.1 1.0 × 2	12-13, 79, 84-86	3	-0	< 0.01 (<LOD)	
			12-13, 72-76, 82		14	< 0.01 (<LOD)	
			12-13, 67-69, 76-77		21	< 0.01 (<LOD)	
			12-13, 61-62, 71-72		28	< 0.01 (<LOD)	
	2.1 1.0 × 3	12-13, 71-72, 79, 84-86	4	34	< 0.01 (<LOD)		
		12-13, 62-64, 72-76, 82		-0	< 0.01 (<LOD)		
		12-13, 50-51, 67-69, 76-77		14	< 0.01 (<LOD)		
				21	< 0.01 (<LOD)		
			28	< 0.01 (<LOD)			
Greece, 2006 Profitis, Thessaloniki, Central Macedonia (Raiko) Sweet pepper Indoor (greenhouse)	SL	2.1 1.0 × 2	12-13, 82-84, 86	3	-0	< 0.01 (<LOD)	
			12-13, 73-80, 85		14	< 0.01 (<LOD)	
			12-13, 66-73, 81-83		21	< 0.01 (<LOD)	
			12-13, 60-61, 71-78		28	< 0.01 (<LOD)	
	2.1 1.0 × 3	12-13, 71-78, 82-84, 86	4	35	< 0.01 (<LOD)		
		12-13, 63-65, 73-80, 85		-0	< 0.01 (<LOD)		
		12-13, 29, 66-73, 81-83		14	< 0.01 (<LOD)		
				21	< 0.01 (<LOD)		
			28	< 0.01 (<LOD)			
Italy, 2006 Murello, Piemonte (Quadrato di Cuneo) Sweet pepper Indoor (plastic tunnel)	SL	2.0 1.0 × 2	16, 71, 81-82	3	-0	< 0.01 (<LOD)	
			16, 69-70, 71		13	< 0.01 (<LOD)	
			16, 62, 70		21	< 0.01 (<LOD)	
			16, 61, 65		28	< 0.01 (<LOD)	
	2.0 1.0 × 3	16, 65, 71, 81-82	4	34	< 0.01 (<LOD)		
		16, 61-62, 69-70, 71		-0	< 0.01 (<LOD)		
				13	< 0.01 (<LOD)		
				21	< 0.01 (<LOD)		
			28	< 0.01 (<LOD)			

Pepper country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
Spain, 2009 Puebla de Vicar, Andalucia (Vere) Sweet pepper Indoor (plastic greenhouse)	SL	2.1	13	1	105	< 0.01 (<LOD)	DuPont-29315  Mean recovery for oxamyl: 88 ± 5% (n=4) at 0.01 mg/kg 88 ± 4% (n=4) at 0.1 mg/kg  Sampling to analysis: 42-132 days
		2.1 1.0 × 2	13, 53, 61	3	45	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 22, 23/51, 55	4	50	< 0.01 (<LOD)	
			13, 17, 18, 23		65	< 0.01 (<LOD)	
Spain, 2009 Puebla de Vicar, Andalucia (Rigoletto) Sweet pepper Indoor (plastic greenhouse)	SL	2.1	14	1	101	< 0.01 (<LOD)	
		2.1 1.0 × 2	14, 23-51, 53	3	41	< 0.01 (<LOD)	
		2.1 1.0 × 3	14, 19, 23, 52	4	46	< 0.01 (<LOD)	
14, 18, 19, 22	61		< 0.01 (<LOD)				
Spain, 2010 Puente del Rio, Andalucia (Monza) Sweet pepper Indoor (plastic greenhouse)	SL	2.1	14, 16	2	109	< 0.01 (<LOD)	
		2.1 1.0 × 2	14, 16, 17	3	99	< 0.01 (<LOD)	
		2.1 1.0 × 3	14, 22, 52, 53	4	54	< 0.01 (<LOD)	
			14, 19, 21, 51		74	< 0.01 (<LOD)	
Spain, 2010 Puente del Rio, Andalucia (Airone) Sweet pepper Indoor (plastic greenhouse)	SL	2.1	13, 16	2	116	< 0.01 (<LOD)	
		2.1 1.0 × 2	13, 16, 17-18	3	106	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 22, 51, 54	4	66	< 0.01 (<LOD)	
13, 19, 21, 27	81		< 0.01 (<LOD)				
Spain, 2010 Puente del Rio, Andalucia (Estrada) Sweet pepper Indoor (plastic greenhouse)	SL	2.1	13, 14	2	109	< 0.01 (<LOD)	
		2.1 1.0 × 2	13, 14, 16	3	99	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 19, 22, 51	4	59	< 0.01 (<LOD)	
			13, 16-18, 19, 21		74	< 0.01 (<LOD)	
Greece, 2010 Nea Magnisia, Thessaloniki (Ballo) Chili pepper Indoor (greenhouse)	SL	2.0	15	1	55	< 0.01 (<LOD)	
		2.0 1.0	15, 19	2	46	< 0.01 (<LOD)	
		2.1 1.0 × 2	15, 25, 55	3	31	< 0.01 (<LOD)	
		2.1 1.0 × 3	15, 19, 51, 61	4	24	< 0.01 (<LOD)	
Greece, 2010 Boutaiika, Achaia (Raiko) Sweet pepper Indoor (greenhouse)	SL	2.0	14	1	55	< 0.01 (<LOD)	
		2.0 1.0	14, 18	2	44	< 0.01 (<LOD)	
		2.1 1.0 × 2	14, 18, 51	3	30	< 0.01 (<LOD)	
		2.1 1.0 × 3	14, 18, 24, 55	4	26	< 0.01 (<LOD)	
Italy, 2010 Contrada Pozzo Bollente, Vittoria, Sicily (Livor) Sweet pepper Indoor (greenhouse)	SL	2.0	15, 19, 61, 64	4	31	< 0.01 (0.007)	
		1.0 × 3	15, 19, 19, 61		45	< 0.01 (<LOD)	
			15, 19, 19, 19		59	< 0.01 (<LOD)	
			15, 18, 19, 19		74	< 0.01 (<LOD)	

Portion analysed: fruit

### Subgroup of Eggplants

#### Eggplant

The Meeting received 12 trials on protected eggplants which were conducted in Southern Europe (Boissinot *et al.*, 2007 and Haigh *et al.*, 2011, 2012). In each trials, a SL formulation (100 g/L oxamyl) was applied four to five times via drip irrigation or acidified water (pH 5–6) *via* drip irrigation systems.. The first application was made to each treated plot immediately after transplanting. Treatment plots received three or four additional applications. Applications occurred at  $10 \pm 1$  day intervals triggered by the predicted first commercial harvest. Oxamyl first application was applied at a target rate of 2.0 kg ai/ha and 2nd-5th applications were applied at a target rate of 1.0 kg ai/ha.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125 (Boissinot *et al.*, 2007: DuPont-19520, Revision No. 1 and Haigh, 2011: DuPont-29317). The LOQ was 0.01 mg/kg and the LOD was 0.007 mg/kg.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 1901 described in Report No. DuPont-33191 (Haigh, 2012: DuPont-31509). The LOQ was 0.01 mg/kg and the LOD was 0.003 mg/kg.

Treated protected eggplant samples were stored at  $-18 \pm 5$  °C for less than 8 months between sampling and analysis.

Table 58 Residues of oxamyl on protected eggplants from supervised trials in Southern Europe

Eggplant country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
<i>GAP, Italy &amp; Spain</i>	SL	1.0-2.0 kg ai/ha max 5.0 kg ai/ha/season		4	28		
Spain, 2006 Los Palacios, Andalucia (Calanda) Indoor (greenhouse)	SL	2.1 1.0 × 3	12, 65, 71, 72	4	-0	< 0.01 (0.008)	DuPont-19520, Revision No. 1  Mean recovery for oxamyl: 82 ± 7% (n=4) at 0.010 mg/kg 83 ± 6% (n=4) at 0.10 mg/kg
			14		< 0.01 (<LOD)		
		21	< 0.01 (<LOD)				
		28	< 0.01 (<LOD)				
	2.1 1.0 × 4	12, 61, 66, 72	5	35	< 0.01 (<LOD)		
				42	< 0.01 (<LOD)		
		12, 61, 65, 71, 72		-0	< 0.01 (<LOD)		
		14		0.010			
2.1 1.0 × 4	12, 51, 61, 66, 72	5	21	< 0.01 (<LOD)			
			28	< 0.01 (<LOD)			
	35		< 0.01 (<LOD)				
	42		< 0.01 (<LOD)				
Italy, 2006 Triginto di Mediglia, Lombardia (Fantastic) Indoor (greenhouse)	SL	2.1 1.0 × 3	14, 51, 71, 81	4	-0	0.030	Sampling to analysis: 57-213 days
			14		< 0.01 (<LOD)		
		21	< 0.01 (<LOD)				
		28	< 0.01 (<LOD)				
	2.1 1.0 × 4	14, 19, 51, 72	5	35	< 0.01 (<LOD)		
				40	< 0.01 (<LOD)		
		14, 19, 51, 71, 81		-0	0.016		
		14		< 0.01 (<LOD)			
2.1 1.0 × 4	14, 19, 19, 51, 72	5	21	< 0.01 (<LOD)			
			28	< 0.01 (<LOD)			
	35		< 0.01 (<LOD)				
	40		< 0.01 (<LOD)				
Spain, 2006 Bellvis, Lleida, Catalunya (Fujiyama) Indoor (greenhouse)	SL	2.1 1.0 × 3	14, 61, 69-71, 64-71	4	-0	< 0.01 (<LOD)	
			14		< 0.01 (<LOD)		
		21	< 0.01 (<LOD)				
		28	< 0.01 (<LOD)				
	2.1 1.0 × 4	14, 51, 63-64, 70-71	5	35	< 0.01 (<LOD)		
				42	< 0.01 (<LOD)		
		14, 26, 61, 69-71, 64-71		-0	< 0.01 (<LOD)		
		14		< 0.01 (<LOD)			



Eggplant country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref		
	Form	kg ai/ha	BBCH	no.					
			14, 17-18, 51, 63-64, 70-71		21 28 35 42	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)			
Greece, 2006 Profitis, Thessaloniki, Central Macedonia (Neilos) Indoor (greenhouse)	SL	2.0 1.0 × 3	13-14, 54-55, 63-64, 72-74	4	-0 14	< 0.01 (<LOD) < 0.01 (<LOD)			
			13-14, 54, 56, 64-68		22 29 36 43	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)			
		2.0 1.0 × 4	13-14, 51-53, 54-55, 63-64, 72-74	5	-0 14	< 0.01 (<LOD) < 0.01 (<LOD)			
			13-14, 50-51, 54, 63- 64, 72-74		22 29 36 43	< 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)			
	Spain, 2010 Balerma, Almeria, Andalucia (Angela) Indoor (plastic greenhouse)	SL	2.1	13	1	83		< 0.01 (<LOD)	DuPont-29317  Mean recovery for oxamyl: 89 ± 4% (n=4) at 0.01 mg/kg 85 ± 7% (n=4) at 0.10 mg/kg
			2.1 1.0	13, 15	2	73		< 0.01 (<LOD)	
			2.1 1.0 × 2	13, 15, 17	3	63		< 0.01 (<LOD)	
			2.1 1.0 × 3	13, 15, 17, 19	4	53		< 0.01 (<LOD)	
2.1 1.0 × 4			13, 15, 17, 19, 22	5	43	< 0.01 (<LOD)			
Spain, 2010 El Ejido, Almeria, Andalucia (Cristal) Indoor (plastic greenhouse)	SL	2.1	13	1	77	< 0.01 (<LOD)	Sampling to analysis: 89-158 days		
		2.1 1.0	13, 14	2	67	< 0.01 (<LOD)			
		2.1 1.0 × 2	13, 14, 16	3	57	< 0.01 (<LOD)			
		2.1 1.0 × 3	13, 14, 16, 18	4	47	< 0.01 (<LOD)			
		2.1 1.0 × 4	13, 14, 16, 18, 23	5	37	< 0.01 (<LOD)			
Spain, 2010 El Ejido, Almeria, Andalucia (Telma) Indoor (plastic greenhouse)	SL	2.1	13	1	70	< 0.01 (<LOD)			
		2.1 1.0	13, 14	2	60	< 0.01 (<LOD)			
		2.1 1.0 × 2	13, 14, 16	3	50	< 0.01 (<LOD)			
		2.1 1.0 × 3	13, 14, 16, 19	4	40	< 0.01 (0.009)			
		2.1 1.0 × 4	13, 14, 16, 19, 23	5	30	0.014			
Greece, 2010 Nea Magnisia, Thessaloniki (Neelo) Indoor (greenhouse)	SL	2.0	15-19	1	60	< 0.01 (<LOD)			
		2.0 1.0	15-19, 22	2	50	< 0.01 (<LOD) < 0.01 (<LOD)			
		2.0 1.0 × 2	15-19, 22, 51-52	3	39	< 0.01 (<LOD)			
		2.0 1.0 × 3	15-19, 22, 51-52, 61	4	28	< 0.01 (<LOD)			
		2.0	15-19, 22, 51-52, 61,	5	18	< 0.01 (<LOD)			

Eggplant country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
		1.0 × 4	62				
Italy, 2010 Contrada Moglie Dirillo, Acate, Sicily (Birgah) Indoor (greenhouse)	SL	2.1	13	1	73	< 0.01 (<LOD)	
		2.1 1.0	13, 14	2	64	< 0.01 (<LOD)	
		2.1 1.0 × 2	13, 14, 17	3	54	< 0.01 (<LOD)	
		2.1 1.0 × 3	13, 14, 17, 18	4	44	< 0.01 (<LOD)	
		2.1 1.0 × 4	13, 14, 17, 18, 19	5	33	< 0.01 (<LOD)	
Italy, 2011 Contrada Sughero Torto, Vittoria, Sicily (Black Bell) Indoor (greenhouse)	SL	2.1	12, 16, 19, 63, 64	5	15	0.028	DuPont-31509  Mean recovery for oxamyl: 81% (n=2) at 0.010 mg/kg 86% (n=2) at 0.10 mg/kg
		1.1 × 4	12, 15, 18, 61, 63		23	< 0.01 (0.007)	
			12, 14, 15, 19, 62		30	< 0.01 (0.003)	
				12, 13, 14, 16	4	47	
Spain, 2011 La Mojonera, Andalucia (Thelma) Indoor (plastic greenhouse)	SL	2.1	12, 51, 61, 63, 65	5	14	< 0.01 (0.008)	81% (n=2) at 0.010 mg/kg 86% (n=2) at 0.10 mg/kg
		1.0 × 4	12, 22, 52, 62, 63		21	< 0.01 (<LOD)	
			12, 21, 24, 52, 62		28	< 0.01 (<LOD)	
			12, 14, 16, 21, 51		45	< 0.01 (<LOD)	
Spain, 2011 La Mojonera, Andalucia (Ecavi) Indoor (plastic greenhouse)	SL	2.1	12, 51, 61, 63, 65	5	14	0.020	Sampling to analysis: 181- 189 days
		1.0 × 4	12, 22, 52, 62, 63		21	< 0.01 (<LOD)	
			12, 21, 24, 52, 62		28	< 0.01 (<LOD)	
			12, 14, 16, 21, 51		45	< 0.01 (<LOD)	

Portion analysed: fruits

### Root and tuber vegetables

#### Subgroup of Root vegetables

#### Carrot

The Meeting received nine trials (two decline and seven at harvest trials) on carrots which were conducted in Europe (Foster *et al.*, 2003 and 2004). In each trial, oxamyl 100 g/kg GR formulation was applied once by granular furrow application at a target rate of 0.090 g ai/m row or 0.074 g ai/m row. Duplicate specimens of carrot roots, tops removed were taken for analysis from the treated plot and the control plot at each sampling interval (Foster *et al.*, 2003: DuPont-13037). In each trial, oxamyl 100 g/kg GR formulation was applied once using a granular applicator, mounted on a drill, to the treated plot at planting. Application was at rates (linear, in furrow) of 0.083-0.43 g ai/m row (Foster *et al.*, 2004: DuPont-14668).

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125. The LOQ was 0.01 mg/kg and the LOD was 0.007 mg/kg. Treated carrot roots were stored at *ca.* -20 °C for no longer than 4 months between sampling and analysis.

Table 59 Residues of oxamyl on carrots from supervised trials in Europe

Carrot country, year (variety)	Application				DALA	Residues, mg/kg	Ref
	Form	g ai/m furrow	BBCH	no.			
<i>GAP, UK</i>	<i>GR</i>	<i>0.090</i>	<i>at drilling</i>	<i>1</i>	<i>12 weeks</i>		
UK, 2003 Chatteris, Cambridgeshire (Nairobi) Outdoor	GR	0.091	00 at planting	1	8 weeks 10 weeks 12 weeks 14 weeks 16 weeks	< 0.01 (<LOD) < 0.01 (<LOD) <u>&lt; 0.01</u> (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	DuPont-13037  Mean recovery for oxamyl: 80 ± 11% (n=4) at 0.01 mg/kg 81 ± 5% (n=4) at 0.10 mg/kg
UK, 2003 Forfar, Angus (Sugar Snax 54) Outdoor	GR	0.074	00 at planting	1	8 weeks 10 weeks 12 weeks 14 weeks 16 weeks	< 0.01 (<LOD) < 0.01 (<LOD) <u>&lt; 0.01</u> (<LOD) < 0.01 (<LOD) < 0.01 (<LOD)	
Netherlands, 2003 8308 RS Nagele (Nerac) Outdoor	GR	0.088	00 at planting	1	12 weeks 16 weeks	<u>&lt; 0.01</u> (<LOD) < 0.01 (<LOD)	Sampling to analysis: 67-97 days
Netherlands, 2003 8307 PX Ens (Nerac) Outdoor	GR	0.088	during sowing	1	12 weeks 16 weeks	<u>&lt; 0.01</u> (<LOD) < 0.01 (<LOD)	
France, 2004 Bagé-le-Châtel, Rhône- Alpes (Bolero) Outdoor	GR	0.089 (5.4 kg ai/ha)	00	1	75 days 90 days	< 0.01 (<LOD) <u>&lt; 0.01</u> (<LOD)	DuPont-14668  Mean recovery for oxamyl: 87% (n=2) at 0.01 mg/kg 89% (n=2) at 0.10 mg/kg
Italy, 2004 Via Cristina, Emilia Romagna (Dondoque) Outdoor	GR	0.083 (5.5 kg ai/ha)	00	1	75 days 91 days	< 0.01 (<LOD) <u>&lt; 0.01</u> (<LOD)	
Italy, 2004 Via Sacché, Emilia Romagna (Bolero) Outdoor	GR	0.083 (5.5 kg ai/ha)	00	1	76 days 90 days	< 0.01 (<LOD) <u>&lt; 0.01</u> (<LOD)	Sampling to analysis: 36-126 days
Greece, 2004 Apolonia, Central Macedonia (Sundor) Outdoor	GR	0.13 (3.6 kg ai/ha)	00	1	75 days 90 days	< 0.01 (<LOD) < 0.01 (<LOD)	
Greece, 2004 Chalkidona, Central Macedonia (Bolero) Outdoor	GR	0.43 (5.7 kg ai/ha)	00	1	75 days 90 days	< 0.01 (<LOD) < 0.01 (<LOD)	

Portion analysed: root

### *Sugar beet*

The Meeting received 19 trials on sugarbeet which were conducted in Europe. In each trial, One application of oxamyl (as 100 g/kg or 50 g/kg GR formulation) was made in furrow, targeting approximately 2.5 kg ai/ha, at planting.

Oxamyl residues were determined by HPLC with column switching and UV detection (HPLC-CS/UV) according to the method validated in DuPont-3702. The LOQ was 0.02 mg/kg and the LOD was 0.01 mg/kg (Françon *et al.*, 2000: DuPont-2408).

Oxamyl residues were determined by HPLC with post-column derivatization and fluorescence detection (HPLC-PCD/Fluo) according to the method validated in DuPont-4722. The LOQ was 0.01 mg/kg and the LOD was 0.005 mg/kg (Françon *et al.*, 2001: DuPont-3940 and Zenide

et al., 2002: DuPont-4582, Revision No. 1). Specimens from this study were stored at about -20 °C or below for periods not exceeding 6 months.

Table 60 Residues of oxamyl on sugar beets from supervised trials in Europe

Sugar beet country, year (variety)	Application				DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	BBCH	no		Root	Leaves + top	
<i>GAP, Netherlands</i>	GR	0.75-2.5	at sowing	1				
Greece, 1999 Athira, Pella (Turbo) Outdoor	GR	2.6	01	1	146	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	DuPont-2408
Greece, 1999 Kilada, Kozani (Turbo) Outdoor	GR	2.6	01	1	146	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	Mean recovery for oxamyl: Roots 83 ± 18% (n=3) at 0.02 mg/kg 72 ± 11% (n=3) at 0.20 mg/kg Leaves+tops 83 ± 21% (n=3) at 0.02 mg/kg 73 ± 4% (n=3) at 0.20 mg/kg
Spain, 1999-2000 Los Palacios, Sevilla (Marisma) Outdoor	GR	2.6	00 At planting	1	198	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	
Spain, 1999-2000 El Palmar, Sevilla (Jerez) Outdoor	GR	2.6	00 At planting	1	192	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	
Netherlands, 1999 Elst, Gelderland (Mondio 534) Outdoor	GR	2.5	Before planting	1	176	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	Sampling to analysis: 25-253 days
Netherlands, 1999 Angeren, Gelderland (Aristo) Outdoor	GR	2.7	Before planting	1	190	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	
UK, 1999 Melbourne, Yorkshire (Roberta) Outdoor	GR	2.6	00 At drilling	1	199	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	
UK, 1999 Smisby, Leicestershire (Saxone) Outdoor	GR	2.6	00 At drilling	1	190	$\leq 0.02$ (<LOD)	< 0.02 (<LOD)	
Greece, 2000 Kilada, Kozani (Turbo) Outdoor	GR	2.6	00	1	140	$\leq 0.01$ (<LOD)	< 0.01 (<LOD)	DuPont-3940
Greece, 2000 Athira, Pella (Rizor) Outdoor	GR	2.6	00	1	169	$\leq 0.01$ (<LOD)	< 0.01 (<LOD)	Mean recovery for oxamyl: Roots 75 ± 5% (n=3) at 0.01 mg/kg 83 ± 1% (n=3) at 0.10 mg/kg Leaves+tops 78 ± 8% (n=3) at 0.01 mg/kg 76 ± 5% (n=3) at 0.10 mg/kg
Spain, 2000 El Perdigon, Zamora (Sherif) Outdoor	GR	2.6	00 pre-planting	1	145	$\leq 0.01$ (<LOD)	< 0.01 (<LOD)	
Spain, 2000 Cubo del Vino, Zamora (KSA monogermen) Outdoor	GR	2.6	00	1	145	$\leq 0.01$ (<LOD)	< 0.01 (<LOD)	Sampling to analysis: 114-177 days
Spain, 2000 Corrales del Vino, Zamora (Monpeso) Outdoor	GR	2.6	00 Pre-planting	1	145	$\leq 0.01$ (<LOD)	< 0.01 (<LOD)	

Sugar beet country, year (variety)	Application				DALA Days	Residues, mg/kg		Ref
	For m	kg ai/ha	BBCH	no		Root	Leaves + top	
Netherlands, 2000 Elst, Gelderland (Rebecca) Outdoor	GR	2.6	00 Before sowing	1	165	< 0.01 (<LOD)	< 0.01 (<LOD)	
Netherlands, 2000 Angeren, Gelderland (Leonora) Outdoor	GR	2.6	00 Before sowing	1	140	< 0.01 (<LOD)	< 0.01 (<LOD)	
UK, 2000 Barton, Nottinghamshire (Roberta) Outdoor	GR	2.6	00 Pre- planting	1	187	< 0.01 (<LOD)	< 0.01 (<LOD)	
UK, 2000 Whixley, North Yorkshire (Roberta) Outdoor	GR	2.6	00 Pre- planting	1	180	< 0.01 (<LOD)	< 0.01 (<LOD)	
Italy, 2001 Manfredonia, Foggia Puglia (Autunnia) Outdoor	GR	2.7	00	1	222	< 0.01 (<LOD)	< 0.01 (<LOD)	DuPont-4582, Revision No. 1  Mean recovery for oxamyl: Roots 95 ± 6% (n=6) at 0.01-0.1 mg/kg Leaves+tops 97 ± 9% (n=6) at 0.01-0.1 mg/kg
Italy, 2001 Borgotressanti, Foggia Puglia (Autunnia) Outdoor	GR	2.7	00	1	247	< 0.01 (<LOD)	< 0.01 (<LOD)	Sampling to analysis: 124- 132 days

### *Subgroup of Tuberous and corm vegetables*

#### *Potato*

The Meeting received 12 trials on potatoes which were conducted in Europe. One in-furrow application of oxamyl (as 5 g/kg GR formulation) was conducted at planting, at a rate of approximately 3 kg ai/ha at each trial sites in Southern Europe.

Oxamyl residues were determined by HPLC with post-column derivatization and fluorescence detection (HPLC-PCD/Fluo) according to the method validated in DuPont-4722. The LOQ was 0.01 mg/kg and the LOD was 0.005 mg/kg. Specimens from this study were stored at about -20 °C or below for periods not exceeding 4 months (Zenide, 2002: DuPont-5989).

Oxamyl 100 g/kg GR formulation was applied once *via* granular soil application at the time of planting. Application was made to main variety potatoes at a target rate of 5.5 kg ai/ha and 4.0 kg ai/ha, respectively.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125. The LOQ was 0.005 mg/kg and the LOD was 0.0033 mg/kg. Treated potatoes samples were stored at -18 ± 5 °C for less than 6 months between sampling and analysis (Boissinot, 2007: DuPont-19526).

Table 61 Residues of oxamyl on potatoes from supervised trials in Europe

Potato country, year (variety)	Application				DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	BBCH	no.			
<i>GAP, UK</i>	<i>GR</i>	<i>5.5</i>	<i>At planting</i>	<i>1</i>	<i>80</i>		
Greece, 2001 Polimilos, Kozani (Spunta) Outdoor	GR	3.0	00	1	112	< 0.01 (<LOD)	DuPont-5989  Mean recovery for oxamyl: 78 ± 5% (n=4) at 0.01-0.1 mg/kg
Italy, 2001 Bagnarola di Budrio, Bologna (Primura) Outdoor	GR	3.2	00 At seeding	1	117	< 0.01 (<LOD)	Sampling to analysis: 41-104 days
UK, 2006 St Osyth, Clacton on Sea, Essex (Maris Piper) Outdoor	GR	5.2	00	1	148	< 0.005 (<LOD)	DuPont-19526  Mean recovery for oxamyl: 87 ± 5% (n=4) at 0.005 mg/kg
Spain, 2006 Olivares, Andalucia (Carlita) Outdoor	GR	4.1	00	1	97	< 0.005 (<LOD)	92 ± 9% (n=4) at 0.1 mg/kg
France, 2006 Allouagne, Nord Pas-de-Calais (Amila) Outdoor	GR	5.6	00	1	153	< 0.005 (<LOD)	Sampling to analysis: 39-171 days
Italy, 2006 Corana, Lombardia (Annabella) Outdoor	GR	5.6	00	1	102	< 0.005 (<LOD)	
France, 2006 La Chapelle Villars, Rhone-Alpes (Europa) Outdoor	GR	5.6	00	1	120	< 0.005 (<LOD)	
France, 2006 Le Mas Rillier, Rhone-Alpes (Mora Lisa) Outdoor	GR	5.6	00	1	132	< 0.005 (<LOD)	
Poland, 2006 Rozbity Kamieñn Central Poland Mazovian Region (Irga) Outdoor	GR	5.6	00-03	1	135	< 0.005 (<LOD)	
Spain, 2006 Partida rec nou, Cami de l'Albi s/n, Alpicat, Lleida (Kenebec) Outdoor	GR	5.6	00	1	92	< 0.005 (<LOD)	
Greece, 2006 Nea Magnisia, Thessaloniki, Central Macedonia (Agria) Outdoor	GR	4.0	00	1	100	< 0.005 (<LOD)	
Germany, 2006 D-04668 Motterwitz, Saxony (Prinzess) Outdoor	GR	5.6	01	1	124	< 0.005 (<LOD)	

Portion analysed: tuber

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### *In Processing*

The Meeting received information on high temperature hydrolysis of oxamyl and the fate of oxamyl residues during the processing of potatoes.

Potatoes that the Meeting received information on supervised field trials may be processed prior to consumption. Processing factors have been calculated for oxamyl residues in potatoes.

#### *High temperature hydrolysis*

The hydrolysis of [<sup>14</sup>C]-oxamyl was studied in sterile buffered solutions of pH 4, 5 and 6 (Lee, 2001: DuPont-4025). The buffered solutions used in these studies were 0.01 M acetate (pH 4 and pH 5), and 0.01 M phosphate (pH 6). These studies were conducted in test systems consisting of the sterile treated buffer solutions contained in individual, capped sterile vials for each test sample. The test systems were treated under conditions that test the effects of pasteurization (pH 4, 90 °C for 20 min), baking/boiling (pH 5, 100 °C for 60 min), and sterilization (pH 6, 120 °C for 20 min). Samples were taken immediately after the addition of test substance to the buffered solutions and, after heating, following a period of time to return the samples to room temperature (approximately 20 °C). Samples were analysed for total radioactivity, and for the parent and hydrolysis product(s).

Duplicate 10 µL aliquots were removed from each sampling interval for radioactivity determination *via* LSC. An aliquot from each sample was also immediately analysed for radioactivity distribution using the LC-ARC system.

Under the simulated food processing conditions, [<sup>14</sup>C]-oxamyl degraded at different rates, depending on processing conditions. At pH 4 heating at 90 °C for 20 min, no degradation was observed. At pH 5 heating at 100 °C for 60 min, 57.7% of test material degraded to IN-A2213, while all the test material degrades to IN-A2213 after incubation at pH 6, at 120 °C for 20 min.

Table 62 Identification of radioactivity under the conditions for processing simulation

Conditions	Recovery of Applied Radioactivity [1 mg/L]		
	Oxamyl	IN-A2213	Total
pH 4, 90 °C, 20min	100, 100	0, 0	100, 100
pH 5, 100 °C, 60 min	56.5, 59.1	42.5, 40.3	99.0, 99.4
pH 6, 120 °C, 20 min	0, 0	100, 100	100, 100

#### *Potatoes*

The study was conducted to determine the magnitude of residues of oxamyl in/on raw potato tubers and processed fractions of potato tubers in Europe following exaggerated treatment with Oxamyl 100 g/kg GR formulation during the 2009 growing season (Foster, 2009: DuPont-27667). Three tests consisting of three different early potato varieties planted in control and treated boxes containing acidic compost (pH 4.0–4.5) were established in Northern Europe. In all tests, Oxamyl 100 g/kg GR formulation was applied six times commencing with the first application on the day of tuber planting. The second application was repeated after 28 days followed at 7-day intervals with applications 3rd–6th inclusive. The 1st application was applied at a target rate of 16.5 kg ai/ha. The 2<sup>nd</sup>–6<sup>th</sup> application inclusive were made at a target rate of 5.5 kg ai/ha for a seasonal target application of 44 kg ai/ha, 8 × the critical EU GAP.

For all tests, bulk specimens of potato tubers were collected at maturity, 46 days after the last application. Initially, per test, one raw potato tuber control specimen together with three raw potato tuber treated specimens were submitted for analysis. Following the determination of detectable oxamyl residues in the raw agricultural commodity (uncooked potato tubers), a cooking phase was conducted to generate cooked potato tuber specimens (separate baked, boiled and microwaved specimens). The cooking phase occurred three days after harvesting the bulk potato tuber samples. During these three days the bulk uncooked samples were stored in a cool, dark location which simulated commercial practice. All the analysed specimens (raw and cooked) were unpeeled and had been lightly washed to remove adhering compost at the time of harvest.

Cooked specimens were generated by boiling whole unpeeled tubers for 20 min, baking whole unpeeled tubers for 45 min or microwaving whole unpeeled tubers for 15 min. Processed

(cooked) potato samples were deep-frozen (after cooling) within approximately 5 hours of being processed.

Specimens were analysed for residues of oxamyl using Charles River Laboratories Analytical Method No. 0259 described in Report No. DuPont-11125. The determined LOQ in potato tubers was 0.005 mg/kg. The LOD was 0.0033 mg/kg. The mean overall percent recovery for oxamyl from 15 control unpeeled potato tubers freshly fortified at 0.005 mg/kg and 0.10 mg/kg (uncooked, microwaved, boiled and baked) was  $94 \pm 10\%$ . Processed potato tuber samples were stored at  $ca -18 \pm 5$  °C for less than 1 month between processing and analysis.

Table 63 Residues of oxamyl in processed commodities of potatoes

country, year (variety)	Application			DALA Days	Commodity	Residues, mg/kg	Processing Factor	
	kg ai/ha	BBCH	no .					
UK, 2009 Tranent East Lothian (Rocket)	16.3	03-07	1	46	Uncooked tuber	0.077, 0.10, 0.11 mean 0.096	0.15	
	5.5	19			Baked tuber	0.017, 0.011 mean 0.014		
	5.5	33-36			Boiled tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.04
	5.5	36-37			Microwaved tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.04
	5.5	51-55						
	total							
	43.6							
UK, 2009 Tranent East Lothian (Arran Pilot)	16.3	03-07	1	46	Uncooked tuber	0.057, 0.10, 0.11 mean 0.089	-	
	5.5	19			Baked tuber	0.0094, 0.0056 mean 0.0075		0.08
	5.5	33-36			Boiled tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.04
	5.5	36-37			Microwaved tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.04
	5.5	37-38						
	total							
	43.6							
UK, 2009 Tranent East Lothian (Wilja)	16.3	03-07	1	46	Uncooked tuber	0.053, 0.070, 0.086 mean 0.070	< 0.05	
	5.5	09			Baked tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.05
	5.5	21-23			Boiled tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.05
	5.5	31-32			Microwaved tuber	< 0.005 (<LOD), < 0.005(<LOD) mean < 0.005 (<LOD)		< 0.05
	5.5	34-35						
	total							
	43.6							

Processing Factor = Oxamyl residues in processed commodity/ Oxamyl residues in unprocessed potato tubers

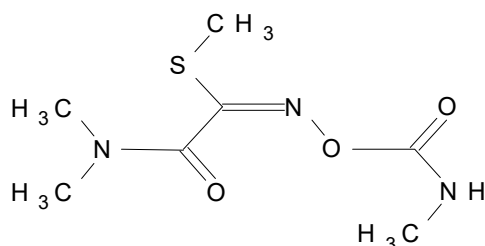
For calculations, 0.0033 mg/kg was used for fractions with <LOD residues.

## APPRAISAL

Oxamyl is a carbamate insecticide that acts by inhibiting acetylcholinesterase activity. It was first evaluated by JMPR in 1980 (T, R) and a periodic re-evaluation conducted in 2002 (T, R). It was included in the Priority List under the Periodic Re-Evaluation Program at the 48<sup>th</sup> Session of the CCPR (2016) for evaluation by the 2017 JMPR.

The Meeting received information on physical and chemical properties, animal and plant metabolism, rotational crop residues, environmental fate, analytical methods, GAP information, storage stability, processing and residue trial data on Brussel sprout, cucumber, courgette, melon, tomato, pepper, eggplant, carrot, sugar beet and potato.





*N,N*-dimethyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide

In this appraisal, the following abbreviated names were used for metabolites.

IN-A2213  Synonym: Oxamyl-oxime Methyl 2-(dimethylamino)- <i>N</i> -hydroxy-2-oxoethanimidothioate	IN-D2708  (Dimethylamino)oxoacetic acid	IN-N0079  Synonym: DMCF Dimethylcarbonocyanidic amide
IN-QKT34  Synonym: IN-A2213 glucoside [(Hexopyranosyloxy)imino]- <i>N,N</i> -dimethyl-2-(methylthio)acetamide	IN-F3905  Methyl ( <i>E</i> ) 2-(dimethylamino)- <i>N</i> -hydroxy-2-oxoethanimidothioate	IN-L2953  Methyl <i>N</i> -hydroxy-2-(methylamino)-2-oxoethanimidothioate
IN-KP532  (Methylamino)oxoacetic acid	IN-KV998  <i>N</i> -Methylethanediamide	IN-T2921  <i>N,N</i> -Dimethylethanediamide

### Plant metabolism

The Meeting received plant metabolism studies on potato and tomato conducted with [1-<sup>14</sup>C]-oxamyl.

In a potato metabolism study, [<sup>14</sup>C]-oxamyl was applied to soil at a rate of 8 kg ai/ha immediately after seed potatoes were planted in plastic pots. TRR in the peels and peeled potatoes from the <sup>14</sup>C-oxamyl treated plants were 1.1 and 0.86 mg eq/kg, respectively. The majority (~91%) of the radioactivity was extracted from the peels (1.0 mg eq/kg) and peeled potatoes (0.79 mg eq/kg).

The major extracted residue in the peels (68% TRR, 0.76 mg eq/kg) and the peeled potato (71% TRR, 0.61 mg eq/kg), extracted with methanol/water, was IN-D2708. Other extracted components ranged in concentration from 0.02–0.07 mg eq/kg. The concentration of each of these polar and unknown metabolites were < 0.04 mg eq/kg in the whole potato. No oxamyl or IN-A2213 (oxamyl-oxime) was detected.

The majority of the residues in foliage were extracted with methanol/water (78% TRR, 1.2 mg eq/kg). The major foliage metabolite (46% TRR, 0.69 mg eq/kg) was a water-soluble component that was resistant to enzyme ( $\beta$ -glucosidase) and acid (0.1 M HCl, 90 °C, 6 hours) hydrolysis. This metabolite was characterized as IN-QKT34 (IN-A2213 glucoside).

In a tomato metabolism study, [ $^{14}$ C]-oxamyl was applied at a rate of 2.0 kg ai/ha immediately after transplant of the tomato plants. The 3 subsequent applications for both the foliar and soil experiments were carried out 14 days apart at a rate of 1.0 kg ai/ha for each application. Following one 2 kg ai/ha and three 1 kg ai/ha foliar applications of [ $^{14}$ C]-oxamyl, TRRs ranged between 0.72–1.4 mg eq/kg in fruit and 4.8–40 mg eq/kg in foliage. Following soil applications at the same rates and application timing, fruit TRRs ranged between 0.33–0.81 mg eq/kg and foliage 5.5–11 mg eq/kg.

For the foliar applications, TRR in fruit collected 7 and 21 days after the fourth foliar treatment (DALA) were 0.72 and 0.99 mg eq/kg, respectively. A surface wash with water of fruit at 7 and 21 DALA released 31% TRR (0.22 mg eq/kg) and 1.3% TRR (0.013 mg eq/kg), respectively. The surface wash and methanol/water extract from homogenised samples were combined, resulting in a total extractability of 94–96%. Oxamyl was the principal extracted fruit residue at 7 DALA accounting for 31% TRR (0.22 mg eq/kg). Oxamyl concentrations in the 21 DALA fruit sample decreased to 2.9% TRR (0.027 mg eq/kg). Other components identified as major metabolites in the 7 and 21 DALA fruit included IN-N0079 (9.0–13% TRR, 0.088–0.090 mg eq/kg) and IN-D2708 (21% TRR, 0.21 mg eq/kg).

TRR in foliage collected 7 and 21 DALA of the foliar treatment were 9.9 and 40 mg eq/kg, respectively. Surface wash with water of foliage at 7 and 21 DALA were 37% TRR (3.6 mg eq/kg) and 22% TRR (8.7 mg eq/kg), respectively. The TRRs of surface wash and the methanol/water extract of homogenised samples were combined, resulting in a total extractability of 97–98%. Oxamyl was the predominant component (73–78% TRR, 7.2–31 mg eq/kg) detected in the foliage at both sampling points. IN-QKT34 (11–13%TRR, 1.2–4.2 mg eq/kg) was the principal metabolite in tomato foliage.

For soil applications, TRR in fruit collected 7, 14 and 21 DALA of the soil treatment ranged from 0.33–0.81 mg eq/kg. Low levels of oxamyl were detected (5.9% TRR, 0.047 mg eq/kg) in the 7 DALA fruit but not in subsequent fruit samples. Fruit major metabolites included IN-A2213 (8.4–11% TRR, 0.031–0.089 mg eq/kg), IN-QKT34 (4.8–11% TRR, 0.016–0.071 mg eq/kg), IN-N0079 (2.3–22% TRR, 0.015–0.073 mg eq/kg) and IN-D2708 (21% TRR, 0.14 mg eq/kg).

TRR in foliage collected 7, 14 and 21 DALA of the soil treatment were 5.5, 7.1 and 11 mg eq/kg, respectively. Oxamyl was detected at 19, 11 and 6.3% TRR (1.1, 0.75 and 0.73 mg eq/kg) in the 7, 14 and 21 DALA foliage, respectively. IN-QKT34 (35–63% TRR, 1.9–7.1 mg eq/kg) was the principal foliage metabolite.

In summary, oxamyl was primarily metabolised in potato and tomato by hydrolysis of the methylcarbamoyl group and subsequent conjugation to yield IN-A2213 and IN-QKT34 (IN-A2213 glucoside). IN-A2213 was also metabolised by cleavage to give IN-N0079 which is further metabolized by oxidation to IN-D2708.

### ***Animal metabolism***

The Meeting received animal metabolism studies with oxamyl on lactating goat and laying hens. The metabolism and distribution of oxamyl in animals was investigated using the [ $^{14}$ C]-oxamyl.

Lactating goats were orally dosed with 59 mg [ $^{14}$ C]-oxamyl daily for 5 consecutive days at a dose level of 31 ppm in the diet. The majority (52%) of the administered dose was eliminated in urine and faeces.

Following the administration of [ $^{14}$ C]-oxamyl, TRRs were 8.4 mg eq/kg in liver, 4.6 mg eq/kg in kidney, 1.3 mg eq/kg in muscle and 0.64 mg eq/kg in fat. Methanol/water extracted 30–67% of TRR in tissues. Most of the unextracted residue (31–58% TRR) in tissues was released by protease digestion. TRRs in milk reached a maximum of 4.6 mg eq/L on day 5 (a plateau had not been reached). Approximately 2% of the TRR in milk was found in the chloroform extract, 67–73% TRR

was in methanol/water extract and about 25–31% TRR in milk remained in the pellet. Most of unextracted residue (23–29% TRR) in milk was released by protease digestion.

Thiocyanate was the major metabolite found in milk (23–36% TRR, 0.35–1.5 mg eq/L in the methanol/water extract with a further 9.4–12% TRR (0.17–0.51 mg eq/L) in the protease extract) and it was also detected in all tissue extracts (2.8–31% TRR, 0.14–0.43 mg eq/kg). No measured parent oxamyl was found in any of analysed tissues or milk fractions.

Laying hens were orally dosed with 3.6 mg [ $1\text{-}^{14}\text{C}$ ]-oxamyl daily for 3 consecutive days at a dose level of 43 ppm in the diet. The hens excreted 71% of the total administered dose.

TRRs were 2.0 mg eq/kg in liver, 1.7 mg eq/kg in kidney, 0.44–0.68 mg eq/kg in muscle, 0.71 mg eq/kg in skin and 0.064 mg eq/kg in fat. Eggs collected following the last dose, 48–72 hour (Day 3) samples, contained 1.1 mg eq/kg and 1.2 mg eq/kg, respectively, for the yolk and white. 47–93% of TRR in tissues and egg was extracted by hexane, methyl chloride, ethyl acetate and methanol/water. 22% TRR in liver was extracted into methanol/water and 24% TRR in liver was extracted with the ethyl acetate. Most of unextracted residue (33% TRR) in liver was released by protease digestion.

No oxamyl was present in any tissue sample and there were no carbamate-containing metabolites detected. The major metabolite in all tissues and eggs was identified as thiocyanate. It represented 14% TRR in liver (0.27 mg eq/kg), 14% TRR in muscle (0.088 mg eq/kg), 26% TRR (0.30 mg eq/kg) in the Day 3 egg white and 33% TRR (0.35 mg eq/kg) in the Day 3 egg yolk.

In summary, oxamyl was degraded extensively into small molecular weight compounds such as thiocyanate and  $\text{CO}_2$  in goats and hens. The residues associated with protein in tissues was 31–58% TRR. In rats, the radioactivity in tissues was not identified, but was found to be neither oxamyl nor its metabolites.

### ***Environmental fate***

#### *In soil*

The Meeting considered information on degradation in aerobic soil, soil photolysis and field dissipation studies because oxamyl is intended for use as soil treatment.

In degradation in aerobic soil studies, [ $1\text{-}^{14}\text{C}$ ]-oxamyl was applied at a rate of 2 or 5 mg/kg (dry soil basis) to viable agricultural soils and incubated at 20 °C.

There were three significant degradation products - IN-A2213, IN-D2708 and  $^{14}\text{CO}_2$ . No other metabolite was observed at >3.8% of the applied radioactivity. The  $^{14}\text{CO}_2$ , found in the caustic trap, was the final and most significant degradate. For the eight soils tested the  $\text{DT}_{50}$  for oxamyl was 3–12 days, except in one soil that had a  $\text{DT}_{50}$  of 112 days. Maximum levels of IN-A2213 and IN-D2708 were 5.0–51% and 25–78% of applied radioactivity (AR), respectively.

In field soil dissipation studies, oxamyl was applied once at 1.5–20 kg ai/ha to soils in Europe and the USA. The  $\text{DT}_{50}$  values of oxamyl, IN-A2213 and IN-D2708 for European soils were 3.3–11, 1.7–5.7 and 0.52–6.7 days, respectively. The  $\text{DT}_{50}$  values of oxamyl for US soils were 9–29 days.

In conclusion, oxamyl is not persistent in soil ( $\text{DT}_{50}$ : 3–29 days).

In a soil photolysis study, thin layers of soil (2 mm) were treated with oxamyl to obtain a concentration of 5.3 mg ai/kg dry weight soil. The soil samples were maintained at approximately  $21 \pm 2$  °C with continuous irradiation for up to 15 days under simulated natural sunlight produced by a Xenon arc lamp.

The degradation products were IN-D2708, IN-N0079 and IN-A2213, which reached average maximum concentrations of 45% AR (Day 15), 8.7% AR (Day 5), and 3.6% AR (Day 3), respectively. The  $\text{DT}_{50}$  and  $\text{DT}_{90}$  for oxamyl were 4.7 and 15.7 days in irradiated samples and 24.2 and 80.5 days in non-irradiated samples, respectively.

Photolysis on the soil surface is a degradation route for oxamyl.

*In water*

The Meeting considered information on hydrolysis.

In the hydrolytic degradation study, oxamyl was hydrolytically stable at pH 4, but unstable at pH 7 and 9 after incubation at 20–30 °C. The DT<sub>50</sub> of oxamyl was 4.2–21 days at pH 7 and < 1 day at pH 9.

**Rotational crop studies**

The Meeting received confined rotational crop studies with [<sup>14</sup>C]-labelled oxamyl and field rotational crop studies with unlabelled compound.

In confined rotational crop studies, rotational crops (barley, cabbage, beet, sorghum and lettuce) were sown at 30, 120 and 363 days after soil treatment (PBI). [<sup>14</sup>C]-oxamyl was applied as a single application to soil at a rate of 8–20 kg ai/ha.

A sandy loam soil in containers was treated on the surface with [<sup>14</sup>C]-oxamyl at 8.96 kg ai/ha, and aged in the greenhouse. Crops (red beet, cabbage and sorghum) seeds were sown in the containers at PBIs of 30 and 120 days. Crops planted into soil which had been aged 30 days exhibited TRRs of 0.6–4.2 mg eq/kg. The residue that could be ascribed to oxamyl and/or IN-A2213 was 0.01–0.12 mg eq/kg in crops planted at 30-day PBI.

[<sup>14</sup>C]-oxamyl was applied to the surface of a sandy loam soil in containers at a nominal rate of 20.2 kg ai/ha. The crops (lettuce, beet and barley) were planted after an ageing period of 30, 120 and 363 days. TRRs were 3.1–38 mg eq/kg for crops sown at the 30-day PBI, 0.27–6.8 mg eq/kg for crops sown at the 120-day PBI and 0.03–0.29 mg eq/kg for crops sown at the 363-day PBI. Oxamyl and IN-A2213 were significant residues (oxamyl: 31–58% TRR, 0.53–12 mg eq/kg and IN-A2213: 11–18% TRR, 0.30–2.2 mg eq/kg) in barley forage at both the 30-day and the 120-day PBIs. These two components were not detected in barley grain. Oxamyl and IN-A2213 were detected in beet root and foliage, and lettuce, at the 30-day PBI, but in general never exceeded 11% TRR. At 120-day PBI, IN-A2213 was only detected (4.3% TRR, 0.04 mg eq/kg) in beet roots and no oxamyl was detected in any of the beet RAC. Neither oxamyl nor IN-A2213 was detected in lettuce from the 120-day PBI. However, polar unknowns were significant components of the TRR in barley forage (max 51% TRR, 0.87 mg eq/kg), barley straw (max 32% TRR, 12 mg eq/kg), barley grain (58% TRR, 0.76 mg eq/kg) and lettuce (82% TRR, 0.22 mg eq/kg).

[<sup>14</sup>C]-oxamyl was applied as a single application at a rate of 8 kg ai/ha and barley was sown at a 30-day PBI. TRRs were 0.32 mg eq/kg in barley grain, 6.7 mg eq/kg in barley forage, 1.2 mg eq/kg in barley hay and 1.6 mg eq/kg in barley straw. The major extracted residue in the barley grain (51% TRR, 0.16 mg eq/kg) was IN-D2708. No oxamyl or other metabolites were identified in the grain. The major metabolite present in barley forage, hay and straw was IN-QKT34 (IN-A2213 glucoside) which was present at 24% TRR (1.6 mg eq/kg) in forage, 40% TRR (0.48 mg eq/kg) in hay and 28% TRR (0.45 mg eq/kg) in straw. In barley forage, hay and straw, IN-D2708 (2.9–8.2% TRR, 0.05–0.23 mg eq/kg), IN-A2213 (4.6–13% TRR, 0.06–0.90 mg eq/kg) and oxamyl (5.9–24% TRR, 0.07–1.6 mg eq/kg) were also identified.

The residue in confined rotational crops is comprised of several compounds including oxamyl, IN-D2708, IN-A2213 and IN-QKT34. The concentration of these compounds is likely to be lower at longer plantback intervals.

In a field rotational crop study in Northern Europe, the GR formulation was applied using a granular applicator at planting of potatoes at a target rate of 5.5 kg ai/ha. Potatoes were removed at 80 or 120 days after the final application to allow planting of succeeding crops (lettuce, carrot, winter barley and winter wheat) at the targeted PBIs.

Oxamyl residues in succeeding crops (lettuce, carrot roots and tops, and cereal grain, hay, and straw) planted 80 and 120 days after GR application and harvested at maturity were < 0.01 (<LOD) mg/kg.

In another field rotational crop study in Southern Europe, the SL formulation was applied by simulated drip irrigation immediately after transplanting melons and four additional times with the last application being 21 days before melon harvest. Applications were made on a 10-day retreatment interval. First application was made at a target rate of 2 kg ai/ha and other applications were made at a target rate of 1 kg ai/ha. Melons were removed after the final application to allow the succeeding crops to be planted at the targeted PBIs of 30, 60, 90 or 120 days.

Oxamyl residues in rotational crops (lettuce and radish roots and radish tops) planted at 30, 60, 90 and 120 days after SL application and harvested at maturity were < 0.01 (<LOD) mg/kg.

In rotational crops, no significant oxamyl residues are expected.

### ***Methods of analysis***

The Meeting received descriptions and validation data for analytical methods for residues of oxamyl in plant and animal commodities.

In several similar methods for determination of oxamyl in plants, homogenized samples were extracted with acetone, and partitioned into dichloromethane/petroleum ether (1:1, v/v). An aliquot of extract was cleaned-up by a solid phase extraction using an aminopropyl-bonded cartridge. Oxamyl residues were determined by HPLC with post-column derivatization and fluorescence detection, MS or MS/MS detection. The methods of analysis were validated at various fortification levels with LOQs of 0.01 mg/kg for oxamyl. Another method utilised accelerated solvent extraction with acetone, and the extract was cleaned-up using an ENVI-Carb SPE cartridge and a Silica Mega Bond Elute SPE cartridge. An HPLC-UV equipped with column switching valve was used for analysis of both oxamyl and IN-A2213. The LOQs were 0.02 mg/kg for both analytes.

In the methods for determination of oxamyl in animal commodities, samples were homogenized with 0.1% formic acid in methanol. Following extraction, the sample was cleaned up with hexane and SAX (Strong Anion Exchanger) sorbent. The cleaned extracts were then subjected to LC-MS/MS analysis. The method of analysis was validated with LOQs of 0.01 mg/kg for oxamyl.

The QuEChERS method was used for determination of oxamyl residues in plant and animal commodities. The LOQ was 0.01 mg/kg for oxamyl.

The methods are suitable for the analysis of oxamyl residues in plant and animal commodities.

### ***Stability of residues in stored analytical samples***

The Meeting received information on the freezer storage stability of oxamyl in plant matrices (lettuce, tomato, sugar beet root, potato tuber and orange peel).

Storage stability results indicate that oxamyl residue was stable at approximately -18 °C for at least 24 months in lettuce (high water), tomato (high water), sugar beet root (high starch), potato tuber (high starch) and orange peel.

The periods of storage stability studies generally cover the sample storage intervals of residue trials.

### ***Definition of the residue***

Oxamyl is applied as a soil treatment for Brussels sprouts, fruiting vegetables and root and tuber vegetables. The plant metabolism studies for potato and tomato can predict the metabolites of oxamyl expected in fruiting vegetables and root and tuber vegetables. Rotational crop studies for cabbage and lettuce can predict the metabolites of oxamyl expected in Brussels sprouts.

In plant metabolism studies involving soil treatment conducted in potatoes and tomatoes, significant levels of oxamyl were only found in the foliage of both plants (1.1% TRR in potato and 6.3–19% TRR in tomato).

The metabolites IN-A2213, IN-QKT34 (IN-A2213 glucoside), IN-D2708 and IN-N0079 were major metabolites (> 10% TRR) in plants. These metabolites no longer contain the carbamate moiety.

IN-D2708 was identified in potato peels (68% TRR, 0.76 mg eq/kg) and peeled potatoes (71% TRR, 0.61 mg eq/kg). IN-QKT34 was found in tomato fruits (3.5–11% TRR, 0.016–0.077 mg eq/kg). IN-A2213 and IN-N0079 were detected in tomato fruits at 0.031–0.096 mg eq/kg (5.3–12% TRR) and 0.013–0.090 mg eq/kg (1.8–22% TRR), respectively.

No other individual metabolite was present at a level greater than 10% TRR.

The confined rotational crop studies also show that oxamyl is rapidly degraded to metabolites that no longer contain the carbamate moiety; IN-A2213, IN-D2708, IN-QKT34.

In processed commodities, oxamyl is degraded to IN-A2213 with increasing temperatures.

The toxicity of metabolites, that do not contain the carbamate moiety (IN-A2213, IN-QKT34, IN-D2708 and IN-N0079), is considered to be less than that of parent oxamyl and would be covered by the ADI and ARfD of oxamyl.

Based on the metabolism and toxicity data the Meeting concluded that oxamyl is the only residue of concern for both enforcement and for dietary risk assessment.

Analytical methods are available for the determination of oxamyl in plants.

The Meeting concluded that the residue definition for plants, for both compliance and risk assessment, is oxamyl only.

In animal metabolism studies, oxamyl was rapidly metabolized and oxamyl was not identified in any animal products. In addition, no metabolites containing the carbamate moiety were identified. The major metabolite identified in milk, eggs and tissues was thiocyanate.

Thiocyanate is a non-specific analyte present at high background levels in animals and is therefore not suitable as either a marker residue for enforcement or for the dietary risk assessment.

The Meeting concluded that oxamyl is the only residue of concern for both enforcement and for dietary risk assessment.

Analytical methods to determine residues of oxamyl in animal commodities are available.

The octanol/water coefficient (log Pow) of oxamyl is -0.43. The Meeting concluded that the residue is not fat soluble.

The Meeting recommended the following residue definition:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plants and animals: *Oxamyl*

The residue is not fat soluble.

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

The Meeting received supervised trial data for soil application of oxamyl on Brussels sprouts, cucumber, courgette, melons, tomato, peppers, eggplant, carrot, sugar beet and potato. Residue trials were conducted in Germany, Netherlands, the UK, France, Greece, Italy and Spain.

Labels from Netherlands, the UK, Italy and Spain were available.

Since no residue data (and/or use patterns) were provided for citrus fruit, apple, cotton seed, peanut, peanut fodder and spices, the Meeting withdraws the previous recommendations for maximum residue levels for these commodities.

#### *Brussels sprouts*

Data were available from supervised trials on Brussels sprouts in the Netherlands.

The GAP for Brussels sprouts in the Netherlands allows one soil application before planting at a rate of 4.0 kg ai/ha.

Oxamyl residues in Brussels sprouts from independent trials in the Netherlands matching GAP were (n=3): < 0.01 (3) mg/kg.

No oxamyl residues (< 0.01 mg/kg) are expected in Brussels sprouts according to the residues in other plant commodities such as tomatoes, peppers and eggplants and field rotational crop studies.

Based on the trials on Brussels sprouts in the Netherlands, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR and an HR value of 0 mg/kg for oxamyl in Brussels sprouts.

#### *Fruiting vegetables, Cucurbits*

##### *Cucumber*

Data were available from supervised trials on protected cucumber in Southern European countries.

The GAP for protected cucumber of Italy is two soil applications (drip irrigation) at a maximum crop cycle rate of 3.0 kg ai/ha and 10–14 days application interval with a PHI of 50 day.

Oxamyl residues in cucumbers from independent trials in Southern Europe matching Italian GAP were (n=6): < 0.01 (5) and 0.016 mg/kg.

Based on the trials on cucumbers in Southern Europe, the Meeting estimated a maximum residue level of 0.02 mg/kg, an STMR value of 0.01 mg/kg and an HR value of 0.016 mg/kg for oxamyl in cucumber to replace the previous recommendation for cucumber.

##### *Summer Squash*

Data were available from supervised trials on protected courgette in Southern European countries.

The GAP for protected courgette in Italy is two soil applications (drip irrigation) at a maximum crop cycle rate of 3.0 kg ai/ha and 10–14 days application interval with a PHI of 50 days.

Oxamyl residues in courgettes from independent trials in Southern Europe matching Italian GAP were (n=6): < 0.01 (5) and 0.022 mg/kg.

Based on the trials on courgettes in Southern Europe, the Meeting estimated a maximum residue level of 0.04 mg/kg, an STMR value of 0.01 mg/kg and an HR value of 0.022 mg/kg for oxamyl in summer squash.

##### *Melons*

Data were available from supervised trials on protected melon in Southern European countries.

The GAP in Italy and Spain for melons and watermelons is two soil applications (drip irrigation or irrigation via sprinkler) at a maximum crop cycle rate of 3.0 kg ai/ha and 10–14 days application interval with a PHI of 50 days.

Oxamyl residues in the whole fruits of melon from independent trials in Southern Europe matching GAP were (n=7): < 0.005 (5), 0.0053 and 0.0054 mg/kg.

Oxamyl residues in flesh of melon from independent trials in Southern Europe matching GAP were (n=7): < 0.005 (7) mg/kg.

Based on the trials on melon in Southern Europe, the Meeting estimated a maximum residue level of 0.01 mg/kg, an STMR value of 0.005 mg/kg and an HR value of 0.005 mg/kg for oxamyl in melons except watermelon to replace the previous recommendation for melons except watermelon.

The Meeting agreed that the maximum residue level for melons could be extrapolated to that of watermelon.

*Fruiting vegetables, other than Cucurbits**Tomato*

Data were available from supervised trials on protected tomatoes in Southern European countries.

The GAP in Italy and Spain for tomato is four soil applications (drip irrigation or irrigation via sprinkler) using a SL formulation at a maximum crop cycle rate of 5.0 kg ai/ha and 10–14 days application interval with a PHI of 28 days.

Oxamyl residues in tomatoes and cherry tomatoes from independent trials in Southern Europe matching GAP were (n=20): < 0.01 (22) mg/kg.

The GAP in Italy for protected tomato is one soil application before or at planting using a GR formulation at 3.0–3.5 kg ai/ha for soil incorporation in furrow and at 4.5–5.5 kg ai/ha for soil broadcast spray with incorporation.

Oxamyl residues in tomatoes from independent trials in Southern Europe matching GAP were (n=8): < 0.01 (8) mg/kg.

Based on the trials on tomato in Southern Europe, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR value of 0.01 mg/kg and an HR value of 0.01 mg/kg for oxamyl in tomato and cherry tomato to replace the previous recommendation for tomato.

*Peppers*

Data were available from supervised trials on protected peppers in Southern European countries.

The GAP in Italy and Spain for peppers is for three soil applications (drip irrigation or irrigation via sprinkler) using a SL formulation at a maximum crop cycle rate of 4.0 kg ai/ha and 10–14 days application interval with a PHI of 35 days.

Oxamyl residues in peppers from independent trials in Southern Europe matching GAP were (n=10): < 0.01 (10) mg/kg.

Based on the trials on pepper in Southern Europe, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR value of 0.01 mg/kg and an HR value of 0.01 mg/kg for oxamyl in subgroup of peppers (except Martynia, Okra and Roselle) to replace the previous recommendation for peppers.

*Eggplant*

Data were available from supervised trials on protected eggplants in Southern European countries.

The GAP in Italy and Spain for eggplants is four soil applications (drip irrigation or irrigation via sprinkler) using a SL formulation at a maximum crop cycle rate of 5.0 kg ai/ha and 10–14 days application interval with a PHI of 28 days.

Oxamyl residues in eggplants from independent trials in Southern Europe matching GAP were (n=5): < 0.01 (5) mg/kg.

Based on the trials on eggplant in Southern Europe, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR value of 0.01 mg/kg and an HR value of 0.01 mg/kg for oxamyl in subgroup of eggplants.

*Root and tuber vegetables**Carrot*

Data were available from supervised trials on carrots in European countries.

The GAP in the UK for carrots and parsnips is one soil application (incorporation) in seed furrow at planting of a GR formulation at a rate of 0.090 g ai/m with a PHI of 12 weeks.



Oxamyl residues in carrots from independent trials in Europe matching GAP were (n=7): < 0.01 (7) mg/kg. The residues on carrots were all less than the LOD (0.007 mg/kg).

Based on the trials on carrot in Europe, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR value of 0 mg/kg and an HR value of 0 mg/kg for oxamyl in carrot to replace the previous recommendation for carrot.

The Meeting agreed that the maximum residue level for carrots could be extrapolated to that of parsnip.

#### *Sugar beet*

Data were available from supervised trials on sugar beet in European countries.

The GAP of the Netherlands for sugar beet is one soil application (incorporation) in furrow at drilling of a GR formulation at a rate of 0.75–2.5 kg ai/ha.

Oxamyl residues in sugar beets from independent trials in Europe matching GAP were (n=19): < 0.01 (11) and < 0.02 (8) mg/kg. The residues on sugar beets were all less than the LOD (0.005 or 0.01 mg/kg).

Based on the trials on sugar beet in Europe, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR value of 0 mg/kg and an HR value of 0 mg/kg for oxamyl in sugar beet.

#### *Potato*

Data were available from supervised trials on potatoes in European countries.

The GAP in the UK for potato is one soil application (incorporation) at planting of a GR formulation at a rate of 5.5 kg ai/ha with a PHI of 80 days.

Oxamyl residues in potatoes from independent trials in Europe matching GAP were (n=8): < 0.005 (8) mg/kg. The residues on potatoes were all less than the LOD (0.0033 mg/kg).

Based on the trials on potato in Europe, the Meeting estimated a maximum residue level of 0.01 (\*) mg/kg, an STMR value of 0 mg/kg and an HR value of 0 mg/kg for oxamyl in potato to replace the previous recommendation for potato.

### ***Fate of residues during processing***

#### *High temperature hydrolysis*

The hydrolytic stability of [<sup>14</sup>C]-oxamyl was studied under conditions of high temperature in sterile aqueous buffers at pH 4, 5 and 6 for periods of up to 60 minutes so as to simulate common processing practices (pasteurization, baking/boiling, and sterilization). At pH 4 heating at 90 °C for 20 min, no degradation was observed. At pH 5 heating at 100 °C for 60 min, 58% of test material degraded to IN-A2213, while all the test material degrades to IN-A2213 after incubation at pH 6, at 120 °C for 20 min.

#### *Residues in processed commodities*

The fate of oxamyl residues has been examined in potato processing studies. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P and HR-P for food and feed

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
		Oxamyl	Oxamyl				
Potato tuber	Baked	< 0.05, 0.08, 0.15	0.08	0	0	0	0
	Boiled	< 0.04, < 0.04, < 0.05	< 0.04		0		0
	Microwaved	< 0.04, < 0.04, < 0.05	< 0.04		0		0

\* Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

### ***Residue in animal commodities***

#### *Farm animal dietary burden*

The Meeting estimated the dietary burden of oxamyl in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual third edition, 2016. Calculations from the highest residue, STMR (some bulk commodities) and STMR-P values provide levels in feed suitable for estimating MRLs, while calculations using STMR and STMR-P values for feed are suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed on a dry weight basis.

#### *Estimated maximum and mean dietary burdens of farm animals*

The calculations were made according to the animal rations from US-Canada, EU, Australia and Japan in the Table (Appendix IX of the 2016 edition of the FAO manual).

Potential feed items include: tomato wet pomace, carrot culls, sugar beet molasses and potato culls.

Livestock dietary burden, oxamyl, ppm of dry matter diet								
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0	0	0	0	0.005 <sup>a</sup>	0.005 <sup>b</sup>	0	0
Dairy cattle	0	0	0	0	0.005 <sup>c</sup>	0.005 <sup>d</sup>	0	0
Poultry – broiler	-	-	-	-	-	-	-	-
Poultry – layer	-	-	-	-	-	-	-	-

<sup>a</sup> - Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian meat, fat and edible offal

<sup>b</sup> - Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian meat, fat and edible offal

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

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

#### *Farm animal feeding studies*

Farm animal feeding studies were not submitted.

#### ***Animal commodities maximum residue levels***

For MRL estimation, the residue definition in the animal commodities is oxamyl.

The maximum dietary burden for beef and dairy cattle is 0.005 ppm which is lower than the dose level in the lactating goat metabolism study (31 ppm). In the studies, no measured residues of oxamyl were found in any of analysed tissues and milk. No feed items for poultry were applicable.

The Meeting estimated maximum residue levels at the LOQ of 0.01\* mg/kg and STMRs/HRs of 0 for animal products as milk, mammalian meat, mammalian edible offal and mammalian fat to replace the previous recommendations for milk, mammalian meat and mammalian edible offal. The Meeting withdrew the previous recommendations for eggs, poultry meat and poultry, edible offal of.

## **RECOMMENDATIONS**

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

Plant and Animal commodities:

Definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary exposure): *Oxamyl*.

*The residue is not fat soluble.*

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
FP 0226	Apple	W	2		
VB 0402	Brussels sprouts	0.01*	-	0	0
VR 0577	Carrot	0.01*	0.1	0	0
VO 2700	Cherry tomato	0.01*	-	0.01	0.01
FC 0001	Citrus fruit	W	3		
SO 0691	Cotton seed	W	0.2		
VC 0424	Cucumber	0.02	1	0.01	0.016
MO 0105	Edible offal (Mammalian)	0.01*	0.02*	0	0
PE 0112	Eggs	W	0.02*		
MF 0100	Mammalian fats (except milk fats)	0.01*	-	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.01*	0.02*	0	0
VC 0046	Melons, except Watermelon	0.01	1	0.005	0.005
ML 0106	Milks	0.01*	0.02*	0	0
VR 0588	Parsnip	0.01*	-	0	0
SO 0697	Peanut	W	0.05		
AL 0697	Peanut fodder	W	0.2 (dry weight)		
VR 0589	Potato	0.01*	0.1	0	0
PO 0111	Poultry, Edible offal of	W	0.02*		
PM 0110	Poultry meat	W	0.02*		
HS 0191	Spices, Fruits and Berries	W	0.07		
HS 0193	Spices, Roots and Rhizomes	W	0.05		
VC 0431	Squash, Summer	0.04	-	0.01	0.022
VO 2046	Subgroup of Eggplants (includes all commodities in this subgroup)	0.01*	-	0.01	0.01
VO 0051	Subgroup of Peppers (except Martynia, Okra and Roselle)	0.01*	5	0.01	0.01
VR 0596	Sugar beet	0.01*	-	0	0
VO 0448	Tomato	0.01*	2	0.01	0.01
VC 0432	Watermelon	0.01	-	0.005	0.005

Table of additional STMR and HR values for use in dietary intake estimation

Commodity name	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
Potato, baked	0	0
Potato, boiled/microwaved	0	0

## DIETARY RISK ASSESSMENT

### *Long-term dietary exposure*

The International Estimated Daily Intakes (IEDIs) of oxamyl were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (Annex 3 of the 2017 Report). The ADI is 0–0.009 mg/kg bw and the calculated IEDIs were 0–1% of the maximum ADI (0.009 mg/kg bw). The Meeting concluded that the long-term dietary exposure to residues of oxamyl, resulting from the uses considered by current JMPR, is unlikely to present a public health concern.

*Short-term dietary exposure*

The International Estimated Short-Term Intakes (IESTI) of oxamyl were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (Annex 4 of the 2017 Report). The ARfD is 0.009 mg/kg bw and the calculated IESTIs were a maximum of 20% of the ARfD for the general population and 10% of the ARfD for children. The Meeting concluded that the short-term dietary exposure to residues of oxamyl, when used in ways that have been considered by the current JMPR, is unlikely to present a public health concern.

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