DIMETHOATE (027)/OMETHOATE (055)

First draft prepared by Dr S Margerison and Dr P Humphrey, Australian Pesticides and Veterinary Medicines Authority, Australia

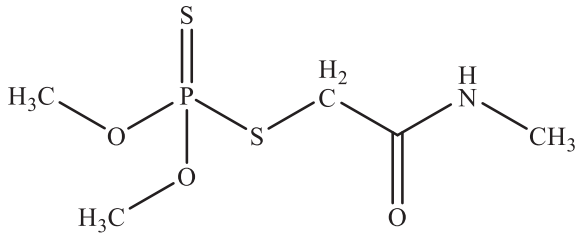
EXPLANATION

Dimethoate is an organophosphate insecticide which acts through acetylcholinesterase inhibition. It was scheduled for periodic review evaluation by the 2019 JMPR at the Fiftieth Session of the CCPR (2018). Dimethoate has been evaluated on numerous occasions by the JMPR commencing in 1963. The most recent periodic review was in 1996 (toxicology) and 1998 (residues), with a subsequent evaluation for toxicology and residues in 2003 to establish an acute reference dose and consider additional plant metabolism studies. Residue data for additional MRLs were evaluated in 2006, and 2008.

The Meeting considered information supplied by the sponsor on identity, physicochemical properties, plant and animal metabolism and environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing, and animal feeding studies. Additional supervised residue trial data was supplied by Australia for mandarin, oranges, avocados, mangoes, capsicum and pulses, and by Thailand for yard-long bean.

Dimethoate has been evaluated by the Joint Meeting on Pesticide Specifications (JMPS) and the most recent specification was published in 2012.

IDENTITY

ISO common name:	Dimethoate
IUPAC name:	*O,O*-Dimethyl *S*-methylcarbamoylmethyl phosphorodithioate Or 2-methoxyphosphinothioylthio-*N*-methylacetamide
Chemical Abstracts name:	*O,O*-Dimethyl *S*-[2-(methylamino)-2-oxoethyl] phosphorodithioate
CAS No.:	60-51-5
Molecular Formula:	$C_5H_{12}NO_3PS_2$
Structural Formula:	
Molecular Weight:	229.26 g/mol

ISO common name:	Omethoate
IUPAC name:	*O,O*-Dimethyl *S*-methylcarbamoylmethyl phosphorothioate Or 2-methoxyphosphinoylthio-*N*-methylacetamide
Chemical Abstracts name:	*O,O*-Dimethyl *S*-[2-(methylamino)-2-oxoethyl] phosphorothioate
CAS No.:	1113-02-6
Molecular Formula:	$C_5H_{12}NO_4PS$

Dimethoate

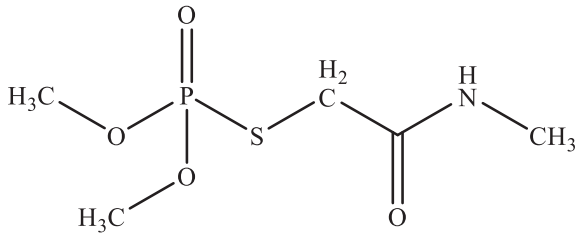
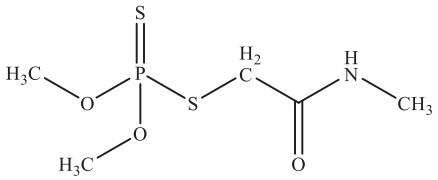
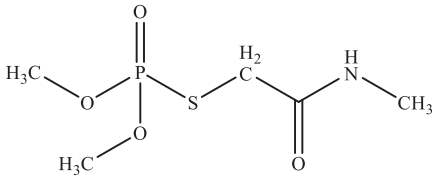
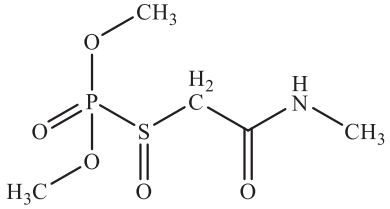
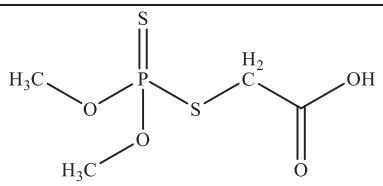
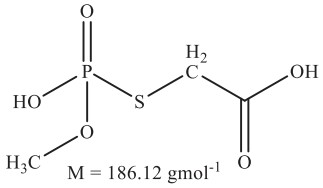
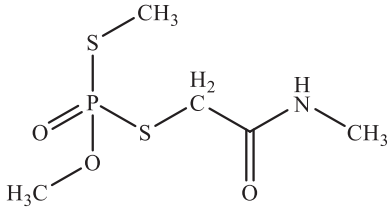
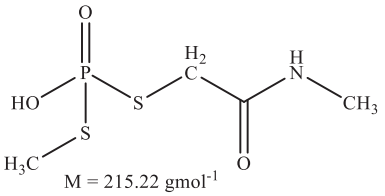
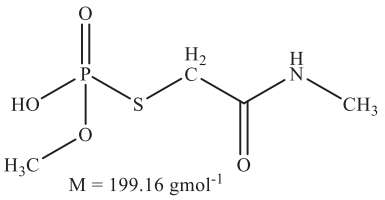
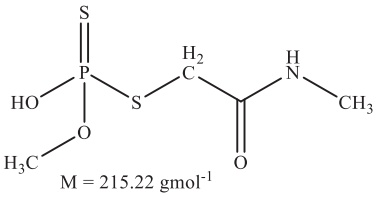
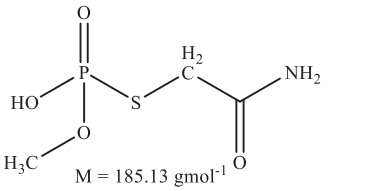
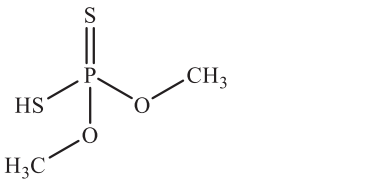
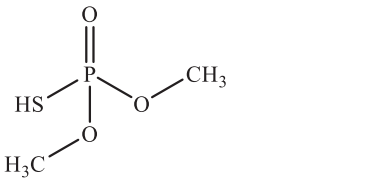
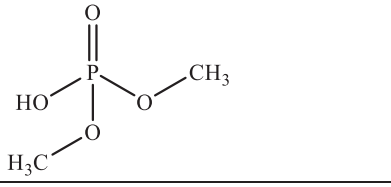
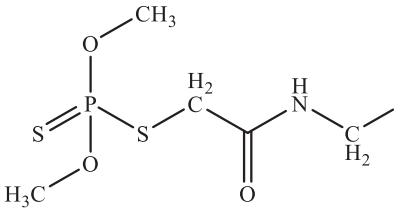
ISO common name:	Omethoate
Structural Formula:	
Molecular Weight:	213.19 g/mol

Table 1 Metabolites of dimethoate

Component name	Structure	Origin
Dimethoate		Parent compound
Omethoate		Potato, olives, wheat, rat, goat, hen
Omethoate sulfoxide		Goat (intermediate), hen (intermediate)
Dimethoate carboxylic acid	 M = 216.21 gmol^{-1}	Potato, olives, wheat, rat, goat, hen
O-desmethyl omethoate carboxylic acid	 M = 186.12 gmol^{-1}	Potato, wheat
Isodimethoate		Olives (intermediate), wheat (probable intermediate)

Component name	Structure	Origin
O-desmethyl isodimethoate	 M = 215.22 gmol^{-1}	Potato, olives, wheat
O-desmethyl omethoate	 M = 199.16 gmol^{-1}	Potato, olives, wheat
Desmethyl dimethoate	 M = 215.22 gmol^{-1}	Potato, hydrolysis, soil (minor component)
O-desmethyl N-desmethyl omethoate	 M = 185.13 gmol^{-1}	Potato, olives, wheat
Dimethyl dithiophosphate and conjugates		Potato, wheat, rat, goat, hen
Dimethyl thiophosphate		Hydrolysis
Dimethyl phosphate		Rat
Hydroxy dimethoate glucose conjugate		Potato, wheat

PHYSICAL AND CHEMICAL PROPERTIES

Table 2 Physicochemical properties of dimethoate – for pure active ingredient (except where noted otherwise)

Property	Results	Test method	Test material purity and specification	Reference
Dimethoate				
Appearance	White solid with a mercaptanic odour, some tendency to cake	Visual and olfactory inspection	Technical	Jorgensen-2002
Melting point	49–52 °C	Capillary/metal block – visual inspection	99.4%	Comb – 2005a, SCI111/052216
Boiling point	Decomposed above 113 °C	Capillary method	99.4%	Comb – 2005b, SCI110/052209
Relative density	$D_4^{20} = 1.31$	Pycnometer	99.1%	Young – 2001, SCI067/004784, 430 DMT
Flammability	Not flammable	EEC Method A10, EPA/ OPPTS 830.6315	Technical, 98.0%	Comb – 2005c, SCI109/052215
Auto-ignition temperature	350 °C	EEC Method A15	Technical, 98.0%	Comb – 2005d, SCI108/052211
Explosive properties	Not explosive (under thermal, shock or friction stresses)	EEC Method A14 – flame heating, hammer and anvil test, porcelain friction plate and peg	Technical, 98.0%	Comb – 2005e, SCI07/052210
Vapour pressure	2.5×10^{-3} Pa (25 °C)	Vapour pressure microbalance, EEC method A4, OECD 104	99.1%	Comb – 2013a, 1500 DMT
Water solubility (20 °C)	Purified water: 28.2 g/L pH 5 buffer: 25.8 g/L pH 7 buffer: 25.9 g/L pH 9 buffer: 27.1 g/L	Shaken flask method, EEC method A6, OECD 105	99.1%	Comb – 2013b, 1555 DMT
Organic solvent solubility (25 °C)	Solubility ± s.d. (g/100 mL) Acetone: 139 ± 11 Acetonitrile: 142 ± 14 Cyclohexanone: 122 ± 4 Dodecane: 0.043 ± 0.002 Ethanol: 153 ± 8 Ethyl acetate: 124 ± 11 Hexane: 0.0295 ± 0.0009 Isopropanol: 120 ± 9 Methanol: 159 ± 14 Dichloromethane: 150 ± 9 1-Octanol: 52.2 ± 4.7 Toluene: 103 ± 9 Xylenes: 31.3 ± 0.3 1,2-Dichloroethane: 121 ± 9 n-Heptane: 0.0242 ± 0.002	Stirred flask method	99.1%	Madsen – 1994, 221 DMT

Property	Results	Test method	Test material purity and specification	Reference
Octanol-water partition coefficient (20 °C)	$\log_{10}P_{OW} = 0.75$	Shaken flask method, EEC method A8, OECD 107	99.1%	Comb – 2013c, 1554 DMT
Henry's Law Constant (20 °C)	1.52×10^{-5} Pa m^3mol^{-1}	Calculated		Hogh – 2015, 1703 DMT
Hydrolysis (half-life, days – at 25 °C)	pH 5 buffer: 156 pH 7 buffer: 68 pH 9 buffer: 4.4		^{14}C-O-methyl dimethoate	Kirkpatrick – 1986a, 217 DMT
Aqueous photolysis (25 °C)	pH 5 buffer: negligible degradation, half-life >175 days		^{14}C-O-methyl dimethoate	Kirkpatrick – 1986b, 206 DMT
^{1}H NMR chemical shifts (ppm)	2.85 (d), 3.54 (d), 3.80 (d), ~6.5 (s)	400 MHz Bruker Avance II 400 NMR spectrometer	99.6%	Lindhart 2007, 020-05
^{13}C NMR chemical shifts (ppm)	27.76, 36.25, 54.53, 167.99		99.6%	Lindhart 2007, 020-05
^{31}P NMR chemical shift (ppm)	99.03		99.6%	Lindhart 2007, 020-05
IR spectroscopy (cm^{-1})	3259 + 3092 (N-H stretch) 3000-2800 (C-H stretch) 1649 (C=O stretch) 1570 (N-H bending) 1009 (P-O-C stretch) 832 + 651 (P=S stretch)	Perkin-Elmer Paragon 1000 FT-IR spectrometer, spectrum measured as a KBr disc	99.6%	Lindhart 2007, 020-05
UV spectroscopy	Absorbance at ~195 nm	HPLC with UV diode array detector	99.6%	Lindhart 2007, 020-05

Table 3 Physicochemical properties of omethoate

Property	Value	Reference
Melting point	-28 °C	BCPC Pesticide Manual
Vapour pressure	0.0033 Pa (20 °C)	BCPC Pesticide Manual
Water solubility	>500 g/L (25 °C)	BCPC Pesticide Manual
Organic solvent solubility	Soluble in acetone and alcohols, slightly soluble in diethyl ether, practically insoluble in petroleum ether	BCPC Pesticide Manual
Octanol/water partition coefficient	$\log_{10}K_{OW} = -0.74$	BCPC Pesticide Manual

METABOLISM AND ENVIRONMENTAL FATE

PLANT METABOLISM

Metabolism studies were provided for ^{14}C-methyl-labelled dimethoate in olives, potatoes, and wheat. In addition, reports from studies with more limited information were available for [^{32}P]-dimethoate in lemons, sugar beet, maize, cotton, peas, and potatoes, and for [^{14}C-methoxy]-dimethoate in beans.

Olives

Dimethoate labelled with ^{14}C in both methoxy groups was mixed with an EC formulation containing non-labelled material (Corden – 2005) and applied to an olive tree (Manzanillo variety) grown

outdoors in California as a foliar spray (4 × 0.72 kg ai/ha applications with retreatment intervals of 57, 30, and 44 days). The first application was made at BBCH 51–69 (inflorescence emergence to end of flowering) with the final application at BBCH 75–89 (fruit at 50% final size and above), 28 days before harvest maturity. Only one branch of the tree was treated, with the rest of the tree shielded from the spray by polythene sheeting. Fruit was sampled just before and just after the third application, at two intervals between the third and fourth applications, and at 0-, 0+ (green and black olives), 14, 21 and 28 days (green and black olives) after the final application. Leaves were sampled after the first application, 43 days after the third application and 14, 21 and 28 days after the fourth application.

Samples were stored in coolers for transport to the field phase laboratory, frozen (<-15 °C) then transported on dry ice to the analytical laboratory, where they were kept frozen until analysis.

Leaf samples were surface washed three times with acetonitrile, then homogenised with acetonitrile. The acetonitrile extraction was repeated twice, followed by two extractions with acetonitrile/water (1:1 v/v). The leaf washes and extracts were analysed by LSC, and then pooled and concentrated for analysis by TLC and HPLC. The post-extraction solids were subsampled, and analysed for residual radioactivity by combustion and LSC.

Whole olives were surface washed three times with acetonitrile. Washed olive samples were separated into flesh and stone. Both fractions were homogenised and the total radioactivity determined by combustion and LSC.

Homogenised flesh samples were extracted first by homogenisation and ultrasonication with three times with hexane, three times with acetonitrile, three times with acetonitrile/water (1:1 v/v), and twice with water. Extracts were analysed by LSC. The post-extraction solids were subsampled, and analysed for residual radioactivity by combustion and LSC. Hexane extracts were pooled and partitioned with three aliquots of acetonitrile, acetonitrile extracts were pooled and partitioned with three aliquots of hexane, while water/acetonitrile and water extracts were partitioned with ethyl acetate. Hexane fractions with significant levels of radioactive residue were pooled and concentrated, as were acetonitrile fractions, acetonitrile/water extracts and water extracts with significant levels of radioactivity. Fractions were then analysed by TLC and HPLC. The post-extraction solids were subsampled again for further alternate aggressive extraction methods: acid (0.1 or 1 M HCl for 20 hours at 37 °C), base (0.1 or 1 M NaOH for 20 hours at 37 °C) or enzymatic (bacterial protease type VIII from *Bacillus licheniformis* in phosphate buffer for 20 hours at 37 °C, β-glucosidase type II from almonds in acetate buffer for 20 hours at 37 °C, or hemicellulase and cellulase from *Aspergillus niger* in acetate buffer for 20 hours at 37 °C) treatment, followed by extraction (after neutralisation of the acid/base treated samples with NaOH or HCl as appropriate) with acetonitrile. Where significant residues (> 10% TRR) remained, further treatment with strong base (6 M) was undertaken, followed by extraction with acetonitrile. Subsamples of the aqueous extractions from olive samples taken 29 days after application 3 were treated by incubation with 1 M HCl, 1 M NaOH, or β-glucosidase, followed by TLC analysis.

Homogenised stone samples were extracted by homogenisation twice each with acetonitrile, acetonitrile/water (1:1 v/v), and water. Levels of radioactivity were determined in the extracts by LSC and in the post-extraction solids by combustion and LSC. Extracts containing significant levels of radioactivity were pooled and concentrated and analysed by TLC and HPLC.

Extensive further work was conducted to characterise a number of minor components present in olive flesh and/or stone, and which did not co-chromatograph with any of the available reference substances. This included dialysis of extracts, saponification by refluxing with ethanolic potassium hydroxide, partitioning of acetonitrile extracts with ethyl acetate with adjustment of the pH of the aqueous fraction to 1-2 followed by further partitioning with ethyl acetate, isolation and further TLC and 2D-TLC, and treatment with acid, base or β-glucosidase.

Analytical techniques used to identify, characterise and quantify residue components included LSC, combustion with LSC of captured $^{14}CO_2$, reverse phase radio-HPLC-UV with co-

chromatography with reference standards and fraction collection and LSC, and normal and reverse phase TLC with co-chromatography.

All samples were analysed within 3 months of collection.

Table 4 Total radioactive residues in olive fruit and leaves

Date of collection	Timing	TRR (mg eq/kg)		
		Olive flesh	Olive stone	Leaves
3 June 2002	0DAA1	NA	NA	42.4
29 August 2002	30DAA2	3.28	2.02	NA
29 August 2002	0DAA3	4.91	2.58	NA
27 September 2002	29DAA3	5.33	2.74	NA
11 October 2002	43DAA3	4.36	2.99	NA
11 October 2002	0DAA4	5.94	2.93	NA
8 November 2002	28DAA4 (green olives)	3.93	3.27	NA
8 November 2002	28DAA4 (black olives)	3.69	2.70	NA

As expected, for leaves collected on the day of the first application, residues were easily removed by washing or simple solvent extraction, with 69.6% TRR (29.5 mg eq/kg) removed by the surface washes, and 30.3% TRR (12.8 mg eq/kg) extracted with acetonitrile and acetonitrile water, leaving only 0.2% TRR (0.08 mg eq/kg) unextractable. Most of the residue was present as unchanged parent compound (see Table 5 for further details).

Table 5 Residue components in olive leaves after 1 × 0.72 kg ai/ha applications of dimethoate

Component	Olive leaves 0DAA1	
	% TRR	mg eq/kg
OL-A-1	1.2	0.51
Omethoate	< 0.2	< 0.08
Dimethoate	95.8	40.6
Other	2.9	1.23
Total extracted	99.9	42.3
Unextractable residues	0.2	0.08
Total	100	42.4

For olive flesh, small fractions of radioactivity were removed in surface washes (1.5–17.2% TRR or 0.05-1.02 mg eq.kg), with the majority being extracted with hexane, acetonitrile, and acetonitrile/water (72.8–87.7% TRR or 2.49–4.32 mg eq/kg). Harsher extractions removed further proportions of the radioactive residues, with based extractions being the most successful, removing up to 10.2% TRR, reflecting the incorporation of some radioactivity into fatty acids. Details of the residue components identified in olive flesh are given in Table 6 and Table 7 below.

Table 6 Residue components in olive flesh after 2 or 3 × 0.72 kg ai/ha applications of dimethoate

Component	Olive flesh 30DAA2		Olive flesh 0DAA3		Olive flesh 29DAA3		Olive flesh 43DAA3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
OL-A[a]	3.6	0.12	2.1	0.11	2.1	0.11	2.2	0.01

Component	Olive flesh 30DAA2		Olive flesh 0DAA3		Olive flesh 29DAA3		Olive flesh 43DAA3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
OL-B	1.7	0.06	1.6	0.08	2.1	0.11	< 0.1	< 0.01
O-desmethyl N-desmethyl omethoate	44.7	1.46	35.7	1.76	53.7	2.86	53.4	2.32
O-desmethyl omethoate	< 0.1	< 0.01	< 0.1	< 0.01	1.7	0.09	< 0.1	< 0.01
OL-Db	< 0.1	< 0.01	< 0.1	< 0.01	1.1	0.06	< 0.1	< 0.01
O-desmethyl isodimethoate	2.4	0.07	1.5	0.07	4.0	0.21	5.7	0.25
Dimethoate carboxylic acid	2.7	0.08	1.1	0.06	1.1	0.05	2.3	0.10
OL-G	0.1	< 0.01	0.2	0.01	< 0.1	< 0.01	< 0.1	< 0.01
Isodimethoate	2.5	0.08	0.5	0.03	0.2	0.01	< 0.1	< 0.01
Omethoate	2.9	0.10	3.2	0.16	2.0	0.11	0.4	0.02
OL-Ia	< 0.1	< 0.01	< 0.01	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Dimethoate	1.2	0.03	30.9	1.52	0.7	0.04	0.3	0.01
OL-Ka	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-Kb	< 0.1	< 0.01	< 0.1	< 0.01	0.2	0.01	1.2	0.05
OL-L1	0.1	< 0.01	0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L2	0.1	< 0.01	< 0.1	< 0.01	0.1	0.01	< 0.1	< 0.01
OL-L3	0.1	< 0.01	0.1	< 0.01	< 0.01	< 0.01	< 0.1	< 0.01
OL-L4[b]	5.8	0.19	3.5	0.16	6.4	0.34	9.7	0.42
Others	9.4	0.30	6.7	0.33	8.4	0.50	10.9	0.47
Total extracted	77.1	2.52	87.3	4.29	83.9	4.47	86.0	3.75
Extracts not analysed	0.3	0.01	-	-	0.4	0.02	0.4	0.02
1 M base extractable[c]	10.2	0.33	6.6	0.32	4.4	0.23	5.3	0.23
6 M base extractable	8.3	0.27	-	-	9.9	0.53	-	-
Unextractable residue	4.1	0.13	6.2	0.30	1.5	0.08	8.4	0.37
Total	100	3.28	100	4.91	100	5.33	100	4.35

[a] Composed of several minor components.

[b] Radioactivity associated with natural constituent – triglycerides and sterols.

[c] Composed of O-desmethyl N-desmethyl omethoate, polar baseline material, eq/kg OL-Ia, and OL-Ka.

Table 7 Residue components in olive flesh after 4 × 0.72 kg ai/ha applications of dimethoate

Component	Olive flesh 0DAA4		Green olive flesh 28DAA4		Black olive flesh 28DAA4	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
OL-A[a]	1.8	0.11	2.8	0.11	4.6	0.17
OL-B	< 0.1	< 0.01	< 0.1	< 0.01	1.4	0.05
O-desmethyl N-desmethyl omethoate	35.5	2.13	57.0	2.23	59.9	2.22

Component	Olive flesh 0DAA4		Green olive flesh 28DAA4		Black olive flesh 28DAA4	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
O-desmethyl omethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-Db	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-desmethyl isodimethoate	3.2	0.19	6.9	0.27	6.7	0.25
Dimethoate carboxylic acid	1.2	0.07	1.2	0.05	< 0.1	< 0.01
OL-G	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Isodimethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Omethoate	1.8	0.11	2.3	0.10	2.5	0.09
OL-Ia	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Dimethoate	32.7	1.94	0.5	0.02	0.4	0.02
OL-Ka	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-Kb	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L1	0.2	0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L2	0.2	0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L3	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L4 [b]	6.0	0.37	8.6	0.34	8.6	0.32
Others	6.9	0.41	8.8	0.34	6.9	0.25
Total extracted	89.9	5.34	88.1	3.46	90.9	3.36
Extracts not analysed	0.1	0.01	0.6	0.02	0.3	0.01
1 M base extractable[c]	4.1	0.24	4.9	0.19	7.3	0.27
6 M base extractable	-	-	-	-	-	-
Unextractable residue	5.9	0.35	6.5	0.26	1.5	0.06
Total	100	5.94	100	3.93	100	3.65

[a] Composed of several minor components.

[b] Radioactivity associated with natural constituent – triglycerides and sterols.

[c] Composed of O-desmethyl N-desmethyl omethoate, polar baseline material, eq/kg OL-Ia, and OL-Ka.

Much lower proportions of the residue were readily extractable from olive stone samples, with acetonitrile, acetonitrile/water and water extractions removing only 28.8–44.4% TRR (0.58–1.13 mg eq/kg), with unextractable residues correspondingly comprising 55.6–71.2% TRR (1.32–1.86 mg eq/kg). Further details of the residue components identified in stone are given in Tables 8 and 9 below.

Table 8 Residue components in olive stone after 2 or 3 × 0.72 kg ai/ha applications of dimethoate

Component	Olive stone 30DAA2		Olive stone 0DAA3		Olive stone 29DAA3		Olive stone 43DAA3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
OL-A [a]	2.3	0.05	1.6	0.04	< 0.1	< 0.01	1.9	0.06
OL-B	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-desmethyl N-desmethyl omethoate	10.2	0.21	11.7	0.30	7.2	0.20	20.2	0.61

Component	Olive stone 30DAA2		Olive stone 0DAA3		Olive stone 29DAA3		Olive stone 43DAA3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
O-desmethyl omethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-Db	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-desmethyl isodimethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Dimethoate carboxylic acid	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-G	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Isodimethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Omethoate	1.6	0.03	1.8	0.05	< 0.1	< 0.01	1.1	0.03
OL-Ia	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Dimethoate	4.1	0.08	8.8	0.23	< 0.1	< 0.01	2.2	0.07
OL-Ka	< 0.1	< 0.01	< 0.1	< 0.01	32.4	0.89	< 0.1	< 0.01
OL-Kb	1.3	0.03	1.0	0.03	0.3	0.01	1.4	0.04
OL-L1	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L2	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L3	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L4	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Others	9.2	0.19	8.8	0.23	0.2	< 0.01	10.8	0.32
Total extracted	28.8	0.58	33.6	0.87	40.1	1.10	37.7	1.13
Extracts not analysed	-	-	-	-	-	-	-	-
1 M base extractable	-	-	-	-	-	-	-	-
6 M base extractable	-	-	-	-	-	-	-	-
Unextractable residue	71.2	1.44	66.4	1.71	59.9	1.64	62.3	1.86
Total	100	2.02	100	2.58	100	2.74	100	2.99

[a] Composed of several minor components.

Table 9 Residue components in olive stone after 4 × 0.72 kg ai/ha applications of dimethoate

Component	Olive stone 0DAA4		Green olive stone 28DAA4		Black olive stone 28DAA4	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
OL-A [a]	2.1	0.06	2.7	0.06	3.5	0.09
OL-B	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-desmethyl N-desmethyl omethoate	23.3	0.68	28.4	0.67	21.4	0.57
O-desmethyl omethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-Db	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-desmethyl isodimethoate	< 0.1	< 0.01	1.7	0.04	< 0.1	< 0.01

Component	Olive stone 0DAA4		Green olive stone 28DAA4		Black olive stone 28DAA4	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dimethoate carboxylic acid	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-G	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Isodimethoate	< 0.1	< 0.01	< 0.1	< 0.01	1.5	0.04
Omethoate	< 0.1	< 0.01	1.6	0.04	1.0	0.03
OL-Ia	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Dimethoate	5.2	0.15	0.8	0.02	2.2	0.06
OL-Ka	< 0.1	< 0.01	1.4	0.03	< 0.1	< 0.01
OL-Kb	1.4	0.04	< 0.1	< 0.01	< 0.1	< 0.01
OL-L1	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L2	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L3	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
OL-L4	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Others	10.2	0.30	7.8	0.18	3.5	0.09
Total extracted	42.2	1.24	44.4	1.05	33.1	0.89
Extracts not analysed	-	-	-	-	-	-
1 M base extractable	-	-	-	-	-	-
6 M base extractable	-	-	-	-	-	-
Unextractable residue	57.8	1.69	55.6	1.32	66.9	1.81
Total	100	2.93	100	2.37	100	2.70

[a] Composed of several minor components.

In olive flesh, the largest component of the residue at all intervals was O-desmethyl-N-desmethyl omethoate, at 35.5–59.9% TRR (1.46–2.86 mg eq/kg). Parent dimethoate was a major component only immediately after an application, at 30.9% TRR (1.52 mg eq/kg) on the day of the third application, and 32.7% TRR (1.94 mg eq/kg) on the day of the fourth application. At all other intervals, parent was only a minor component, at 0.3–1.2% TRR (0.01–0.04 mg eq/kg). After O-desmethyl-N-desmethyl omethoate, the next largest component was OL-L4, which comprised mainly radioactivity incorporated into triglycerides and sterols, at 3.5–9.7% TRR (0.16–0.42 mg eq/kg). Other components identified in olive flesh were O-desmethyl isodimethoate, at 1.5–6.9% TRR (0.07–0.27 mg eq/kg), omethoate, at 0.4–3.2% TRR (0.02–0.16 mg eq/kg), dimethoate carboxylic acid, at < 0.1–2.7% TRR (< 0.01–0.10 mg eq/kg), O-desmethyl omethoate, at < 0.1–1.7% TRR (< 0.01–0.09 mg eq/kg), and isodimethoate, at < 0.1–2.5% TRR (< 0.01–0.08 mg eq/kg).

The major metabolic pathways for dimethoate in olives are:

- Oxidation to omethoate, followed by O- and N-demethylation, with O-desmethyl-N-desmethyl omethoate as the final product;
- Isomerisation to isodimethoate, followed by O-demethylation;
- Hydrolysis of the amide to give dimethoate carboxylic acid.

Minor pathways include incorporation into natural products, particularly fatty acids and sterols.

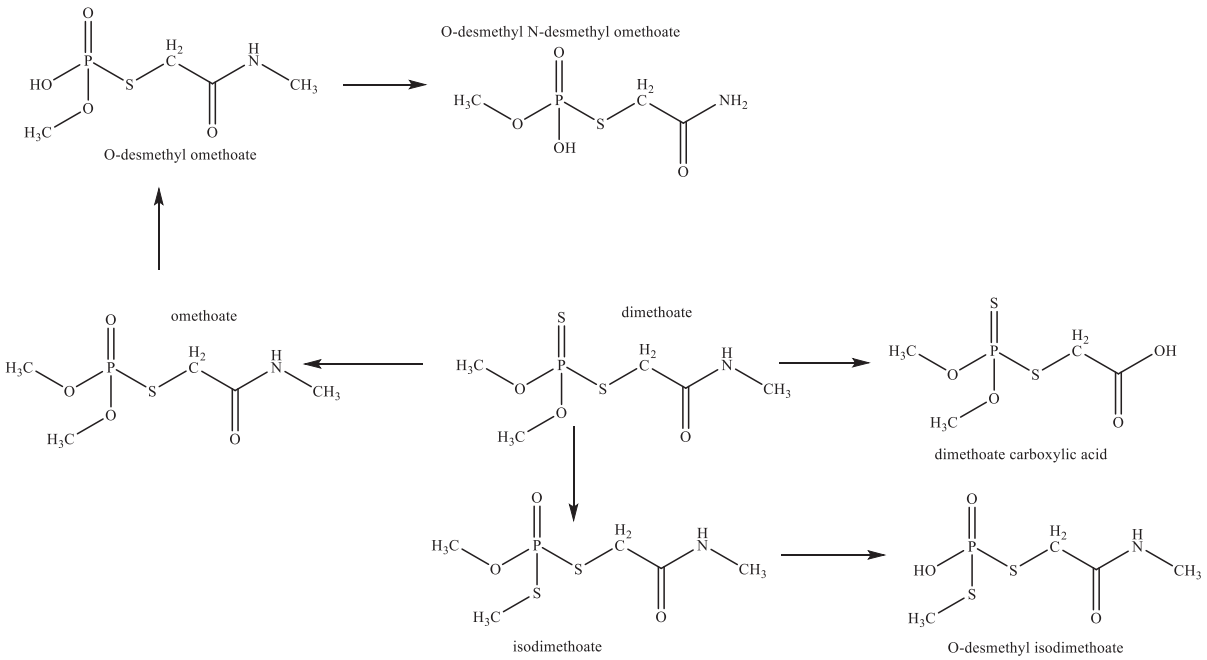


Figure 1 Metabolic pathway for dimethoate in olives

Potatoes

Dimethoate labelled with ^{14}C in both methoxy groups was mixed with an EC formulation containing non-labelled material (Corden-2000). The mixture was applied to potatoes (BBCH 45–47) as a foliar spray at a target rate of 2 × 0.34 kg ai/ha with 14 days between applications. The potato plants were grown to maturity outdoors in individual containers. Samples were collected immediately after each application and at intervals up to 28 days after the second application. Foliage was surface washed with acetonitrile, then homogenised and extracted with acetonitrile, acetonitrile/water and water. Tubers were homogenised and extracted with acetonitrile and acetonitrile/water. Levels of radioactivity in the surface washes and extracts were determined by liquid scintillation counting (LSC), and levels of radioactivity in unextractable residues were determined by combustion followed by LSC. Extracts containing significant radioactivity were analysed by HPLC and TLC with comparison against reference substances. Unextractable residues were further investigated by treating subsamples in parallel with acid (0.1 M HCl for 20 hours at 37 °C), base (0.1 M NaOH for 20 hours at 37 °C), or enzyme (bacterial protease type VIII digestion in phosphate buffer at 37 °C for 20 hours). Day 14 samples after protease extraction were further base extracted with 0.1 and 1.0 M NaOH.

Further work was carried out in a subsequent study (Corden – 2001a) to identify three unknown components characterised initially in the potato metabolism study, components A, G, and K.

Component A was present in foliage (up to 0.8%, 0.10 mg/kg) and tubers (up to 7.4%, 0.02 mg/kg). TLC demonstrated that it was a polar component which remained on the baseline following elution with moderately polar systems. Component A was isolated from extracts of potato foliage by partitioning and preparative TLC at low yield. Dialysis suggested component A was a chromatographic artefact due to high molecular weight co-extractives in the samples being analysed. In investigations of a component in the wheat study co-eluting with the potato component A, the artefact was due to retained O-desmethyl N-desmethyl omethoate, however the concentrations in potato matrices were too low to permit conclusive identification.

Component G was present in potato foliage representing up to 7.7% of the TRR (0.36 mg/kg) on day 7, decreasing to 4.8% (0.06 mg/kg) after 14 days. Component G was isolated by TLC and HPLC and investigated by LC-MS and GC-MS. Hydrolytic treatments demonstrated that component G is a glucose conjugate of hydroxy dimethoate.

Component K was present in potato foliage and tubers (up to 3.2%, 0.10 mg/kg in foliage). Component K was isolated by TLC and HPLC. Chromatographic investigations demonstrated that component K was composed of up to 6 minor components all representing < 0.05 mg/kg.

Table 10 Extractability of dimethoate residues from potato tubers and foliage following 2 × 0.34 kg ai/ha foliar applications

Fraction	Day 0		Day 2		Day 7		Day 14		Day 21		Day 28	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
	Foliage											
Washes	40.7	5.01	31.0	1.35	13.3	0.62	12.9	0.17	5.0	0.08	0.5	0.02
Extracts	44.5	4.47	46.1	2.01	70.1	3.25	42.8	0.56	25.6	0.41	46.0	1.59
PES*	14.8	1.82	22.8	1.00	16.6	0.77	44.2	0.58	69.3	1.11	53.5	1.85
Total	100	12.30	100	4.37	100	4.63	100	1.32	100	1.60	100	3.46
	Tuber											
Extracts	89.9	0.27	na	na	88.2	0.22	85.3	0.16	81.6	0.19	81.8	0.20
PES*	10.1	0.03	na	na	11.8	0.03	14.7	0.03	18.4	0.04	18.2	0.04
Total	100	0.30	100	0.25	100	0.25	100	0.19	100	0.23	100	0.24

Day –14 (after 1st application): Foliage surface washes: 66.0 % (4.7 mg/kg); extracts: 26.6 % (1.89 mg/kg); residues: 7.5 % (0.53 mg/kg); Total: 100 % (7.12 mg/kg)

na: not analysed

* Unextractable residues: not extractable with solvents of increasing polarity (acetonitrile, acetonitrile:water (1:1) and water)

Table 11 Residue components in potato foliage following 2 × 0.34 kg ai/ha foliar applications

Component	Day 0		Day 2		Day 7		Day 14	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dimethoate	68.1	8.38	54.8	2.39	40.9	1.89	15.0	0.20
Omethoate	5.9	0.73	7.3	0.32	15.6	0.72	9.3	0.12
O-Desmethyl omethoate carboxylic acid	< 0.2	< 0.02	< 0.2	< 0.01	< 0.2	< 0.01	1.5	0.02
O-Desmethyl N-desmethyl omethoate	1.8	0.22	4.2	0.18	5.2	0.24	8.7	0.11
O-Desmethyl omethoate	3.2	0.39	0.7	0.03	4.6	0.21	3.2	0.04
O-Desmethyl isodimethoate/ dimethoate carboxylic acid	0.8	0.10	< 0.2	< 0.01	3.7	0.17	3.1	0.04
O,O-Dimethyl dithio-phosphoric acid	0.3	0.04	< 0.2	< 0.01	< 0.2	< 0.01	4.5	0.06
O-Desmethyl dimethoate	< 0.2	< 0.08	< 0.2	< 0.01	< 0.2	< 0.01	1.2	0.02
Component A [a]	0.8	0.10	< 0.2	< 0.01	< 0.2	< 0.01	0.5	0.01
Glucose conjugate of hydroxy dimethoate (Component G) [b]	2.3	0.28	4.3	0.19	7.7	0.36	4.8	0.06
Component K [c]	0.8	0.10	1.1	0.05	1.5	0.07	3.2	0.04
Other	1.2	0.15	4.7	0.21	4.2	0.19	0.8	0.01
Total extracts	85.2	10.48	77.1	3.37	83.4	3.86	55.7	0.74
Water extractable	na	na	na	na	3.1	0.14	5.7	0.08

Component	Day 0		Day 2		Day 7		Day 14	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Protease extractable	4.3	0.53	9.4	0.41	5.5	0.25	16.8	0.22
Organo soluble	na	na	na	na	na	na	0.8	0.01
Water soluble	na	na	na	na	na	na	16.0	0.21
Base extractable	na	na	na	na	na	na	14.9	0.20
Unextractable Residues	10.5	1.29	13.4	0.54	8.0	0.37	6.9	0.09
Total	100	12.30	100	4.37	100	4.63	100	1.32

Results expressed as % sample radioactivity and mg equivalent/kg tissue fresh weight

na: not analysed

[a] Could not be conclusively identified in the potato study due to low concenctration. In the wheat study, a component eluting at a similar time was identified as O-desmethyl N-desmethyl omethoate which was trapped by high molecular weight material at or near the point of application on TLC plates.

[b] Glucose conjugate of hydroxy dimethoate

[c] shown to be composed of up to six minor components all < 0.05 mg/kg

Table 12 Residue components in potato tubers following 2 × 0.34 kg ai/ha foliar applications

Component	Day 0		Day 7		Day 14		Day 21		Day 28	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dimethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Omethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-Desmethyl omethoate carboxylic acid	6.1	0.02	9.7	0.02	18.4	0.03	11.1	0.03	12.1	0.03
O-Desmethyl N-desmethyl omethoate	76.4	0.23	46.0	0.12	45.5	0.09	40.3	0.09	43.6	0.10
O-Desmethyl omethoate	< 0.1	< 0.01	28.1	0.07	12.5	0.02	17.7	0.04	14.8	0.04
O-Desmethyl isodimethoate/ Dimethoate carboxylic acid	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O,O-Dimethyl dithio-phosphoric acid	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
O-Desmethyl dimethoate	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Component A [a]	7.4	0.02	2.0	0.01	5.5	0.01	3.2	0.01	3.1	0.01
Glucose conjugate of hydroxy dimethoate (Component G) [b]	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Component K [c]	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01
Other	< 0.1	< 0.01	2.4	0.01	3.4	0.01	6.7	0.02	5.3	0.01
Total extracts	89.9	0.27	88.2	0.22	85.3	0.16	79.0	0.18	78.9	0.19
Water extractable	na	na	0.5	< 0.01	0.3	< 0.01	4.5	0.01	4.7	0.01
Protease extractable	1.8	0.01	1.1	< 0.01	1.4	< 0.01	0.7	< 0.01	0.8	< 0.01
Organo soluble	na	na	na	na	0.4	< 0.01	na	na	na	na
Water soluble	na	na	na	na	1.0	< 0.01	na	na	na	na
Base extractable	na	na	Na	na	3.6	0.01	4.0	0.01	4.0	0.01
Unextractable Residues	8.3	0.02	10.2	0.03	9.4	0.02	11.8	0.03	11.6	0.03
Total	100	0.30	100	0.25	100	0.19	100	0.23	100	0.24

Results expressed as % sample radioactivity and mg equivalent/kg tissue fresh weight

na: not analysed

[a] Could not be conclusively identified in the potato study due to low concenctration. In the wheat study, a component eluting at a similar time was identified as O-desmethyl N-desmethyl omethoate which was trapped by high molecular weight material at or near the point of application on TLC plates.

[b] Glucose conjugate of hydroxy dimethoate

[c] shown to be composed of up to six minor components all < 0.05 mg/kg

Total radioactive residues in foliage were 12.30 mg eq/kg at day 0 (after the second application), declining to 1.3 mg eq/kg by day 14, with a subsequent increase to 3.5 mg eq/kg day 28 due to drying of the foliage. Tuber residues were lower at 0.30 mg eq/kg at day 0, declining only slightly to 0.19 mg eq/kg at day 14 and 0.24 mg eq/kg at day 28.

Solvent extractability (via surface washes with acetonitrile) for foliage and acetonitrile, acetonitrile/water and water for both tubers and foliage) was 85% in foliage at day 0, decreasing to 56% at day 14 and 46% at day 28, and 90% in tubers at day 0, remaining high at 81% and 83% at days 14 and 28 respectively. Further amounts were extracted from foliage and tubers by acid, base and protease treatment (carried out in parallel on subsamples of the PES). Extractions from foliage by protease released 4.3% at day 0, and 17% at day 14, acid released 3.5% at day 0, and 8.5% at day 14 and base released 4.0% at day 0, and 14% at day 14, while from tubers protease released 1.8% at day 0, 1.4% at day 14, and 0.8% at day 28, acid released 0.5% at day 0, 1.6% at day 14, and 0.7% at day 28 and base released 1.7% at day 0, 3.3% at day 14, and 2.7% at day 28.

In foliage, dimethoate and omethoate accounted for 68% and 6% of the TRR respectively (8.4 mg and 0.73 mg eq/kg) on day 0. By day 14, dimethoate and omethoate accounted for 15% and 9.3% of the TRR in foliage respectively (0.2 and 0.12 mg eq/kg). The largest residue components in foliage were O-desmethyl N-desmethyl omethoate (1.8% TRR, 0.22 mg eq/kg at day 0 and 8.7% TRR, 0.11 mg eq/kg at day 14), followed by O,O,-dimethyl dithiophosphoric acid (0.3% TRR, 0.04 mg eq/kg at day 0 and 3.5% TRR, 0.06 mg eq/kg at day 14), O-desmethyl omethoate (3.2% TRR, 0.39 mg eq/kg at day 0 and 3.2% TRR, 0.04 mg eq/kg at day 14), co-eluting O-desmethyl isodimethoate and dimethoate carboxylic acid (0.8% TRR, 0.10 mg eq/kg at day 0 and 3.1% TRR, 0.04 mg eq/kg at day 14), hydroxy dimethoate glucose conjugate (2.3% TRR, 0.28 mg eq/kg at day 0 and 4.6% TRR, 0.06 mg eq/kg at day 14), desmethyl dimethoate (1.2% TRR, 0.02 mg eq/kg at day 14), and O-desmethyl omethoate carboxylic acid (1.5% TRR, 0.02 mg eq/kg at day 14).

Dimethoate and omethoate were not detected in the tubers. The components identified in potato tubers were O-desmethyl N-desmethyl omethoate (0.23 mg eq/kg, 76% TRR at day 0, 0.09 mg eq/kg, 46% TRR at day 14, and 0.10 mg eq/kgb 44% TRR at day 28), O-desmethyl omethoate, (< 0.01 mg eq/kg at day 0, 0.02 mg eq/kg, 12% TRR at day 14 and 0.04 mg eq/kg, 15% TRR at day 28), and O-desmethyl omethoate carboxylic acid (0.02 mg eq/kg, 6.1% TRR at day 0, 0.03 mg eq/kg, 18% TRR at day 14), and 0.03 mg eq/kg, 12% TRR at day 28). .

Neither dimethoate nor omethoate are translocated from the foliage to the tubers and metabolism occurs mainly in the foliage.

The major metabolic reactions observed were:

1) Oxidation to yield omethoate.
2) O-demethylation and N-demethylation of omethoate to yield O-desmethyl N-desmethyl omethoate.
3) Hydrolysis of the amide bond to give dimethoate carboxylic acid and subsequent degradation to give dimethyl dithiophosphate.
4) Demethylation to yield desmethyl dimethoate or des-O-methyl isodimethoate.
5) Demethylation of omethoate to give O-desmethyl omethoate and subsequent hydrolysis of the amide bond to give O-desmethyl omethoate carboxylic acid.
6) Hydroxylation of the N-methyl group (hydroxyl-dimethoate) and subsequent conjugation with glucose.

Based on these results the following metabolic pathway (Figure 3) was proposed for potatoes.

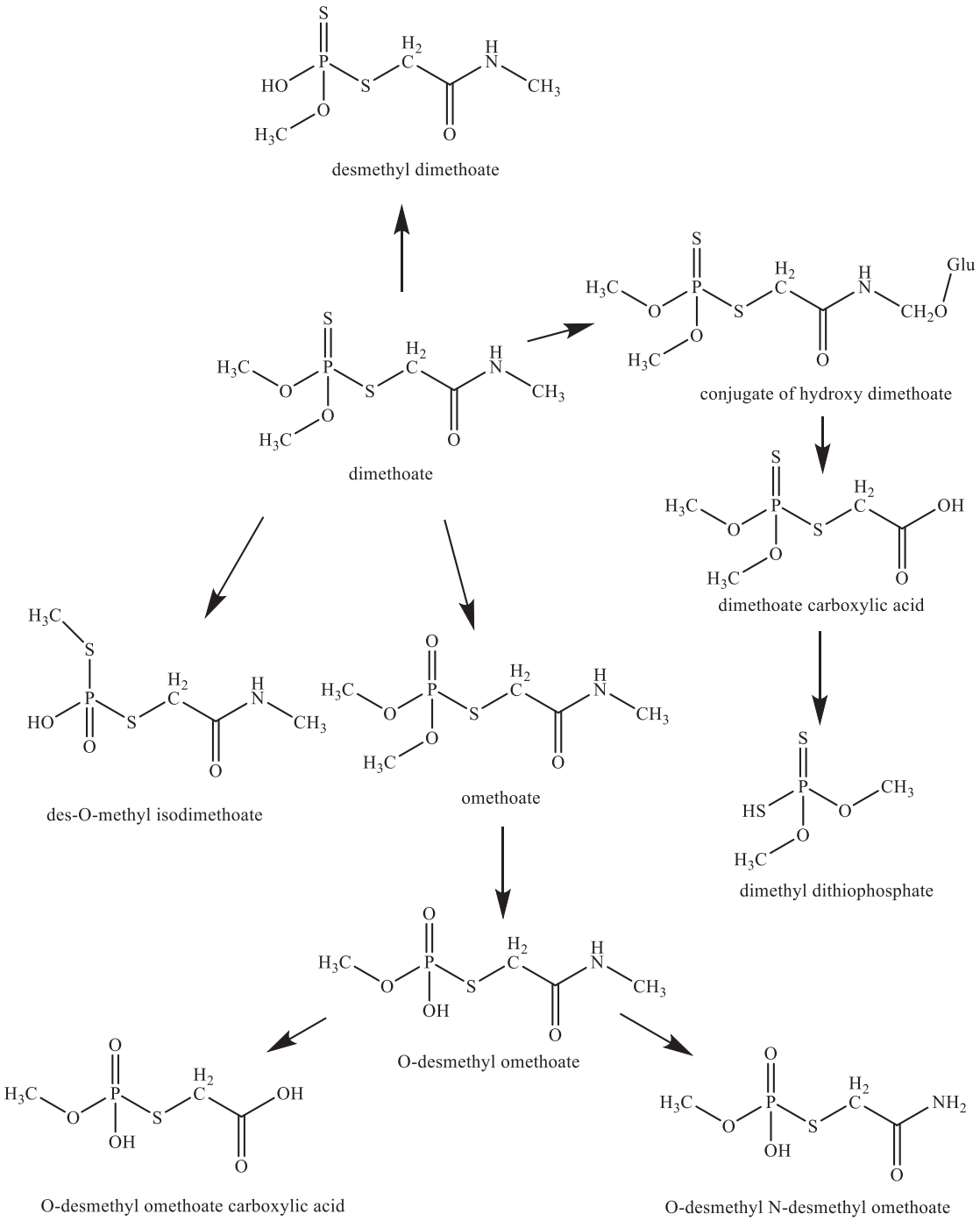


Figure 3 Metabolic pathway for dimethoate in potatoes

Wheat

Dimethoate labelled with ^{14}C in both methoxy groups was mixed with an EC formulation containing non-labelled material (Corden – 2001b). The mixture was applied to wheat as a foliar spray at 0.68 kg ai/ha at BBCH 24 followed by 0.4 kg ai/ha at BBCH 69. The experiment was also performed using an exaggerated application rate (5×, 3.4 and 2.0 kg ai/ha), with applications being made at the same

growth stages. The wheat plants were grown to maturity in individual containers located outdoors. Samples were collected after the first application (day 0) and after 14, 26 and 39 days. Samples were also taken after the second application (day 41), and after 62 (early harvest) and 73 days (normal harvest). For the exaggerated rate trial, only grain, hull and straw were collected at 73 days. Depending on the growth stage of the plant, samples consisted of whole plant, ear, remaining plant, grain, hull or straw. Samples were extracted with acetonitrile/water. Levels of radioactivity in extracts were determined by LSC and the levels in the post-extraction solids (PES) were determined by combustion followed by LSC. Extracts containing significant radioactivity were analysed by HPLC and TLC with comparison against reference substances.

Residues in PES were further characterised by treating with dilute acid (0.1 M HCl), dilute base (0.1 M or 1 M NaOH), strong base (6 M NaOH) or enzymes (protease, cellulose/hemicellulose, and amylase).

Residues were highest in plant material immediately after application, with TRRs of 30 mg eq/kg in whole plant at day 0 after one application at 0.68 kg ai/ha, declining to 1.7 mg eq/kg at day 14 and 0.90 mg eq/kg in remaining plant at day 39 (before the second application). Immediately after the second application (0.68 + 0.40 kg ai/ha), TRRs were 23 mg eq/kg in ears and 16 mg eq/kg in remaining plant. At harvest maturity, TRRs were 2.3 mg eq/kg at 21 days after application 2 and 4.3 mg eq/kg at 32 days in grain, 23 and 34 mg eq/kg at days 21 and 32 in hulls, and 6.5 and 7.8 mg eq/kg at days 21 and 32 in straw.

Only grain at 32 days after the second application was analysed from the exaggerated rate trial, with a TRR of 20 mg eq/kg. 63% of TRR was extractable with acetonitrile/water.

Solvent extractability was generally high in plant material, at 99.8% of TRR at day 0 after the first (0.68 kg ai/ha application, declining to 91% of TRR in ears and 78% of the TRR in remaining plant at 39 days after the first application. At harvest, extractability was 81% and 66% from grain of TRR at 21 and 32 days after 0.68 + 0.40 kg ai/ha applications, 92% and 84% from hulls respectively and 79% and 72% from straw respectively.

In whole wheat plants immediately after the first application at 0.68 kg ai/ha, the residues was mainly unmetabolized dimethoate at 98% TRR (29 mg eq/kg), with small amounts of omethoate (0.7% TRR, 0.21 mg eq/kg), and O-desmethyl N-desmethyl omethoate (0.4% TRR, 0.12 mg eq/kg). Dimethoate was rapidly metabolized in wheat. At 14 days after the first application, the residue components were O-desmethyl isodimethoate (30% TRR, 0.49 mg eq/kg), O-desmethyl N-desmethyl omethoate (26% TRR, 0.44 mg eq/kg), omethoate (7.8% TRR, 0.13 mg eq/kg), dimethoate (4.1% TRR, 0.07 mg eq/kg), O,O-dimethyl dithiophosphate (4.5% TRR, 0.08 mg eq/kg), and O-desmethyl omethoate carboxylic acid (1.1% TRR, 0.02 mg eq/kg). At 39 days, residue components in plant material after removal of ears were O-desmethyl N-desmethyl omethoate (42% TRR, 0.37 mg eq/kg), O-desmethyl isodimethoate (22% TRR, 0.20 mg eq/kg), O-desmethyl omethoate carboxylic acid (5.7% TRR, 0.05 mg eq/kg), and O,O-dimethyl dithiophosphate (2.6% TRR, 0.02 mg eq/kg) – no dimethoate or omethoate were found.

At harvest, no residues of dimethoate or omethoate were found in grain. The residue components in grain sampled at 21 days after the second application (0.68 + 0.40 kg ai/ha) were O-desmethyl N-desmethyl omethoate (54% TRR, 1.2 mg eq/kg), O-desmethyl isodimethoate (11% TRR, 0.26 mg eq/kg), and O-desmethyl omethoate carboxylic acid (3.8% TRR, 0.09 mg eq/kg). At 32 days, the same components were identified. IN straw at 21 days, residue components were O-desmethyl N-desmethyl omethoate (36% TRR, 2.3 mg eq/kg), O-desmethyl isodimethoate (20% TRR, 1.3 mg eq/kg), dimethoate (6.2% TRR, 0.40 mg eq/kg), O-desmethyl omethoate carboxylic acid (4.6% TRR, 0.30 mg eq/kg), omethoate (3.4% TRR, 0.22 mg eq/kg), and O,O-dimethyl dithiophosphoric acid (2.8% TRR, 0.18 mg eq/kg). Levels in straw were qualitatively similar at 32 days after the second application.

In the exaggerated rate grain samples, residue components were O-desmethyl N-desmethyl omethoate (39% TRR, 8.0 mg eq/kg), O-desmethyl isodimethoate (16% TRR, 3.1 mg eq/kg), O-

desmethyl omethoate carboxylic acid (6.4% TRR, 1.3 mg eq/kg), dimethoate (0.5% TRR, 0.10 mg eq/kg), and omethoate (0.3% TRR, 0.06 mg eq/kg).

Of the additional extraction procedures conducted on the PES, base extraction generally removed the largest proportion of radioactivity. A number of minor components were characterised by TLC or HPLC in the base extract, but none could be identified.

Table 13 Total residues and extractability of dimethoate residues from wheat matrices (whole green plants i.e. forage and ears and remaining plant) following a single application at 0.68 kg ai/ha

Fraction	Day 0		Day 14		Day 26				Day 39 (before 2nd application)			
	Whole plant		Whole plant		Ear		Remaining plant		Ear		Remaining plant	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracts	99.8	29.7	82.9	1.38	86.5	0.19	84.1	1.03	91.4	0.39	78.3	0.70
Residue	0.2	0.06	17.1	0.29	13.5	0.03	15.9	0.20	8.6	0.04	21.7	0.20
Total	100	29.7	100	1.67	100	0.22	100	1.23	100	0.43	100	0.90

Table 14 Total residues and extractability of dimethoate residues from wheat (ear and remaining plant and grain, hull and straw at harvest) matrices following two applications at 0.68 and 0.4 kg ai/ha

Fraction	Day 41 (0 day after 2nd application)				Day 62 (21 days after 2nd application)						Day 73 (32 days after 2nd application)					
	Ear		Remaining plant		Grain		Hull		Straw		Grain		Hull		Straw	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracts	99.3	22.6	97.4	15.7	80.7	1.85	91.5	21.3	78.6	5.05	65.7	2.81	84.5	28.5	72.1	5.65
Residue	0.7	0.16	2.6	0.42	19.3	0.44	8.5	1.98	21.4	1.37	34.3	1.47	15.5	5.22	27.9	2.18
Total	100	22.7	100	16.1	100	2.29	100	23.3	100	6.42	100	4.28	100	33.7	100	7.83

Table 15 Total residues and extractability of dimethoate residues from wheat grain following two applications at 3.4 and 2.0 kg ai/ha

Fraction	Day 73 (32 days after 2nd application)	
	% TRR	mg eq/kg
Extracts	62.7	12.6
Residue	37.3	7.52
Total	100	20.2

Table 16 Residues of dimethoate in wheat whole plant and hay/ear samples after one (0.68 kg ai/ha) or two (0.68 + 0.40 kg ai/ha) foliar applications

Component	Day 0		Day 14		Day 26 (after ear emergence)				Day 39 (before 2nd application)				Day 41 (after 2nd application)			
	Whole plant		Whole plant		Ear		Remaining plant		Ear		Remaining plant		Ear		Remaining plant	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg

Component	Day 0		Day 14		Day 26 (after ear emergence)				Day 39 (before 2nd application)				Day 41 (after 2nd application)			
	Whole plant		Whole plant		Ear		Remaining plant		Ear		Remaining plant		Ear		Remaining plant	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dimethoate	97.5	29.0	4.1	0.07	< 0.1	< 0.01	1.4	0.02	< 0.1	< 0.01	< 0.1	< 0.01	92.2	21.0	83.2	13.4
Omethoate	0.7	0.21	7.8	0.13	< 0.1	< 0.01	2.8	0.03	< 0.1	< 0.01	< 0.1	< 0.01	1.9	0.43	2.6	0.42
O-Desmethyl omethoate carboxylic acid	< 0.1	< 0.03	1.1	0.02	6.4	0.01	3.8	0.05	1.4	0.01	5.7	0.05	< 0.1	< 0.02	< 0.1	< 0.02
O-Desmethyl N-desmethyl omethoate [a]	0.4	0.12	26.5	0.44	80.1	0.18	48.3	0.59	68.7	0.30	41.8	0.37	1.7	0.39	4.4	0.71
D	< 0.1	< 0.03	1.7	0.03	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.02	< 0.1	< 0.02
O-Desmethyl isodimethoate	< 0.1	< 0.03	29.6	0.49	< 0.1	< 0.01	22.4	0.28	17.1	0.07	22.3	0.20	1.4	0.32	3.7	0.60
Dimethyl dithiophosphoric acid	< 0.1	< 0.03	4.5	0.08	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	2.6	0.02	0.5	0.11	2.0	0.32
G [b]	< 0.1	< 0.03	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.02	< 0.1	< 0.02
H	< 0.1	< 0.03	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.02	< 0.1	< 0.02
K [c]	0.4	0.12	1.4	0.02	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.01	0.3	0.07	0.7	0.11
Other	0.8	0.24	6.2	0.10	< 0.1	< 0.01	5.4	0.07	4.2	0.39	5.9	0.05	1.3	0.30	0.8	0.13
Total extractables	99.8	29.7	82.9	1.38	86.5	0.19	84.1	1.03	91.4	0.39	78.3	0.70	99.3	22.6	97.4	15.7
Base extractable	na	na	14.1	0.24	7.6	0.02	11.0	0.14	na	na	17.0	0.15	na	na	na	na
Unextractable residues	0.2	0.06	3.0	0.05	5.9	0.01	4.9	0.06	8.6	0.04	4.7	0.04	0.7	0.16	2.6	0.42
Total	100	29.7	100	1.67	100	0.22	100	1.23	100	0.43	100	0.90	100	22.7	100	16.1

[a] includes component A, which was radioactivity trapped by high molecular weight material at or near the point of application on TLC plates and later identified as O-Desmethyl N-desmethyl omethoate

[b] identified as glucose conjugate of hydroxy dimethoate in the potato metabolism study

[c] shown to be composed of up to six minor, non-polar components in the potato metabolism study

na: not analysed

Table 17 Residues of dimethoate in wheat grain, hull and straw samples after two (0.68 + 0.40 kg ai/ha) foliar applications

Component	Day 62 (early harvest) 21 days after second application						Day 73 (normal harvest) 32 days after second application					
	Grain		Hull		Straw		Grain		Hull		Straw	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dimethoate	< 0.1	< 0.01	5.2	1.21	6.2	0.40	< 0.1	< 0.01	3.0	1.01	3.4	0.27
Omethoate	< 0.1	< 0.01	10.2	2.37	3.4	0.22	< 0.1	< 0.01	5.5	1.85	3.6	0.28
O-Desmethyl omethoate carboxylic acid	3.8	0.09	< 0.1	< 0.02	4.6	0.30	3.6	0.15	3.0	1.01	3.6	0.28
O-Desmethyl N-desmethyl omethoate [a]	54.5	1.25	32.3	7.51	35.7	2.29	46.5	1.99	52.4	17.7	47.3	3.70

Component	Day 62 (early harvest) 21 days after second application						Day 73 (normal harvest) 32 days after second application					
	Grain		Hull		Straw		Grain		Hull		Straw	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
D	< 0.1	< 0.01	< 0.1	< 0.02	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.03	< 0.1	< 0.01
O-Desmethyl isodimethoate	11.3	0.26	31.0	7.21	20.1	1.29	6.8	0.29	8.9	3.00	3.6	0.28
Dimethyl dithiophosphoric acid	< 0.1	< 0.01	3.3	0.77	2.8	0.18	< 0.1	< 0.01	2.1	0.71	2.0	0.16
G [b]	< 0.1	< 0.01	< 0.1	< 0.02	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.03	< 0.1	< 0.01
H	< 0.1	< 0.01	< 0.1	< 0.02	< 0.1	< 0.01	< 0.1	< 0.01	< 0.1	< 0.03	< 0.1	< 0.01
K [c]	3.7	0.08	2.5	0.58	1.3	0.08	1.0	0.04	< 0.1	< 0.03	< 0.1	< 0.01
Other	7.4	0.17	7.0	1.63	4.5	0.29	7.8	0.33	9.6	3.23	8.6	0.67
Total extractables	80.7	1.85	91.5	21.3	78.6	5.05	65.7	2.81	84.5	28.5	72.1	5.65
Base extractable	11.1	0.25	4.7	1.09	15.9	1.02	34.4	1.47	10.7	3.60	27.8	2.18
Organosoluble	2.1	0.05	na	na	na	na	na	na	na	na	na	na
Aqueous soluble	9.0	0.21	na	na	na	na	na	na	na	na	na	na
Unextractable residues	8.2	0.19	3.8	0.88	5.5	0.35	< 0.1	< 0.01	4.8	1.62	< 0.1	< 0.01
Total	100	2.29	100	23.3	100	6.42	100	4.28	100	33.7	100	7.83

[a] includes component A, which was radioactivity trapped by high molecular weight material at or near the point of application on TLC plates and later identified as O-Desmethyl N-desmethyl omethoate

[b] identified as glucose conjugate of hydroxy dimethoate in the potato metabolism study

[c] shown to be composed of up to six minor, non-polar components in the potato metabolism study

na: not analysed

Table 18 Residues of dimethoate in wheat grain samples after two (3.4 + 2.0 kg ai/ha) foliar applications

Component	Day 73 (later harvest) 32 days after second application	
	% TRR	mg eq/kg
Dimethoate	0.5	0.10
Omethoate	0.3	0.06
O-Desmethyl omethoate carboxylic acid	6.4	1.29
O-Desmethyl N-desmethyl omethoate [a]	39.4	7.95
D	< 0.1	< 0.02
O-Desmethyl isodimethoate	15.6	3.14
Dimethyl dithiophosphoric acid	< 0.1	< 0.02
G [b]	< 0.1	< 0.02
H	< 0.1	< 0.02
K [c]	< 0.1	< 0.02
Other	0.5	0.10
Total extractables	62.7	12.6
Unextractable residues	37.3	7.52
Total	100	20.2

[a] includes component A, which was radioactivity trapped by high molecular weight material at or near the point of application on TLC plates and later identified as O-Desmethyl N-desmethyl omethoate

[b] identified as glucose conjugate of hydroxy dimethoate in the potato metabolism study

[c] shown to be composed of up to six minor, non-polar components in the potato metabolism study

The major metabolic reactions observed were:

1) Oxidation to omethoate.
2) O-Demethylation and N-demethylation of omethoate to yield O-desmethyl N-desmethyl omethoate.
3) O-Demethylation and rearrangement to yield des-O-methyl isodimethoate.
4) Hydrolysis of the amide bond and subsequent degradation to give dimethyl dithiophosphate.
5) Demethylation of omethoate and hydrolysis of the amide bond to give O-desmethyl omethoate carboxylic acid.

The proposed metabolic pathway for dimethoate in wheat is outlined below (Figure 4). Intermediates in brackets were not detected in the wheat study, but have been proposed based on the potato metabolism study.

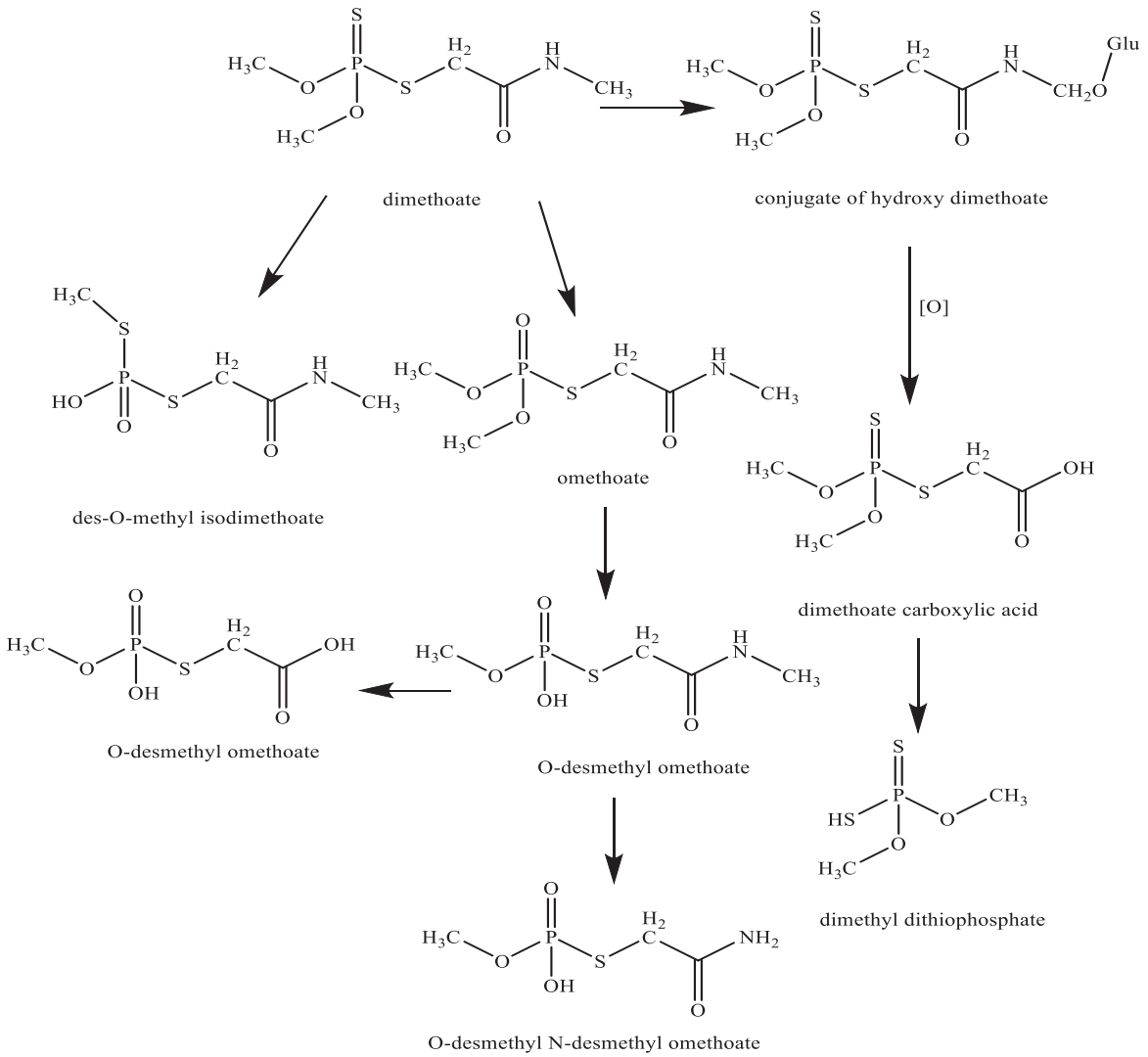


Figure 4 Proposed metabolic pathway for dimethoate in wheat

Other plant metabolism studies

The metabolism of [^{32}P]dimethoate in lemons, sugar beet, maize, cotton, peas, potatoes and beans was reported (Heidemann – 1996). The reports were summaries, which did not provide the level of detail given in a contemporary metabolism study. Generally, the main components of the radiolabelled residue were dimethoate, omethoate, dimethoate carboxylic acid, dimethyl hydrogen phosphate and *O,O*-dimethyl hydrogen phosphorodithioate, indicating oxidation to omethoate, omethoate carboxylic acid and dimethoate carboxylic acid, and cleavage of the P-S linkage either before or after oxidation. The metabolic pathways in these studies were similar to the olive, potato and wheat study.

In the bean study (Lucier and Menzer-1968), after foliar treatment with [^{14}C-methoxy]-dimethoate, parent compound comprised 40% of the foliage TRR, omethoate 1.6% TRR, while N-desmethyl dimethoate, dimethoate carboxylic acid, and O-desmethyl dimethoate carboxylic acid all comprised less than 1% of TRR.

In a study of the metabolism of [^{32}P]-dimethoate in excised cotton leaves (Hacskaylo and Bull-1963) with the cut petioles immersed in an aqueous solution of the radiolabel, dimethoate comprised 70% of the TRR on day 1, declining to 1.9% on day 14. The components identified were dimethoate carboxylic acid (15–50%), O,O-dimethyl dithiophosphoric acid (4–11%), omethoate (~6%), dimethyl phosphate (2.5–11%), O,O-dimethyl thiophosphoric acid (1.6–12%), O-desmethyl dimethoate carboxylic acid (no fraction given), and phosphoric acid (no fraction given).

After trunk application of [^{32}P]-dimethoate to lemon trees (Santi-1961), residue components identified in fruit were dimethoate, omethoate, dimethyl phosphoric acid, phosphoric acid, O,O-dimethyl thiophosphoric acid, and desmethyl dimethoate.

ENVIRONMENTAL FATE

Hydrolysis

Dimethoate radiolabelled with carbon-14 in the O-methyl positions was diluted in sterilised buffer solutions at pH 5, 7 and 9 at 200 mg/L (Kirkpatrick – 1986a). The flasks were stoppered and incubated in the dark at 25 ± 1 °C. Duplicate flasks of each buffer solution were analysed at intervals from 0 to 30 days after fortification with dimethoate and radioassayed by LSC. Additional flasks were deep frozen for later analysis by radio-TLC with co-chromatography and GC-MS for identification of hydrolysis products.

Hydrolysis was rapid at pH 9, with a half-life of 4.4 days, and significantly slower under neutral and acid conditions, with half-lives of 68 and 156 days at pH 7 and 5 respectively. The major hydrolysis products at pH 9 were *O*-desmethyldimethoate and *O,O*-dimethylphosphorothioic acid, comprising 62.1% and 36.0% of the radioactivity at day 30, with parent accounting for only 1.1%. In pH 7 buffer, parent, *O*-desmethyldimethoate and *O,O*-dimethylphosphorothioic acid comprised 74.4%, 22.1% and 1.9% of the radioactivity at day 30, while at pH 5, parent comprised 87.8% and *O*-desmethyldimethoate 12.2%, with no other components identified.

Aqueous photolysis

A 10 mg/L solution of ^{14}C-methyl-dimethoate in pH 5 acetate buffer was prepared and incubated at 25 ± 1 °C for 15 days (Kirkpatrick – 1986b). The solution was irradiated using a xenon arc lamp continuously for 15 days, equivalent to at least 30 days of natural sunlight in equatorial regions. A dark control was run alongside. Only limited degradation had occurred at the end of the experiment, with 94.3% and 96.9% of the radioactivity associated with parent compound for the irradiated and control samples (half-life > 175 days under the conditions of the experiment). No degradation products were identified.

Soil surface photolysis

A study of the photolysis of ^{14}C-methyl-dimethoate at the soil surface was also carried out (Skinner and Shepler-1994). The radiolabel was applied to sieved sandy loam soil spread in a thin layer on a Petri dish at a nominal rate of 2 lb ai/acre (2.24 kg ai/ha). The sample was incubated at 25 °C for 30 days and irradiated with natural late summer (25 August to 24 September) sunlight at 37.4° N (Richmond, California). A dark control sample was also set up. The incubation chambers were equipped with traps for $^{14}CO_2$ and other volatiles. Soil samples were collected for analysis at intervals of 0, 2, 5, 10, 20 and 30 days. Soil was extracted three times with 1:1 v/v acetonitrile/water. The day 30 samples were further extracted with 0.5 M NaOH to yield any radioactivity incorporated into fulvic and humic acid fractions. Total radioactivity was measured by LSC, while degradates were analysed by TLC including co-chromatography with reference compounds, radio-HPLC, and GC-MS was used for identification. Unextracted radioactivity was determined by combustion and LSC.

Recovery of radioactivity was good, at an average of 100.1% and 100.5% for the irradiated and dark control samples respectively. There was no significant difference in the degradation rate for the light and dark samples, with measured half lives of 10.5 and 7.9 days respectively (SFO model). After 30 days, 13.9% of the applied radioactivity remained as parent compound, with the most significant components being dimethylphosphoric acid and dimethylthiophosphoric acid at 27.9% and 25.4% of the AR respectively. Mineralisation to carbon dioxide was only a minor component of the final residue, while bound residues comprised 12.2% of the AR, of which slightly more than half was found in the fulvic acid fraction at 7.1% of AR. The breakdown products were qualitatively similar for the dark control and irradiated samples, indicating no specific photolytic breakdown pathway in soil.

Table 19 Photolysis of ^{14}C-dimethoate on the soil surface – after 30 days incubation

Component	Irradiated soil-%AR	Dark control sample-%AR
Parent	13.9	6.7
Dimethylphosphoric acid	27.9	12.9
Dimethylthiophosphoric acid	25.4	55.7
Degradate 3	6.6	5.5
Other minor components (sum)	5.3	3.3
$^{14}CO_2$	1.9	1.6
Other volatiles	4.6	5.3
Total bound soil residues	12.2	7.9
fulvic acid fraction	7.1	Not determined
humic acid fraction	1.0	Not determined
others	4.1	Not determined
Sum	97.8	98.9

Aerobic soil degradation

An aerobic degradation study of dimethoate labelled with carbon-14 at the carbonyl group was conducted using a sandy loam soil (Hein and Gourlay-2015). Pre-incubated (at 20 ± 2 °C) soil samples were fortified with ^{14}C-dimethoate at a target rate of 0.533 mg/kg (dry weight), corresponding to 400 g ai/ha, assuming a soil density of 1.5 g/cm^3 and even distribution in the top 5 cm layer. All vessels were equipped with traps for carbon dioxide and other volatiles and incubated at 20 ± 2 °C in the dark. The soil was maintained at an approximate water content of 40% of the water holding capacity. Samples were collected at intervals from day 0 to 21. Soil samples were extracted twice with acetonitrile and once with 1:1 v/v acetonitrile/water, with samples from day 1 onwards

additionally being extracted using an acetonitrile/water reflux. Combined extracts were assayed by LSC and HPLC. Residual radioactivity was determined by combustion and LSC.

Recovery of radioactivity was good, at a mean of 96.6% of applied radioactivity. Degradation was rapid, with parent being less than 1% of applied radioactivity at day 21. Mineralisation was the major degradation pathway, with $^{14}CO_2$ being measured at 53.3% of applied radioactivity at day 21, with significant fractions of the applied radioactivity being found as fluvic acids, humins, and humic acids (24.8%, 13.3%, and 4.5% respectively). Levels of other degradation products were low, with desmethyl-dimethoate being found at <5%, and omethoate not being detected at all. The DT50 and DT90 were calculated at 2.6 and 8.6 days respectively by the SFO model.

The dissipation of unlabelled dimethoate in three different soils (sandy loam, clay loam and silty clay) was investigated in samples incubated at 20 ± 3 °C under laboratory conditions (Burden – 1991).

Soil samples (50 g dry weight) were fortified at 1.2 mg/kg (dry weight basis) with dimethoate in acetone. The samples were incubated in the dark for up to 16 days at 20 ± 3 °C. Water content was maintained at 40% of maximum water holding capacity (MWHC), with additions of distilled water every 2–3 days made as necessary. Untreated control samples were also incubated to allow determination of concurrent recoveries. Duplicated samples were withdrawn at intervals for analysis by GC-NPD after overnight Soxhlet extraction with acidified 4:1 v/v acetone/hexane. Mean recoveries at initial validation were 93%, 94% and 89% for the clay loam, silty clay and sandy loam respectively, while the respective mean concurrent recoveries were 85%, 85%, and 81%.

Table 20 Dissipation of dimethoate from three different soils incubated in the laboratory

Time interval (days)	Clay loam (Riverside, North Yorkshire, UK)		Silty clay (Middlefield, North Yorkshire, UK)		Sandy loam (Somersham, Cambridgeshire, UK)	
	Conc. in mg/kg (mean)	% initial	Conc. in mg/kg (mean)	% initial	Conc. in mg/kg (mean)	% initial
0 (1 hour)	1.14, 0.90 (1.02)	100	1.60, 1.46 (1.53)	100	0.90, 0.99 (0.95)	100
1	0.93, 0.94 (0.94)	91	1.00, 0.99 (1.00)	65	0.70, 0.84 (0.77)	82
2	0.57, 0.68 (0.62)	61	0.64, 0.83 (0.74)	48	0.73, 0.76 (0.75)	79
4	0.40, 0.44 (0.42)	41	0.66, 0.91 (0.79)	52	0.49, 0.56 (0.52)	55
5	0.13, 0.13 (0.13)	13	0.19, 0.32 (0.25)	17	0.41, 0.41 (0.41)	44
7	0.11, 0.11 (0.11)	10	0.062, 0.078 (0.070)	5	0.32, 0.53 (0.43)	45
8	0.064, 0.089 (0.077)	8	0.069, 0.093 (0.081)	5	0.25, 0.21 (0.23)	25
10	0.065 (0.065)	6	0.089, 0.037 (0.063)	4	0.051, 0.078 (0.065)	7
12	0.040, 0.041 (0.041)	4	0.036, 0.037 (0.036)	2	0.097, 0.094 (0.096)	10
16	-	-	-	-	0.10, 0.11 (0.10)	11

Exponential decline curves (simple first order model) gave good fits for the data. The decline was relatively rapid, with half-lives (DT50 values) of 2.4, 2.0, and 4.1 days for the clay loam, silty clay and sandy loam respectively, while the DT90 values were 8.0, 6.8, and 13.5 days respectively.

Field dissipation studies

Dimethoate

The dissipation of dimethoate from soil in the field after applications to sorghum in Texas was investigated (Jacobson and Willams – 1994a). Dimethoate was applied on 8 July in a single

application as a 400 g/L emulsifiable concentrate (EC) formulation using a tractor-mounted boom sprayer to a plot of sorghum (2-leaf to 4-leaf stage) grown in a silt loam soil in Snook, TX, at a rate of 1.5 lb ai/acre (1.7 kg ai/ha). Soil cores were collected to a depth of 48 inches (122 cm) from the treated (15 cores per sampling event) and an adjacent untreated control plot (5 cores per sampling event) at intervals from 0 to 90 days after application. The cores were divided into 6-inch (15.2 cm) depth bands and five core samples were composited (by depth) to give three replicate composite treated samples per interval per depth band and one control composite sample per interval per depth band. Samples were frozen immediately after collection, shipped to the laboratory on dry ice, and stored frozen at the laboratory while awaiting extraction and analysis.

Soil moisture was determined by drying to constant weight for at least 24 hours at 105 ± 5 °C. Composite soil samples were homogenised with a hammer or grinding mill in the presence of dry ice. Homogenised soil samples were extracted by blending with 95:5 v/v acetone/water for 3–5 minutes, then filtered. Aliquots of the filtered extract were evaporated to aqueous residue, combined with aqueous sodium chloride, then partitioned twice against dichloromethane. The dichloromethane portions were combined and evaporated to dryness and the samples reconstituted in 1:1 v/v hexane/acetone before cleanup by solid phase extraction using Celite and 4:1 w/w Celite/charcoal columns with elution by 1:1 hexane/acetone. Cleaned up extracts were evaporated to near dryness, and made up to volume in acetone.

The cleaned up extracts were analysed for dimethoate and omethoate by GC with flame photometric detection (FPD) operating in the phosphorus mode. The LOQ was 0.01 mg/kg for each analyte and good recoveries were achieved – control samples fortified at between 0.01 and 0.5 mg/kg showed mean recoveries of 90.7% (s.d. = 11.0%) and 78.3% (s.d. = 17.6%) for dimethoate and omethoate respectively. All samples were extracted and analysed within 4 months, and dimethoate and omethoate residues were shown by stability data on fortified control samples to be stable over that period.

Temperatures were within the normal range expected for late summer and autumn, while rainfall was slightly above average. No residues of either dimethoate or omethoate were found above LOQ in any of the control samples. Residues of dimethoate in the 0–6 inch core section are tabulated below. All results are reported on an as-is (wet weight) basis.

Table 21 Residues of dimethoate and omethoate in 0–6 inch cores of silty loam soil (Snook, TX, USA) after a single application of dimethoate at 1.5 lb ai/acre (1.7 kg ai/ha)

Time (days after application)	Dimethoate residues (mg/kg)	Omethoate residues (mg/kg)
-2	< 0.01 (3) (< 0.01)	< 0.01 (3) (< 0.01)
0+	0.54, 0.49, 0.42 (0.48)	< 0.01 (3) (< 0.01)
1	0.33, 0.41, 0.37 (0.37)	< 0.01 (3) (< 0.01)
2	0.34, 0.41, 0.46 (0.40)	< 0.01 (3) (< 0.01)
3	0.38, 0.38, 0.34 (0.37)	< 0.01 (3) (< 0.01)
6	0.28, 0.30, 0.39 (0.32)	< 0.01 (2), 0.010 (< 0.01)
11	0.24, 0.22, 0.17 (0.21)	< 0.01 (3) (< 0.01)
14	0.21, 0.13, 0.16 (0.17)	< 0.01 (3) (< 0.01)
28	0.077, 0.099, 0.086 (0.088)	< 0.01 (3) (< 0.01)
60	< 0.01 (3) (< 0.01)	< 0.01 (3) (< 0.01)
90	< 0.01 (3) (< 0.01)	< 0.01 (3) (< 0.01)

No residues of dimethoate were found above the LOQ in any samples after day 28. With the exception of a single measurement of 0.010 mg/kg omethoate for one replicate at 6 days for the 6-12 inch core, no residues of dimethoate or omethoate were found at any depth besides the 0–6 inch core.

A simple first order (SFO) decline model gave an excellent fit to the decline curve for dimethoate residues, with a pseudofirst order rate constant of 0.079 $days^{-1}$, giving a half life for dimethoate of 8.8 days with 95% confidence limits of 7.3 and 11 days). The half life of omethoate was estimated to be very short at less than 1 day.

The dissipation of dimethoate from soil in the field after applications to bare ground plots in New York State was investigated (Jacobson and Willams – 1994b). Dimethoate was applied on 30 July in a single application as a 250 g/kg wettable powder (WP) formulation using a tractor-mounted boom sprayer to a plot of bare sandy loam soil in Waterloo, NY, at a rate of 4 lb ai/acre (4.5 kg ai/ha). Soil cores were collected to a depth of 48 inches (122 cm) from the treated (15 cores per sampling event) and an adjacent untreated control plot (5 cores per sampling event) at intervals from 0 to 88 days after application. The cores were divided into 6-inch (15.2 cm) depth bands and five core samples were composited (by depth) to give three replicate composite treated samples per interval per depth band and one control composite sample per interval per depth band. Samples were frozen immediately after collection, shipped to the laboratory on dry ice, and stored frozen at the laboratory while awaiting extraction and analysis.

Soil moisture was determined by drying to constant weight for at least 24 hours at 105 ± 5 °C. Composite soil samples were homogenised with a hammer or grinding mill in the presence of dry ice. Homogenised soil samples were extracted by blending with 95:5 v/v acetone/water for 3-5 minutes, then filtered. Aliquots of the filtered extract were evaporated to aqueous residue, combined with aqueous sodium chloride, then partitioned twice against dichloromethane. The dichloromethane portions were combined and evaporated to dryness and the samples reconstituted in 1:1 v/v hexane/acetone before cleanup by solid phase extraction using Celite and 4:1 w/w Celite/charcoal columns with elution by 1:1 hexane/acetone. Cleaned up extracts were evaporated to near dryness, and made up to volume in acetone.

The cleaned up extracts were analysed for dimethoate and omethoate by GC with flame photometric detection (FPD) operating in the phosphorus mode. The LOQ was 0.01 mg/kg for each analyte and good recoveries were achieved – control samples fortified at between 0.01 and 2.0 mg/kg showed mean recoveries of 92.4% (s.d. = 14.4%) and 77.5% (s.d. = 11.5%) for dimethoate and omethoate respectively. All samples were extracted and analysed within 6 months, and dimethoate and omethoate residues were shown by stability data on fortified control samples to be stable over that period.

Temperatures were within the normal range expected for late summer and autumn, while rainfall was slightly above average. No residues of either dimethoate or omethoate were found above LOQ in any of the control samples. Residues of dimethoate in the 0–6 inch core section are tabulated below. All results are reported on an as-is (wet weight) basis.

Table 22 Residues of dimethoate and omethoate in 0–6 inch cores of sandy loam soil (Waterloo, NY, USA) after a single application of dimethoate at 4 lb ai/acre (4.5 kg ai/ha)

Time (days after application)	Dimethoate residues (mg/kg)	Omethoate residues (mg/kg)
0-	< 0.01 (3) (< 0.01)	< 0.01 (3) (< 0.01)
0+	1.55, 1.28, 1.32 (1.38)	< 0.01 (3) (< 0.01)
1	1.59, 1.35, 1.37 (1.44)	< 0.01 (3) (< 0.01)
2	1.78, 1.14, 1.65 (1.52)	0.01, < 0.01 (2) (< 0.01)
3	1.17, 1.29, 1.71 (1.39)	0.014, 0.016. 0.020 (0.017)
6	0.61, 0.51, 0.66 (0.59)	< 0.01 (3) (< 0.01)
10	0.18, 0.28, 0.23 (0.23)	< 0.01 (3) (< 0.01)
14	0.10, 0.041, 0.13 (0.090)	< 0.01 (3) (< 0.01)
28	0.25, < 0.01, < 0.01 (0.012)	< 0.01 (3) (< 0.01)
60	< 0.01 (3) (< 0.01)	< 0.01 (3) (< 0.01)

Time (days after application)	Dimethoate residues (mg/kg)	Omethoate residues (mg/kg)
88	< 0.01 (3) (< 0.01)	< 0.01 (3) (< 0.01)

No residues of dimethoate were found above the LOQ in any samples after day 28. With the exception of a single measurement of 0.023 mg/kg dimethoate for one replicate at 10 days for the 6–12 inch core, no residues of dimethoate or omethoate were found at any depth besides the 0–6 inch core.

A simple first order (SFO) decline model gave an excellent fit to the decline curve for dimethoate residues, with a pseudofirst order rate constant of 0.15 $days^{-1}$, giving a half life for dimethoate of 4.5 days with 95% confidence limits of 3.7 and 6.9 days). The half life of omethoate was estimated to be very short at less than 1 day.

The dissipation of dimethoate from soil after application to beans, grapes, and bare ground plots was studied in California in (Becker-1991). Details of the applications are summarized below.

Table 23 Summary of applications for the 1991 California field soil dissipation study (Becker – 1991)

Location, crop (variety), soil type	Formulation	Application type, growth stage	Number of applications (retreatment interval, days)	Application rates (kg ai/ha)
Fresno County, CA, USA, snap beans (Kentucky Wonder), sandy loam	480 g/L EC	Foliar broadcast, tractor mounted boomsprayer 1: early bloom 2: bloom to 5 cm pods) 3: 5-7.5 cm pods	3 (7, 7)	0.56, 0.56, 0.56
Fresno County, CA, USA, grapes (Flame seedless), sandy loam	250 g/kg WP	Airblast foliar spray	2 (14)	2.8, 2.8
Fresno County, CA, USA, bare ground, sandy loam	480 g/L EC	Bare earth application, overhead spray boom	2 (14)	2.8, 2.8

Three replicate treated plots and a control plot were established for each crop and for the bare ground study. Soil cores (five per replicate per sampling interval) were collected to a depth of 48 inches (121.9 cm) from the treated and control plots at intervals from the day before the first application through to up to 7.5 months after the final application. The cores were segmented (0–6 inch, 6–12 inch, 12–18 inch, 18–24 inch, 24–36 inch, and 36–48 inch) and a composite sample from each replicate prepared for each depth band. All samples were frozen within 2 hours of collection.

Samples were analysed for dimethoate and omethoate using a GC-FID method. Good concurrent recoveries were achieved for both analytes (mean values in the range 90–113%), with LOQs of 0.01 mg/kg. good results for stability samples were observed over 0–6 months fortified storage, with samples analysed between 7 days and 9 months from collection.

Table 24 Distribution of dimethoate and omethoate in sandy loam soil (Fresno, California) after 3 × 0.56 kg ai/ha applications of dimethoate to beans (Becker – 1991)

Sampling interval	Depth (cm)	Dimethoate (mg/kg)	Omethoate (mg/kg)
0DAA1	0-15.2	0.062	0.01
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
3DAA1	0-15.2	0.045	0.042

Sampling interval	Depth (cm)	Dimethoate (mg/kg)	Omethoate (mg/kg)
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
7DAA1	0-15.2	0.034	0.013
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
7DAA1/0DAA2	0-15.2	0.090	0.008
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
10DAA1/3DAA2	0-15.2	0.125	0.015
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
	45.7-61.0	< 0.01	< 0.01*
14DAA1/7DAA2	0-15.2	0.085	0.021
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
14DAA1/0DAA3	0-15.2	0.179	0.017
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
15DAA1/1DAA3	0-15.2	0.069	0.008
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
17DAA1/3DAA3	0-15.2	0.124	0.025
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
21DAA1/7DAA3	0-15.2	0.095	0.021
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
28DAA1/14DAA3	0-15.2	0.041	0.011
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
48DAA1/34DAA3	0-15.2	0.019	< 0.01
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
76DAA1/62DAA3	0-15.2	0.024	0.008
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01

*0.010 mg/kg in one of three replicates

Table 25 Distribution of dimethoate and omethoate in sandy loam soil (Fresno, California) after 2 × 2.8 kg ai/ha applications of dimethoate to grapes (Becker – 1991)

Sampling interval	Depth (cm)	Dimethoate (mg/kg)	Omethoate (mg/kg)
4DAA1	0-15.2	0.158	0.041
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
7DAA1	0-15.2	0.121	0.024
	15.2-30.5	0.010	< 0.01
	30.5-45.7	< 0.01	< 0.01
13DAA1	0-15.2	0.076	0.034

Sampling interval	Depth (cm)	Dimethoate (mg/kg)	Omethoate (mg/kg)
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
18DAA1/5DAA2	0-15.2	0.215	0.080
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
20DAA1/7DAA2	0-15.2	0.158	0.046
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
27DAA1/14DAA2	0-15.2	0.074	0.040
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
48DAA1/35DAA2	0-15.2	0.019	< 0.01
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01
67DAA1/54DAA2	0-15.2	< 0.01	< 0.01
	15.2-30.5	< 0.01	< 0.01
	30.5-45.7	< 0.01	< 0.01

Table 26 Distribution of dimethoate and omethoate in sandy loam soil (Fresno, California) after 2 × 2.8 kg ai/ha applications of dimethoate to bare ground (Becker – 1991)

Sampling interval	Depth (cm)	Dimethoate (mg/kg)	Omethoate (mg/kg)
0DAA1	0 – 15.2	0.687	0.032
	15.2 – 30.5	0.014	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
3DAA1	0 – 15.2	0.527	0.106
	15.2 – 30.5	0.174	< 0.01
	30.5 – 45.7	0.080	< 0.01
	45.7 – 61.0	0.018	0.007
	61.0 – 91.4	< 0.01	0.011
7DAA1	0 – 15.2	0.536	0.354
	15.2 – 30.5	0.008	< 0.01
	30.5 – 45.7	0.011	< 0.01
	45.7 – 61.0	0.007	0.008
13DAA1	0 – 15.2	0.764	0.178
	15.2 – 30.5	0.012	< 0.01
	30.5 – 45.7	0.010	< 0.01
14DAA1/0DAA2	0 – 15.2	1.421	0.173
	15.2 – 30.5	0.008	< 0.01
	30.5 – 45.7	0.009	< 0.01
	45.7 – 61.0	< 0.01	< 0.01
	61.0 – 121.9	0.011	< 0.01
15DAA1/1DAA2	0 – 15.2	1.302	0.282
	15.2 – 30.5	0.007	< 0.01
	30.5 – 45.7	0.015	< 0.01
	45.7 – 61.0	0.008	< 0.01
17DAA1/3DAA2	0 – 15.2	1.006	0.435
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01

Sampling interval	Depth (cm)	Dimethoate (mg/kg)	Omethoate (mg/kg)
20/21DAA1	0 – 15.2	0.614	0.240
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
	45.7 – 61.0	0.001	< 0.01
27/28DAA1	0 – 15.2	0.500	0.369
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	0.007	< 0.01
48/44DAA1	0 – 15.2	0.280	0.152
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
	0 – 15.2	0.107	0.058
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
67/74DAA1	0 – 15.2	0.068	0.012
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
112/104DAA1	0 – 15.2	0.060	0.012
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
159DAA1	0 – 15.2	< 0.01	< 0.01
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01
290DAA1	0 – 15.2	< 0.01	< 0.01
	15.2 – 30.5	< 0.01	< 0.01
	30.5 – 45.7	< 0.01	< 0.01

Based on a first order kinetics model, DT50 values (half lives) of 9.8, 6.0, and 7.8 days were calculated for dimethoate the bean, grape and bare ground plots respectively.

The dissipation of dimethoate from soil under field conditions was investigated at four sites in Europe in 2010 (Zietz – 2011a). Dimethoate was applied as a 400 g/L EC formulation in a single application at a target rate of 1 kg ai/ha (target spray volume = 400 L/ha) using a small scale ground boomsprayer a to four plots of bare soil at each of four sites (one each in the Netherlands, Germany, Spain and Italy), with a single control plot designated at each site. Each treated plot was further divided into 14 subplots. Samples were collected the day before application, immediately after application and thereafter at intervals of 1 to ~84 days after application. At each interval, five cores were collected from one subplot from each of the four treated plots (each subplot was scheduled for sampling only once during the study). Samples were collected from the untreated control plot at intervals of -1, 0+, 7, 28 and 84 days, with five cores collected per sampling interval. With the exception of the 0+ sampling (when cores were collected to a depth of 30 cm), cores were collected from the treated plot to a depth of 90 cm. The -1 and 84 day samples from the control plot were collected to 90 cm, with the remainder collected to 30 cm. Soil cores were frozen as soon as possible after collection and kept frozen (≤ -18 °C) until analysis. With the exception of the day -1 samples from the Spanish site (when no residues would be expected), all samples were frozen within 24 hours of collection.

At the laboratory, the cores were divided into depth bands (0–10 cm, 10–20 cm, 20–30 cm, 30–50 cm, 50–70 cm, and 70–90 cm) and combined into a single treated (20 cores) or control (5 cores) composite sample for each depth band for each site and sampling interval. The composite samples were homogenised using hammer mill.

Soil moisture was determined by dry to constant weight (at least overnight) at 105–115 °C in an oven. Soil samples were extracted three times with 9:1 v/v acetone/water, and the combined extracts were acidified with formic acid and made up to volume with acetone. An aliquot of the extract was acidified further with formic acid, then evaporated to near dryness under a nitrogen stream before making up to volume with 0.01% aqueous formic acid. The extracts were analysed for dimethoate and omethoate using LC-MS/MS. Mean recoveries of 81–93% were obtained for dimethoate across the four sites for both transitions and at levels of 0.001 mg/kg (LOQ) and 0.01 mg/kg, while mean recoveries of 89–97% were obtained for omethoate at the same fortification levels. Duplicate analyses were performed for each composite treated sample, with single analyses for each composite control sample. Samples were analysed within 7 months of collection.

Average results for the dimethoate and omethoate concentrations in the treated samples are tabulated below. With the exception of two day zero samples from the Italian trial site, no residues were detected in any of the control samples. The bulk of the residue was found in the top 10 cm layer, with much lower concentrations found in the 10–20 cm zone, as well as in the 20–30 cm zone from the Spanish site, although those results were could have been the result of contamination.

Table 27 Dissipation of dimethoate from four sites in Europe after a single bare soil application at 1 kg ai/ha. Results are reported on a dry weight basis, as the mean values in the 0–10 cm layer*, which was where essentially all the residues were found

Trial site (trial number)	Soil type (USDA), pH (in $CaCl_2$)	Days after application	Residues (mg/kg)	
			Dimethoate	Omethoate
Vlagtwedde, NL-9541, the Netherlands (10-NL-108)	Sand, 4.0	-11	< 0.001	< 0.001
		0+	0.78	0.004
		1	0.80	0.005
		4	0.64	0.003
		8	0.29*	< 0.001
		14	0.069*	< 0.001
		28	0.014	< 0.001
		54	0.005	< 0.001
		87	0.004	< 0.001
Halen, Germany, D-49685, (10-DE-110)	Sand, 4.05	-1	< 0.001	< 0.001
		0+	0.89	0.007
		1	0.72	0.011
		3	0.54	0.005
		8	0.17*	0.001
		14	0.048*	< 0.001
		27	0.014*	< 0.001
		55	0.007	< 0.001
		85	0.007	< 0.001
Zafarraya, ES-18128, Spain (10-ES-112)	Silt loam, 6.94	-1	< 0.001	< 0.001
		0+	0.68*	0.001
		1	0.71*	0.002
		3	0.55*	0.002

Trial site (trial number)	Soil type (USDA), pH (in $CaCl_2$)	Days after application	Residues (mg/kg)	
			Dimethoate	Omethoate
		7	0.46*	0.011
		14	0.12*	< 0.001
		28	0.035*	< 0.001
		55	0.010	< 0.001
		91	0.005*	< 0.001
Vittoria, I-97019, Italy (10-IT-114)	Sandy loam, 6.98	-1	< 0.001	< 0.001
		0+	0.47	0.002
		1	0.44	0.006
		5	0.092*	< 0.001
		7	0.022*	< 0.001
		14	0.005*	< 0.001
		28	0.002*	< 0.001
		56	0.003*	< 0.001
		86	0.001	< 0.001

*Exceptions are indicated, where lower level residues found in lower layers were added to the 0-10 cm results.

DT_{50} values (half lives) of 5.8, 4.1, 7.0 and 2.2 days were estimated for dimethoate in the Vlagtwedde, Halen, Zafarraya, and Vittoria sites respectively using the single first order model, along with DT90 values of 19.3, 13.5, 23.3, and 7.4 days.

Omethoate

The dissipation of omethoate from soil under field conditions was investigated at four sites in Europe in 2010 (Zietz – 2012a). Omethoate technical active (93.1%) was applied as a 60% w/w premix in cyclohexanone in a single application at a target rate of 1 kg ai/ha (target spray volume = 400 L/ha) using a small scale ground boomsprayer a to four plots of bare soil at each of four sites (one each in the Netherlands, Germany, Spain and Italy), with a single control plot designated at each site. Each treated plot was further divided into 14 subplots. Samples were collected the day before application, immediately after application and thereafter at intervals of 1 to ~56 days after application. At each interval, five cores were collected from one subplot from each of the four treated plots (each subplot was scheduled for sampling only once during the study). Samples were collected from the untreated control plot at the -1, 0+, 7, and 28 day intervals, with five cores collected per sampling interval. With the exception of the 0+ sampling (when cores were collected to a depth of 30 cm), cores were collected from the treated plot to a depth of 90 cm. The -1 day samples from the control plot were collected to 90 cm, with the remainder collected to 30 cm. Soil cores were frozen as soon as possible after collection and kept frozen (≤ -18 °C) until analysis. With the exception of the day -1 samples from the Spanish site (when no residues would be expected), all samples were frozen within 24 hours of collection.

At the laboratory, the cores were divided into depth bands (0–10 cm, 10–20 cm, 20–30 cm, 30–50 cm, 50–70 cm, and 70–90 cm) and combined into a single treated (20 cores) or control (5 cores) composite sample for each depth band for each site and sampling interval. The composite samples were homogenised using hammer mill.

Soil moisture was determined by dry to constant weight (at least overnight) at 105–115 °C in an oven. Soil samples were extracted three times with 9:1 v/v acetone/water, and the combined extracts were acidified with formic acid and made up to volume with acetone. An aliquot of the extract was acidified further with formic acid, then evaporated to near dryness under a nitrogen stream

before making up to volume with 0.01% aqueous formic acid. The extracts were analysed for omethoate using LC-MS/MS. Mean recoveries of 89–97% were obtained for omethoate across the four sites for both transitions and at levels of 0.001 mg/kg (LOQ) and 0.01 mg/kg. Duplicate analyses were performed for each composite treated sample, with single analyses for each composite control sample. Samples were analysed within 8 months of collection.

Average results for the omethoate concentrations in the treated samples are tabulated below. No residues of omethoate were detected in any of the control samples, or in any of the pre-application treated plot samples. Essentially all of the residue was found in the top 10 cm layer, with two detections in the 10–20 cm zone, one at 0.004 mg/kg at the German trial site (on day 0), and four at up to 0.03 mg/kg at the Spanish site (on days 0–7), and one low level detection at 0.001 mg/kg in the 30–50 cm layer at the Spanish site on day 1.

Table 28 Dissipation of omethoate from four sites in Europe after a single bare soil application at 1 kg ai/ha. Results are reported on a dry weight basis, as the mean values in the 0–10 cm layer*, which was where essentially all the residues were found.

Trial site (trial number)	Soil type (USDA), pH (in $CaCl_2$)	Days after application	Residues of omethoate (mg/kg)
Vlagtwedde, NL-9541, the Netherlands (10-NL-108)	Sand, 4.0	-11	< 0.001
		0+	0.33
		1	0.22
		4	0.016
		8	0.002
		14	< 0.001
		28	< 0.001
		54	< 0.001
Halen, Germany, D-49685, (10-DE-110)	Sand, 4.05	-1	< 0.001
		0+	0.56*
		1	0.45
		2	0.090
		3	0.054
		8	0.001
		14	< 0.001
		28	< 0.001
		56	< 0.001
Zafarraya, ES-18128, Spain (10-ES-112)	Silt loam, 6.94	-1	< 0.001
		0+	0.26*
		1	0.19*
		3	0.095*
		7	0.090*
		14	< 0.001
		28	< 0.001
		55	< 0.001
Vittoria, I-97019, Italy	Sandy loam, 6.98	-1	< 0.001

Trial site (trial number)	Soil type (USDA), pH (in $CaCl_2$)	Days after application	Residues of omethoate (mg/kg)
(10-IT-114)			
		0+	0.20
		1	0.22
		5	< 0.001
		7	< 0.001
		14	< 0.001
		28	< 0.001
		56	< 0.001

*The exceptions are some of the German and Spanish trial results, where low level residues found in lower layers were added to the 0-10 cm results.

DT_{50} values (half lives) of 1.4, 1.3, 2.9, and 1.7 days were estimated for omethoate in the Vlagtwedde, Halen, Zafarraya, and Vittoria sites respectively using the single first order model, along with DT90 values of 4.5, 4.4, 9.8, and 5.6 days.

Confined rotational crops

A confined rotational accumulation study on wheat, lettuce and turnips was conducted using [^{14}C-methoxy]-dimethoate (Adair – 1995). Planting boxes containing a sandy loam soil (pH 6.4, organic matter 1.6%) were treated with the test substance at 0.5 lb ai/A (0.56 kg ai/ha). The test crops were planted in the ^{14}C-dimethoate treated soil at 30 and 120 days after treatment. The planting boxes were maintained outdoors while fallow and in greenhouses after planting. Lettuce and wheat were grown in the same planting box. A separate box was used for turnip. Wheat forage was harvested 62 and 168 days after application for the first and second rotation respectively. Lettuce was harvested 78 and 174 days after application, turnip (root and foliage) at 100 and 208 days after application, wheat hay at 97 and 216 days after application, while wheat grain and straw was harvested 141 and 272 days after application for each rotation. Total radioactive residues found in the crops after each plant back interval are summarized in Table 29.

Table 29 Total radioactive residues in rotational crops after treatment of soil with [^{14}C-methoxy]-dimethoate

Crop	TRR (mg eq/kg)	
	30-day planting interval	120-day planting interval
Lettuce	0.030	0.003
Turnip foliage	0.037	0.005
Turnip root	0.008	0.001
Wheat forage	0.036	0.004
Wheat hay	0.037	0.009
Wheat straw	0.045	0.020
Wheat grain	0.021	0.012

Low levels of radioactivity were taken up by the rotational crops. The TRRs in the crops planted 120 days after treatment were significantly lower than those planted 30 days after treatment. Given the low TRRs observed for the 30-day and 120-day plant back intervals, it was not thought necessary to plant additional crops after longer plant back intervals.

Crop matrices with TRR >0.01 mg eq/kg were subjected to solvent extraction (acetonitrile or acetonitrile/HCl, Table 30). Extractability ranged from 15% in 30-day PBI wheat straw to 74% in 30-day PBI lettuce. Residues in the post-extraction solids ranged from 0.006 to 0.031 mg eq/kg.

Extracts containing >0.01 mg eq/kg were analysed by HPLC. Dimethoate and omethoate were not detected in any of the crop extracts. HPLC analysis of the extracts generally showed a single

peak that eluted close to the void volume of the column and contained 0.11 to 0.025 mg eq/kg (62 to 100% of extractable radioactivity). Attempts were made to derivatise the polar material (from wheat forage) with acetic anhydride but this did not change the chromatographic properties, implying that the radioactivity in the extract is not associated with carbohydrates or alcohols. Similarly, after refluxing with 6M HCl and HPLC analysis, all the radioactivity remained at the solvent front, suggesting the metabolites are not readily hydrolysable. The extractable radioactivity was also treated with a benzylating reagent (α-bromo-2,3,4,5,6-pentafluorotoluene), which reacts with acids to form less polar derivatives. HPLC analysis showed one major new peak and a large number of minor peaks. The material continuing to elute at the solvent front had decreased to 48% of the extract. While the derivatives were not identified, these results suggest that the polar material is multicomponent.

Table 30 Extractable and non-extractable residues from rotational crop matrices after application of [^{14}C-methoxy]-dimethoate

Crop	PBI (days)	TRR (mg/kg)	Extracted radiocarbon				PES		Total %
			Acetonitrile		Acetonitrile/HCl				
			%TRR	mg eq/kg	%TRR	mg eq/kg	%	mg eq/kg	
Lettuce	30	0.030	74.2	0.022	–	–	26.0	0.008	100
Turnip foliage	30	0.037	55.3	0.020	–	–	44.8	0.017	100
Wheat forage	30	0.036	63.1	0.023	–	–	41.5	0.015	105
Wheat hay	30	0.037	34.8	0.013	–	–	83.8	0.031	118
Wheat straw	30	0.045	14.9	0.007	57.6	0.027	29.1	0.013	102
	120	0.020	28.8	0.006	59.0	0.012	28.2	0.006	116
Wheat grain	30	0.021	3.8	0.001	58.4	0.012	51.8	0.011	114
	120	0.012	3.1	0.000	24.3	0.003	81.9	0.010	109

Crop samples were stored for 15–27 days between collection and analysis. Storage stability (Table 31) was shown to be acceptable through fortification of control crop samples with ^{14}C-dimethoate at approximately 1 ppm and storage at < -9 °C for up to 53 days.

Table 31 Summary of storage stability analyses

Sample	Days from fortification to analysis	Dimethoate fortification level (mg/kg)	Extracted radioactivity		Dimethoate in extract	
			%	mg/kg	%	mg/kg
Wheat forage	11	0.98	104	1.03	95.9	0.98
	42	0.97	101	0.98	99.5	0.97
Lettuce	14	1.02	102	1.04	91.4	0.95
	48	1.02	95.6	0.98	100	0.98
Wheat hay	6	1.04	97.4	1.01	99.3	1.00
	32	1.02	65	0.67	100	0.67
Turnip foliage	6	1.04	98.2	1.02	94.8	0.97
	32	0.98	100	0.98	88.1	0.86
Wheat grain	6	1.03	75.0	0.78	81.5	0.63
	53	1.03	95.7	0.98	97.7	0.96
Wheat straw	6	1.03	96.6	0.998	100	0.998
	53	1.03	108	1.11	99.1	1.10

Analysis of the soil demonstrated that soil TRRs decreased from approximately 0.3 to 0.4 mg/kg at application to approximately 0.1 mg/kg at the 30-day planting interval and approximately 0.08 mg/kg at the 120-day planting interval. It was suggested that this is consistent with oxidative metabolism of the O-methyl groups leading to loss of the radiocarbon from the soil as

CO_2. Soil samples were extracted with acetonitrile/water. Extractabilities for the 0-day soils were 92.6 and 81.1%. It was suggested the lower than expected extractability in these samples was due to the samples not being adequately homogeneous. Extractability decreased further for the 30-day and 120-day planting soils. For the 30-day soil 60.7 and 58.8% of the radioactivity was extractable from the lettuce/wheat and turnip plot respectively. For the 120-day planting soil 14.5 and 11.6% of the soil radiocarbon was extractable. These results suggest dimethoate and its metabolites become increasingly bound to the soil. HPLC analysis of the soil extracts indicated that dimethoate accounted for approximately 83% (0.053 mg/kg) of the extractable radioactivity in the 30-day planting soil and 24.9–53.8% (0.003–0.006 mg/kg) of the extractable radioactivity in the 120-day planting soil. Other soil extract components were not identified – the largest component accounted for 10% (0.005 mg/kg) of the 30-day soil extract.

ANIMAL METABOLISM

Rats

Evaluation of the metabolism studies in rats was carried out by the WHO Core Assessment Group.

Residue components observed in rat metabolism were dimethoate, omethoate, dimethoate carboxylic acid, dimethyl dithiophosphate, dimethyl thiophosphate, and dimethyl phosphate.

Lactating goats

Two lactating goats (Jalali – 1995a) were administered [^{14}C-methoxy]dimethoate by capsule once daily for 3 consecutive days at a dose equivalent to 30 ppm in the diet (1.6 mg/kg body weight/day). An additional goat served as a control. Milk samples were collected twice daily from the treated goats during the dosing period. The treated animals were sacrificed 23 hours after the final dose and total radioactive residue levels were determined in tissues by combustion analysis.

The TRR found in the tissues and milk is summarized in Table 32 below. Recovery of the administered dose was good, at 92-96%.

Table 32 Total radioactive residues in goat tissues, excreta and milk (mg equivalents/kg) following dosing equivalent to 30 ppm in the diet for 3 days

Sample	Goat no. 651		Goat no. 658	
	% AR	mg eq/kg	%AR	mg eq/kg
Liver	0.5	1.221	0.5	1.012
Kidney	< 0.1	0.149	< 0.1	0.154
Muscle	0.7	0.070	0.5	0.047
Fat (composite)	0.1	0.045	0.1	0.057
Blood	0.1	0.076	0.1	0.079
Milk	< 0.1 (cumulative)	0.035 to 0.228	< 0.1 (cumulative)	0.052 to 0.135
Urine	90.7 (cumulative)	-	85.6 (cumulative)	-
Faeces	3.2 (cumulative)	-	3.9 (cumulative)	-
GI tract	0.5	-	0.7	-
Total	95.8	-	91.4	-

Residue levels in milk during the course of the study are summarized in Table 33. Total residues in milk ranged from 0.035–0.23 mg eq/kg, and were higher in samples collected within 8 hours post treatment, compared to samples collected 8 to 24 hours post treatment each day, suggesting rapid elimination in milk. It is not clear whether residues had reached a plateau, noting the short duration of the study.

Total residues in tissues were 1.2 mg eq/kg in liver, 0.15 mg eq/kg in kidney, 0.070 mg eq/kg in muscle, and 0.045 mg eq/kg in fat.

Table 33 Variation of residue levels in milk with time

Collection interval	Mean residue (mg eq/kg)	
	Goat 651	Goat 658
0–12 hr	0.146	0.082
12–24 hr	0.035	0.055
24–36 hr	0.176	0.127
36–48 hr	0.081	0.052
48–60 hr	0.228	0.135
60 hr – sac.	0.102	0.070

Solvent extractability (acetonitrile, acetonitrile/water, acetone, and methanol/1 M NH_4OH) was variable, ranging from 28% in fat, to 90% in milk (Table 34). Liver contained the highest residues, and with a PES of 47% of TRR (0.58 mg eq/kg), was subjected to harsher extraction techniques, with protease removing a further 22% of TRR, 6 M HCl at 90 °C removing 14% of TRR, and 3 M NaOH removing 1.8% of TRR.

Metabolites identified in tissue and liver extracts are summarized in Table 33 below. Dimethoate was rapidly metabolized. In tissues and milk, the bulk of the residue comprised radioactivity incorporated into natural products (released by the solvent extraction, protease and acid extraction). Phosphorylated natural products comprised 62% TRR/0.76 mg eq/kg in liver, 87% TRR/ 0.13 mg eq/kg in kidney, 70% TRR/0.049 mg eq/kg in muscle, and 65% TRR/0.15 mg eq/kg in milk. Residues in fat were not further analysed due to the low levels. Anionic species (mainly dimethyl phosphate, dimethyl thiophosphate and dimethyl dithiophosphate) comprised 6.2% TRR/0.076 mg eq/kg in liver, 13% TRR/0.02 mg eq/kg in kidney, 2.9% TRR/0.002 mg eq/kg in muscle, and 2.2% TRR/0.005 mg eq/kg in milk. No parent compound was identified. Omethoate was found at 9.8% TRR/0.12 mg eq/kg in liver, while dimethoate carboxylic acid was found at 2.5% TRR/0.031 mg eq/kg in liver, and 8.3% TRR/0.019 mg eq/kg in milk. Both omethoate and dimethoate carboxylic acid were released from liver by protease digestion. Metabolites identified in urine were dimethoate carboxylic acid, dimethyl thiophosphate and dimethyl phosphate.

Table 34 Extractability of residues from goat tissues and milk following dosing for 3 days with [^{14}C-methoxy]-dimethoate at 1.6 mg/kg bw/day (30 ppm in feed)

Fraction	Liver		Kidney		Muscle		Fat		Milk (Sample 48-60h)	
TRR [mg/kg] [a]	1.221		0.149		0.070		0.045		0.228	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Initial extractions										
Hexane	0.3	0.003	2.0	0.003	0.5 (26.7) [d]	0.000 (0.019)	10.6	0.005	8.5	0.020
ACN/H_2O (8:2 v/v)	41.1	0.502	66.1	0.099	57.0	0.040	Ne	-	81.6	0.185
ACN/H_2O (7:1 v/v)	Ne	-	Ne	-	Ne	-	11.9	0.005	Ne	-
ACN	Ne	-	Ne	-	Ne	-	4.2	0.002	Ne	-
Acetone	Ne	-	Ne	-	Ne	-	1.8	0.001	Ne	-

Fraction	Liver		Kidney		Muscle		Fat		Milk (Sample 48-60h)	
TRR [mg/kg] [a]	1.221		0.149		0.070		0.045		0.228	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
MeOH/1 M NH_4OH (1:1)	2.0	0.024	5.1	0.008	Ne	-	Ne	-	Ne	-
Total extracted	43.4	0.529	73.2	0.110	84.2	0.059	28.5	0.013	90.1	0.205
Initial unextracted [b]	47.1	0.575	34.3	0.051	17.3	0.012	82.1	0.037	9.9	0.023
Additional extractions										
Accountability after initial extraction	90.5	-	107.5	-	101.7	-	110.7	-	99.9	-
Protease	21.5	0.263	Ne	-	Ne	-	Ne	-	Ne	-
6N HCl 90/100 °C	14.4	0.176	Ne	-	Ne	-	Ne	-	Ne	-
3N NaOH 80 °C	1.8	0.022	Ne	-	Ne	-	Ne	-	Ne	-
Total solubilised [c]	81.1	0.990	73.2	0.110	84.3	0.059	28.6	0.013	90.0	0.205
Total final unextracted	18.9	0.231	34.3	0.051	17.3	0.012	82.1	0.037	9.9	0.023

[a] TRR determined from individual animal group

[b] Unextracted TRR following hexane and acetonitrile/water extractions

[c] Combined initial extractions plus further extractions with protease, methanol/ammonium hydroxide and acid/base solvents

[d] Aqueous layer observed beneath hexane extraction layer. Ne – Not extracted with this solvent

Table 35 Identification and characterisation of residue components in goat tissues and milk following dosing for 3 days with [^{14}C-methoxy]-dimethoate at 1.6 mg/kg bw/day (30 ppm in feed)

Compound	Liver		Kidney		Muscle		Milk (Sample 48-60h)	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Total	-	1.221	-	0.149	-	0.070	-	0.228
Dimethoate	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000
Omethoate	9.8	0.120[a]	0.0	0.000	0.0	0.000	0.0	0.000
Dimethoate carboxylic acid	2.5	0.031[a]	0.0	0.000	0.0	0.000	8.3	0.019[f]
^{14}C Phosphorylated natural products[b]	62.4	0.762	87.2	0.130	70.0	0.049	64.9	0.148
Anions such as dimethyl phosphate and dimethyl thiophosphate	6.2	0.076	13.4	0.020	2.9	0.002	2.2	0.005
Total identified [c]	12.4	0.151	0.0	0.000	0.0	0.000	8.3	0.019
Total characterised [d]	68.6	0.838	100.7	0.150	72.9	0.051	67.1	0.153

Compound	Liver		Kidney		Muscle		Milk (Sample 48-60h)	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Total extractable[e]	81.0	0.989	73.2	0.110	84.3	0.059	90.0	0.205
Unextracted (PES) [f]	18.9	0.231	34.3	0.051	17.3	0.012	9.9	0.023
Accountability [g]	100.0	1.221	107.5	0.161	101.6	0.071	100.0	0.228

[a] Released from protease extraction

[b] Combination of TRR from HPLC early elution peak following initial ACN/H_2O extraction (minus benzylated ions) plus subsequent protease, acid/base extractions and inclusive of non-extractable material (PES)

[c] Sum of Dimethoate and metabolites identified in extracts; [d] Sum of characterised material not including identified material

[e] Total solubilised material following initial and additional extractions – see Table 2-5; [f] Not verified with a reference standard due to matrix effects

[f] Residues remaining after exhaustive extractions – characterised as ^{14}C phosphorylated natural products.

[g] Accountability = (Total extractable + Total unextractable)/(TRRs from combustion analysis)

Residues of dimethoate and its metabolites did not concentrate in fat. All extractable fractions of fat contained ≤ 0.005 mg eq/kg.

The composition of the residue in a subsample of milk from the previous study was investigated further (Jalali – 1997). The TRR in the sample was 0.210 mg eq/kg. A hexane extraction removed 7.5% (0.015 mg eq/kg) with a further 68.9% (0.145 mg eq/kg) extracted into acetonitrile-water (8:2).

The acetonitrile-water extract was fractionated on an anion-exchange (SAX) SPE column. About 40% (0.058 mg eq/kg) eluted in water (neutral fraction) with a further 53% (0.077 mg eq/kg) eluted by acid (acidic fraction). Analysis of both fractions by C-18 HPLC showed that the radiocarbon was mostly in the solvent front in both cases. Treatment with protease did not change the HPLC profile, indicating that neither the neutral nor the acidic fraction is composed of radiocarbon conjugated to protein or peptides.

Both fractions were derivatised with pentafluorobenzyl bromide (PFBB) and analysed by HPLC. The neutral fraction showed 4 benzylated products, with the most concentrated present at 0.009 mg eq/kg. The underivatised solvent front band contained 0.032 mg eq/kg. The report suggested that this result demonstrates that the neutral fraction contains multiple conjugates containing carboxylic acid groups or phenol groups. It was suggested that the polar metabolites that form derivatives with PFBB may be amino acid conjugates or metabolic products such as dimethyl phosphate or dimethyl thiophosphate (detected previously in urine). Although these metabolites are anionic, their high solubility in water may result in their elution in the neutral layer.

The acidic fraction only showed one benzylated product (at 0.010 mg eq/kg) after derivatisation with PFBB. Underivatised material in the solvent front accounted for 0.063 mg eq/kg. This result indicates that only a small amount of the radiocarbon in this fraction is conjugated to products containing carboxylic or phenol groups. In addition, attempted derivatisation with o-phthalaldehyde did not produce any new compounds, indicating that the radiocarbon in the acidic fraction does not contain any amino conjugates (or primary amine groups). The proposed metabolic pathway in goats leading to the residues in tissues and milk is summarized in Figure 5. Hydrolysis leads to the formation of dimethoate carboxylic acid. Oxidation leads to the formation of omethoate. Further oxidation leads to an intermediate which is proposed to phosphorylate natural products. Cleavage of the P-SCH_2 link results in the formation of dimethyl thiophosphate and dimethyl phosphate found in urine.

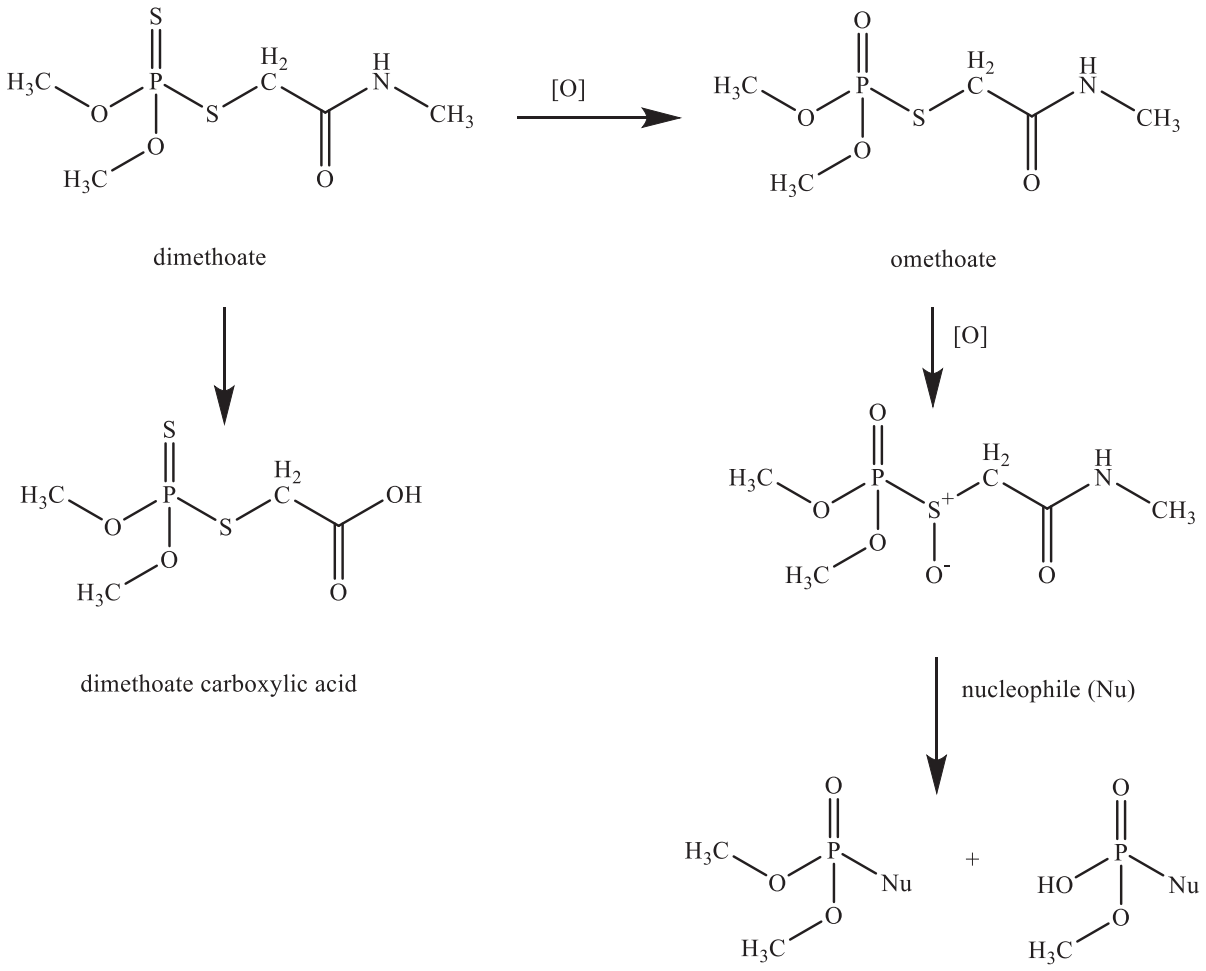


Figure 5 Metabolism of [^{14}C-dimethoxy]-dimethoate in goats

Laying hens

Three groups of five White Leghorn laying hens (Jalali – 1995b) were orally administered [^{14}C-methoxy]dimethoate by capsule once daily for 7 consecutive days at a dose rate equivalent to 10 ppm in the diet (approximately 0.9 mg/kg bw/day). An additional group of hens served as a control. Egg samples were collected twice daily and separated into yolks and whites. Treated hens were sacrificed between 22 and 24 hours after the final dose. Tissues and eggs were composited by treatment group. Total radioactive residue levels (TRRs) were determined by combustion analysis.

Mean cumulative radioactivity recovered in excreta (including cage wash) was 75%, in GI tract < 1% and in bile < 1%. Mean cumulative radioactivity recovered in eggs accounted for < 1% of the administered dose.

Mean daily TRR levels in yolks ranged from 0.018 to 0.34 mg eq/kg, and in whites ranged from 0.090 to 0.18 mg eq/kg. A plateau in egg residues was not reached during dosing. Total residues in tissues were 0.62–0.69 mg eq/kg in liver, 0.079–0.10 mg eq/kg in muscle, 0.024–0.061 mg eq/kg in fat, and 0.042–0.066 mg eq/kg in skin.

TRRs in tissues and eggs are summarized in Table 36 below.

Table 36 Total radioactive residues in tissues and eggs following dosing with [^{14}C-methoxy]-dimethoate at (10 ppm in the diet) for 7 days

Sample	Collection	Mean TRR (mg eq/kg)			
		Group B	Group C	Group D	Mean
Liver	Sacrifice	0.615	0.621	0.687	0.641
Breast muscle		0.098	0.087	0.102	0.096
Thigh muscle		0.079	0.090	0.083	0.084
Fat		0.028	0.024	0.061	0.038
Skin		0.042	0.044	0.066	0.051
Blood		0.234	0.234	0.242	0.237
Egg yolk	0–24 hr	0.018	0.020	0.016	0.018
	24–48 hr	0.040	0.051	0.044	0.045
	48–72 hr	0.106	0.099	0.110	0.105
	72–96 hr	0.156	0.168	0.199	0.174
	96–120 hr	0.277	0.246	0.241	0.255
	120–144 hr	0.279	0.295	0.414	0.329
	144 hr – sac.	0.310	0.351	0.355	0.339
Egg white	0–24 hr	0.080	0.070	0.120	0.090
	24–48 hr	0.092	0.112	0.141	0.115
	48–72 hr	0.090	0.120	0.202	0.137
	72–96 hr	0.139	0.152	0.249	0.180
	96–120 hr	0.183	0.152	0.175	0.170
	120–144 hr	0.146	0.115	0.140	0.134
	144 hr – sac.	0.144	0.161	0.149	0.151

Extractability of radioactivity from tissues and eggs with acetonitrile and water ranged from 8.9% in liver to 50% in egg white (Table 36). Tissues were subjected to protease, weak base, strong acid and strong base hydrolysis to release the radioactivity. Significant proportions were released by these harsher treatments, with 10–44% of TRR released by protease digestion, 1–14% by weak base extraction, 2.4–18% by strong base reflux, and 1.2–9.0% by strong acid reflux.

Dimethoate was not detected in any of the tissues, egg, excreta or blood extracts, indicating rapid metabolism. Omethoate (3.1% TRR/0.004 mg eq/kg in egg white and 9.9% TRR/0.081 mg eq/kg in liver) and dimethoate carboxylic acid (3.9% TRR/0.005 mg eq/kg in egg white and 16% TRR/0.13 mg eq.kg in liver) were identified by HPLC. The largest fractions of these metabolites were released from liver and egg white by protease treatment (Table 37). The bulk of the residue in all matrices was characterised as phosphorylated natural products, at essentially 100% TRR/0.10 mg eq/kg in breast muscle, 92% TRR/0.072 mg eq/kg in thigh muscle, 70% TRR/0.027 mg eq/kg in skin, 62% TRR/0.51 mg eq/kg in liver, 94% TRR/0.18 mg eq/kg in egg yolk, 81% TRR/0.10 mg eq/kg in egg white, and 58% TRR/0.016 mg eq/kg in fat

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Table 37 Extractability of residues from tissues and eggs after dosing of laying hens with [^{14}C-methoxy]-dimethoate at 0.9 mg /kg bw/day (10 ppm in feed) for 7 days

Fraction	Breast muscle		Thigh muscle		Skin		Liver (Sample F)		Liver (Sample D)		Egg yolks (Sample A)		Egg yolks (Sample D)		Egg whites (Sample A)		Fat	
TRR (mg eq/kg) [a]	0.098		0.079		0.038		0.705		0.822		0.192		0.194		0.127		0.028	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Initial extractions																		
Hexane	Ne	-	0.0	0.000	0.0	0.000	0.2	0.001	3.0	0.025	Ne	-	Ne	-	Ne	-	30.6	0.008
ACN/H_2O (8:2 v/v)	46.2	0.045	36.0	0.029	26.7	0.009	8.9	0.063	Ne	-	29.4	0.057	Ne	-	50.0	0.064	9.8	0.003
MeOH/1 M NH_4OH (1:1)	Ne	-	Ne	-	Ne	-	Ne	-	21.7	0.178	Ne	-	72.6	0.141	Ne	-	Ne	-
Initial unextracted [b]	59.1	0.058	54.2	0.043	54.6	0.021	90.5	0.638	75.4	0.620	57.5	0.110	25.2	0.049	37.3	0.047	29.3	0.008
Accountability after initial extraction	105.3	-	90.2	-	81.3	-	99.6	-	100.1	-	86.8	-	97.8	-	87.3	-	69.7	-
Additional extractions																		
Protease	34.3	0.034	36.5	0.029	26.6	0.010	44.1	0.311	35.5	0.292	34.9	0.067	Ne	-	14.4	0.018	10.4	0.003
MeOH/1 M NH_4OH (1:1)	13.6	0.013	6.9	0.005	4.4	0.002	Ne	-	Ne	-	10.7	0.021	Ne	-	9.5	0.012	1.2	0.000
6N HCl 90/100 °C	8.4	0.008	6.2	0.005	9.0	0.003	Ne	-	27.0	0.222	1.2	0.002	Ne	-	4.5	0.006	5.4	0.001

Fraction	Breast muscle		Thigh muscle		Skin		Liver (Sample F)		Liver (Sample D)		Egg yolks (Sample A)		Egg yolks (Sample D)		Egg whites (Sample A)		Fat	
TRR (mg eq/kg) [a]	0.098		0.079		0.038		0.705		0.822		0.192		0.194		0.127		0.028	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Initial extractions																		
3N NaOH 80 °C	Ne	-	6.2	0.005	3.7	0.001	Ne	-	2.4	0.020	18.3	0.035	Ne	-	8.6	0.011	Ne	-
Total solubilised [c]	102.4	0.100	91.7	0.072	70.5	0.027	53.1	0.375	89.6	0.736	94.5	0.181	72.5	0.141	86.7	0.110	57.5	0.016
Total final unextracted	0.0	0.000	8.3	0.007	29.5	0.011	46.9	0.33	10.4	0.086	5.5	0.011	27.5	0.049	13.3	0.017	42.5	0.012

[a] TRR determined from individual animal group

[b] Unextracted TRR following hexane and acetonitrile/water (8:2) extractions

[c] Combined initial extractions plus further extractions with protease, methanol/ammonium hydroxide and acid/base solvents

Ne – Not extracted with this solvent

Table 38 Identification and characterisation of residue components in tissues and eggs after dosing of laying hens with [^{14}C-methoxy]-dimethoate at 0.9 mg /kg bw/day (10 ppm in feed) for 7 days

Compound	Breast muscle		Thigh muscle		Skin		Liver		Egg yolks		Egg whites		Fat	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Total	-	0.098	-	0.079	-	0.038	-	0.822	-	0.192	-	0.127	-	0.028
Dimethoate	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000	0.0	0.000
Omethoate [a]	0.0	0.000	0.0	0.000	0.0	0.000	9.9	0.081	0.0	0.000	3.1	0.004	0.0	0.000
Dimethoate carboxylic acid[a]	0.0	0.000	0.0	0.000	0.0	0.000	15.9	0.131	0.0	0.000	3.9	0.005	0.0	0.000
^{14}C Phosphorylated natural products [b]	102.4	0.100	91.7	0.072	70.5	0.027	62.5	0.514	94.5	0.181	81.1	0.103	57.5	0.016
Total identified [c]	0.0	0.000	0.0	0.000	0.0	0.000	25.8	0.212	0.0	0.000	7.0	0.009	0.0	0.000
Total characterised [d]	102.4	0.100	91.7	0.072	70.5	0.027	62.5	0.514	94.5	0.181	81.1	0.103	57.5	0.016
Total extractable [e]	102.4	0.100	91.7	0.072	70.5	0.027	89.6	0.736	94.5	0.181	86.7	0.110	57.5	0.016
Unextracted (PES) [f]	0.0	0.000	8.3	0.007	29.5	0.011	10.4	0.086	5.5	0.011	13.3	0.017	42.5	0.012
Accountability [g]	102.4	0.100	100.0	0.079	100.0	0.038	100.0	0.822	100.0	0.192	100.0	0.127	100.0	0.028

[a] Released from protease extraction

[b] Combination of TRR from HPLC early elution peak following initial acetonitrile/water extraction plus subsequent protease (minus Omethoate and Dimethoate carboxylic acid identified in liver and egg white), acid/base extractions

[c] Sum of Dimethoate and metabolites identified in extracts

[d] Sum of characterised material not including identified material

[e] Total solubilised material following initial and additional extractions – see Table 2-2

[f] Residues remaining after exhaustive extractions – characterised as ^{14}C phosphorylated natural products.

[g] Accountability = (Total extractable + Total unextractable)/(TRRs from combustion analysis)

A metabolic pathway for the production of radiocarbon residues in tissues was proposed (Figure 6). The pathway is the same as that proposed for the goat metabolism study, with hydrolysis leading to the formation of dimethoate carboxylic acid and oxidation leading to omethoate. Further oxidation is followed by reaction with naturally occurring nucleophiles.

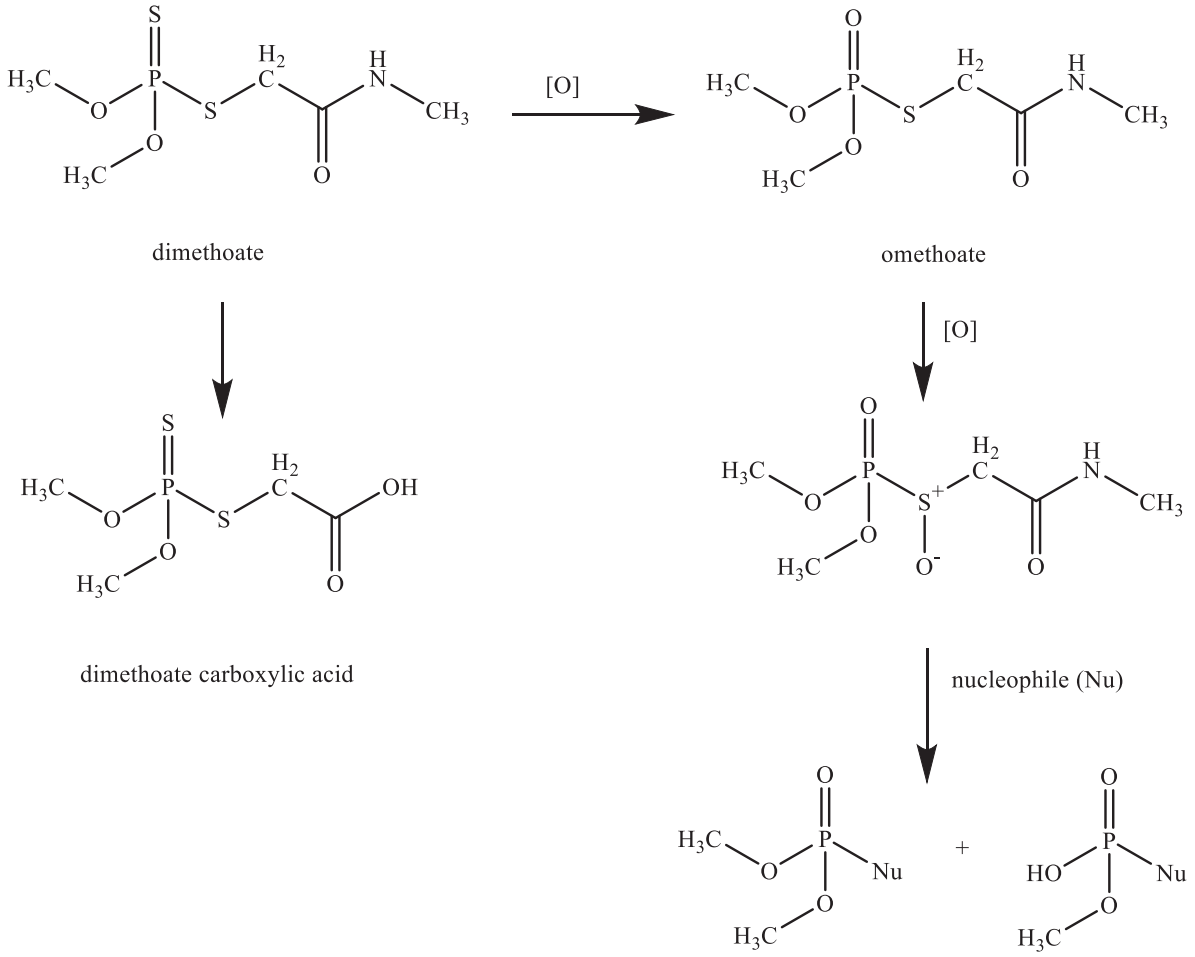


Figure 6 Metabolism of [^{14}C-dimethoxy]-dimethoate in hens

METHODS OF RESIDUE ANALYSIS

Plant matrices

GC-FPD method (study number)

This method was validated for analysis of orange matrices (Cordon – 2001). Samples were extracted by homogenising twice with acetone. Dichloromethane and sodium chloride were added and shaken. The aqueous phase was separated and the pH adjusted as necessary to pH >4 with 0.04 M NaOH and partitioned again with two further portions of dichloromethane. The organic fractions were combined, dried with anhydrous sodium sulfate, and then further cleaned up by dispersive solid phase extraction with charcoal and kieselguhr (diatomaceous earth). The extract was filtered and the kieselguhr/charcoal washed with dichlormethane. A small amount of n-dodecane was added and the extract reduced by rotary evaporation to < 50 mL, then transferred to another flash with an acetone washed and further reduced by evaporation under vacuum. The dried sample was then reconstituted in 3:1 v/v hexane/acetone, and further cleaned up by column chromatography (activated silica gel capped with anhydrous sodium sulfate, elution first with 3:1 v/v hexane/acetone for dimethoate, and then with 1:1 v/v hexane/acetone for omethoate). The eluted fractions were evaporated to dryness after addition of a few drops of n-dodecane, then reconstituted in acetone for analysis by gas chromatography with flame photometric detection (GC-FPD) operating in phosphorus mode (525 nm).

Good linearity was verified over the range 0.05–0.75 mg/L for both analytes. Limits of detection were 0.0002 mg/kg for both dimethoate and omethoate, while the LOQ was 0.01 mg/kg.

Table 39 Recoveries of dimethoate and omethoate for GC-FPD analysis of orange matrices

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	Omethoate recoveries (%)
Orange whole fruit	0.01	78, 83, 86, 88, 90, 90 (mean = 86, RSD = 5.4%)	82, 86, 90, 95, 96 (mean = 90, RSD = 6.4%)
	0.10	76, 85, 91, 93, 93, 96 (mean = 89, RSD = 8.2%)	78, 82, 86, 88, 95, 100 (mean = 88, RSD = 9.1%)
	1.0	83, 84, 86, 87, 91, 94 (mean = 87, RSD = 4.8%)	77, 80, 81, 82, 86, 89 (mean = 83, RSD = 5.2%)
Orange peel	0.01	74, 78, 82, 82, 85, 90 (mean = 82, RSD = 6.7%)	76, 78, 79, 80, 90, 92 (mean = 81, RSD = 8.7%)
	0.10	73, 77, 80, 82, 85, 91 (mean = 81, RSD = 7.6%)	72, 78, 80, 81, 82, 94 (mean = 81, RSD = 8.7%)
	1.0	71, 77, 77, 80, 81, 87 (mean = 79, RSD = 6.9%)	66, 72, 74, 74, 79, 82 (mean = 74, RSD = 7.2%)
Orange pulp	0.01	81, 83, 83, 85, 87, 89 (mean = 85, RSD = 3.5%)	80, 86, 87, 89, 93, 95 (mean = 89, RSD = 5.9%)
	0.10	81, 85, 85, 88, 90, 91 (mean = 86, RSD = 4.2%)	79, 82, 82, 83, 89, 96 (mean = 85, RSD = 7.5%)
	1.0	76, 79, 79, 82, 83, 91 (mean = 82, RSD = 6.5%)	68, 68, 72, 73, 76, 84 (mean = 74, RSD = 8.3%)

All mean recoveries and most individual recoveries were in the range 70–110%, while all RSD values were < 20%, indicating that the method is valid for analysis of orange peel, pulp and whole fruit samples. Sample extracts were shown to be stable for at least 2 weeks storage at 4 °C in the dark.

GC-FPD method (report number PGD-104)

A GC method with flame photometric detection was developed for analysis of dimethoate in plant matrices and validated for representative raw and processed commodities – olive flesh, olive, oil, orange, lettuce, and wheat grain (Jones – 2003).

Olive samples were chopped, the stones separated, homogenised, and extracted first by heating with ethyl acetate together with sodium bicarbonate and sodium sulfate, then homogenised. The supernatant was decanted through a filter, evaporated to near dryness then partitioned between hexane and acetonitrile. The acetonitrile phase was collected and evaporated to dryness, then reconstituted in 1:1 v/v cyclohexane/ethyl acetate.

Olive oil samples were dissolved in hexane then extracted with acetonitrile. The acetonitrile phase was evaporated to dryness and reconstituted in 1:1 v/v cyclohexane/ethyl acetate

Orange, lettuce and wheat grain samples were homogenised and extracted first by heating with ethyl acetate together with sodium bicarbonate and sodium sulfate, then homogenised. The ethyl acetate supernatant was decanted and evaporated to near dryness and reconstituted in 1:1 v/v cyclohexane/ethyl acetate.

Extracts were all cleaned up by gel permeation chromatography (eluted with 1:1 v/v cyclohexane/ethyl acetate). The eluates were reduced in volume and reconstituted in ethyl acetate. Samples were analysed by GC-FPD using matrix matched standards for quantification.

Table 40 Recovery of dimethoate and omethoate from plant matrices after GC-FPD analysis (Jones – 2003)

Matrix	Fortification level (mg/kg)	Dimethoate	Omethoate
Olives	0.01	82, 85, 86, 87, 91 (mean = 86, RSD =	61, 65, 72, 74, 78 (mean = 70, RSD =

Matrix	Fortification level (mg/kg)	Dimethoate	Omethoate
		3.5%)	9.8%)
	0.10	80, 84, 87, 88, 88 (mean = 85, RSD = 4.0%)	78, 79, 87, 87, 88 (mean = 84, RSD = 5.6%)
Olive oil	0.01	74, 80, 81, 84, 95 (mean = 83, RSD = 9.4%)	66, 68, 75, 80, 83 (mean = 75, RSD = 9.7%)
	0.10	77, 79, 82, 83, 91 (mean = 82, RSD = 6.9%	76, 77, 78, 81, 91 (mean = 81, RSD = 7.7%
Oranges	0.01	83, 85, 85, 86, 91 (mean = 86, RSD = 3.3%)	69, 72, 74, 79, 82 (mean = 75, RSD = 7.2%)
	0.10	79, 81, 83, 83, 85 (mean = 82, RSD = 2.7%)	69, 70, 76, 82, 89 (mean = 77, RSD = 11%)
Lettuce	0.01	75, 77, 77, 77, 77 (mean = 76, RSD = 1.3%)	83, 85, 86, 90, 91 (mean = 87, RSD = 4.0%)
	0.10	76, 77, 77, 78, 84 (mean = 78, RSD = 3.9%	79, 80, 80, 84, 91 (mean = 83, RSD = 6.0%)
Wheat grain	0.01	67, 70, 70, 72, 74 (mean = 70, RSD = 3.7%)	65, 70, 72, 73, 77 (mean = 71, RSD = 6.1%)
	0.10	70, 75, 78, 79, 83 (mean = 77, RSD = 6.3%)	59, 63, 69, 70, 76 (mean = 68, RSD = 10%)

Confirmatory analyses using a different column gave very similar results. With the exception of omethoate at 0.10 mg/kg in wheat grain (for which the mean recovery was 68%), mean recoveries were within 70–110% and RSD values were < 20%. Method linearity was good with $r^2 > 0.99$. An LOQ of 0.01 mg/kg was validated for both analytes in all matrices.

The method was independently validated (Kang – 2003a, Kang – 2003b, and Kang – 2003c). Minor modifications were made to the extraction for wheat grain, with the addition of a preliminary soaking in water and sodium bicarbonate for 2 hours prior to extraction.

Table 41 Independent method validation recovery of dimethoate and omethoate from plant matrices after GC-FPD analysis (Kang – 2003a, Kang – 2003b, and Kang – 2003c)

Matrix	Fortification level (mg/kg)	Dimethoate	Omethoate
Olives	0.01	87, 90, 94, 99, 100 (mean = 94, RSD = 6.0%)	97, 100, 105, 108, 109 (mean = 104, RSD = 5.0%)
	0.10	82, 85, 91, 91, 94 (mean = 89, RSD = 5.6%)	99, 104, 106, 110, 110 (mean = 106, RSD = 4.4%)
Olive oil	0.01	64, 85, 91, 95, 96 (mean = 86, RSD = 15%)	67, 97, 98, 106, 111 (mean = 96, RSD = 18%)
	0.10	50, 56, 81, 83, 83 (mean = 71, RSD = 23%)	53, 62, 80, 84, 86 (mean = 73, RSD = 20%)
Oranges	0.01	96, 102, 103, 105, 107 (mean = 103, RSD = 4.0%)	74, 76, 76, 78, 83 (mean = 77, RSD = 4.4%)
	0.10	110, 118, 119, 120, 120 (mean = 117, RSD = 3.6%)	76, 76, 79, 83, 84 (mean = 80, RSD = 4.8%)
Lettuce	0.01	78, 85, 96, 96, 98 (mean = 91, RSD = 9.6%)	95, 101, 105, 108, 110 (mean = 104, RSD = 5.8%)
	0.10	95, 100, 100, 105, 106 (mean = 101, RSD = 4.4%)	102, 103, 106, 107, 111 (mean = 106%, RSD = 3.4%)
Wheat grain	0.01	78, 92, 92, 95, 98 (mean = 91, RSD =	77, 82, 83, 84, 88 (mean = 83, RSD =

Matrix	Fortification level (mg/kg)	Dimethoate	Omethoate
		8.4%	4.8%)
	0.10	89, 91, 93, 94, 101 (mean = 94, RSD = 4.9%)	86, 88, 91, 92, 95 (mean = 90, RSD = 3.9%)

Again, very similar results were achieved using a confirmatory method with a different column. While all mean recoveries were within the range 70–110%, variability was higher for olive oil, with RSD values ranging from 15–23%.

LC-MS method (report number SCI/050)

This method was developed and validated for determination of dimethoate and omethoate residues in a range of plant matrices (Harper – 2001a).

Plant matrices were either obtained from local markets or previous residue studies. Ten gram samples were homogenised and fortified if required, then macerated with 50 mL dichloromethane and centrifuged. The extract was drained into a separate vessel and the sample was macerated with a second 50 mL aliquot of dichloromethane, centrifuged again and the two extracts combined and made up to 100 mL with dichloromethane. A 10 mL aliquot was evaporated to dryness then resuspended in 10 mL hexane, and shaken with 10 mL of water. The phases were allowed to separate, the hexane phase was discarded and the aqueous phase partitioned a second time with 10 mL hexane, which was again allowed to separate and the hexane phase discarded. The aqueous phase was diluted as necessary with water for analysis by LC-MS (m/z = 230 for dimethoate and 214 for omethoate). Matrix matching was not employed.

Good linearity was achieved ($r \geq 0.999$). An LOQ of 0.01 mg/kg was validated for both dimethoate and omethoate in all matrices, while the LOD was 0.002 mg/kg.

Table 42 Recoveries of dimethoate and omethoate from plant matrices by LC-MS method SCI/050 (Harper – 2001a)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	Omethoate recoveries (%)
Apple	0.01	84, 90, 96, 98, 106 (mean = 95, RSD = 9.1%)	77, 89, 92, 93, 104 (mean = 91, RSD = 11%)
	0.10	90, 91, 93, 102, 109 (mean = 97, RSD = 8.5%)	87, 89, 91, 98, 106 (mean = 94, RSD = 8.3%)
	1.0	100, 103, 105, 105, 105 (mean = 104, RSD = 2.1%)	70, 93, 94, 101, 102 (mean = 92, RSD = 14%)
Artichoke	0.01	89, 93, 98, 105, 107 (mean = 98, RSD = 7.8%)	102, 102, 104, 109, 109 (mean = 105, RSD = 3.4%)
	0.10	86, 88, 91, 96, 97 (mean = 92, RSD = 5.3%)	87, 89, 90, 94, 94 (mean = 91, RSD = 3.4%)
	1.0	94, 97, 97, 100, 100 (mean = 98, RSD = 2.6%)	88, 90, 92, 94, 101 (mean = 93, RSD = 5.4%)
Celery	0.01	87, 88, 88, 90, 91 (mean = 89, RSD = 1.9%)	97, 97, 102, 106, 106 (mean = 102, RSD = 4.4%)
	0.10	97, 98, 100, 107, 110 (mean = 102, RSD = 5.6%)	88, 89, 91, 108, 110 (mean = 97, RSD = 11%)
	1.0	95, 99, 100, 101, 108 (mean = 101, RSD = 4.7%)	79, 89, 90, 95, 96 (mean = 90, RSD = 7.5%)
Cherries	0.01	85, 88, 92, 92, 100 (mean = 91, RSD = 6.2%)	76, 85, 86, 86, 87 (mean = 84, RSD = 5.4%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	Omethoate recoveries (%)
	0.10	99, 102, 102, 104, 106 (mean = 103, RSD = 2.5%)	107, 108, 109, 110, 110 (mean = 109, RSD = 1.2%)
	1.0	96, 99, 104, 104, 105 (mean = 102, RSD = 3.8%)	70, 71, 73, 77, 88 (mean = 76, RSD = 9.7%)
Lettuce	0.01	103, 104, 105, 107, 109 (mean = 106, RSD = 2.3)	108, 108, 109, 109, 109 (mean = 109, RSD = 0.5%)
	0.10	96, 99, 100, 104, 106 (mean = 101, RSD = 4.0%)	88, 90, 92, 93, 96 (mean = 92, RSD = 3.3%)
	1.0	89, 91, 94, 97, 102 (mean = 95, RSD = 5.4%)	73, 74, 79, 83, 108 (mean = 83, RSD = 17%)
Tomatoes	0.01	95, 100, 103, 106, 108 (mean = 102, RSD = 5.0%)	91, 101, 102, 104, 105 (mean = 101, RSD = 5.6%)
	0.10	86, 89, 89, 90, 91 (mean = 89, RSD = 2.1%)	78, 78, 82, 86, 86 (mean = 82, RSD = 4.9%)
	1.0	96, 97, 97, 101, 102 (mean = 99, RSD = 2.7%)	80, 81, 81, 82, 83 (mean = 81, RSD = 1.4%)
Wheat grain	0.01	85, 93, 98, 99, 99 (mean = 95, RSD = 6.3%)	104, 107, 108, 108, 109 (mean = 107, RSD = 1.8%)
	0.10	71, 80, 80, 80, 83 (mean = 79, RSD = 5.8%)	81, 82, 91, 91, 94 (mean = 88, RSD = 6.7%)
	1.0	83, 93, 98, 100, 104 (mean = 96, RSD = 8.5%)	94, 104, 108, 109, 110 (mean = 105, RSD = 6.2%)
Wheat whole green plant	0.01	107, 107, 108, 109, 109 (mean = 108, RSD = 0.9%)	102, 104, 106, 107, 109 (mean = 106, RSD = 2.6%)
	0.10	75, 79, 80, 85, 86 (mean = 81, RSD = 5.6%)	85, 89, 90, 90, 91 (mean = 89, RSD = 2.6%)
	1.0	83, 84, 89, 90, 90 (mean = 87, RSD = 3.9%)	70, 71, 73, 75, 76 (mean = 73, RSD = 3.5%)
Wheat straw	0.01	98, 99, 101, 107, 109 (mean = 103, RSD = 4.8%)	93, 95, 99, 102, 104 (mean = 99, RSD = 4.7%)
	0.10	86, 87, 94, 95, 96 (mean = 92, RSD = 5.2%)	92, 92, 96, 98, 99 (mean = 95, RSD = 3.4%)
	1.0	73, 79, 81, 83, 96 (mean = 82, RSD = 10%)	71, 71, 75, 77, 102 (mean = 79, RSD = 16%)
Sugar beet tops	0.01	97, 98, 98, 99, 100 (mean = 98, RSD = 1.2%)	82, 84, 84, 84, 85 (mean = 84, RSD = 1.3%)
	0.10	92, 93, 94, 98, 98 (mean = 95, RSD = 2.7%)	86, 87, 88, 90, 91 (mean = 88, RSD = 2.3%)
	1.0	103, 105, 107, 108, 109 (mean = 106, RSD = 2.3%)	95, 95, 96, 98, 99 (mean = 97, RSD = 1.9%)
Sugar beet roots	0.01	102, 107, 108, 109, 110 (mean = 107, RSD = 2.9%)	100, 100, 104, 105, 105 (mean = 103, RSD = 2.5%)
	0.10	100, 101, 102, 104, 105 (mean = 102, RSD = 2.0%)	92, 93, 96, 96, 96 (mean = 95, RSD = 2.1%)
	1.0	92, 95, 96, 97, 102 (mean = 96, RSD = 3.8%)	84, 85, 87, 90, 91 (mean = 87, RSD = 3.5%)
Asparagus	0.01	97, 102, 105, 105, 107 (mean = 103, RSD = 3.8%)	99, 99, 100, 106, 110 (mean = 103, RSD = 4.8%)
	0.10	89, 91, 95, 110, 110 (mean = 99, RSD = 10%)	82, 83, 88, 96, 97 (mean = 89, RSD = 7.9%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	Omethoate recoveries (%)
	1.0	83, 85, 97, 98, 104 (mean = 9.7%)	87, 89, 96, 97, 98 (mean = 93, RSD = 5.4%)
Melon peel	0.01	93, 98, 99, 107, 108 (mean = 101, RSD = 6.3%)	103, 105, 106, 107, 109 (mean = 106, RSD = 2.1%)
	0.10	81, 84, 86, 86, 88 (mean = 85, RSD = 3.1%)	73, 75, 76, 77, 77 (mean = 76, RSD = 2.2%)
	1.0	86, 91, 94, 100, 102 (mean = 95, RSD = 6.9%)	80, 80, 83, 94, 90 (mean = 83, RSD = 4.9%)
Melon pulp	0.01	87, 93, 94, 97, 97 (mean = 94, RSD = 4.4%)	71, 71, 74, 74, 77 (mean = 73, RSD = 2.6%)
	0.10	101, 101, 103, 103, 106 (mean = 103, RSD = 2.0%)	72, 72, 73, 73, 102 (mean = 78, RSD = 17%)
	1.0	94, 96, 98, 98, 100 (mean = 97, RSD = 2.3%)	71, 71, 72, 77, 88 (mean = 76, RSD = 9.6%)

All mean recoveries and most individual recoveries were in the range 70–110%, and all RSD values were <20%. The method has been validated for analysis of dimethoate and omethoate in a wide range of plant samples over a concentration range of 0.01–1.0 mg/kg. Sample extracts were shown to be stable for at least 2 weeks storage at 4 °C in the dark.

QuEChERS method (P-14.141)

A QuEChERS-based multiresidue method was developed and validated for determination of dimethoate and omethoate residue in a wide range of plant matrices (Lindner – 2009a).

Homogenised samples (1 or 5 g) were weighed into centrifuge tubes and an appropriate amount of water added depended on the matrix, shaken and then acetonitrile was before a further 1 minute of hand shaking. Magnesium sulfate, sodium chloride, trisodium citrate dihydrate, and disodium hydrogen citrate were added and the centrifuge tube was shaken for a further minute, before centrifuging for 2 minutes (4000 rpm). Citrus pulp and whole fruit samples were pH adjusted to 5.0–5.5 with sodium hydroxide. Once the phases were separated an aliquot of the upper acetonitrile layer was transferred into a vial containing Primary-Secondary Amine (PSA) sorbent and magnesium sulfate, shaken for 1 minute, and centrifuged for 2 minute (6000 rpm). 0.3 mL of the upper (acetonitrile) phase were diluted to volume (2.5 mL) with dilute acetic acid.

Samples were analysed by LC-MS/MS (dimethoate transitions: 230 → 125 and 230 → 199, and omethoate transitions: 214 → 125 and 214 → 182). Matrix-matched standards were prepared for comparison with solvent standards to evaluate matrix effects. In practice, matrix effects were found to be insignificant (below ±10%).

Plant matrices were either obtained from local markets, or from previous residue studies (untreated control samples) and homogenised with either a knife mill or a cutter and dry ice. Recoveries samples were fortified with 0.001 or 0.01 mg/kg dimethoate and omethoate. Excellent linearity was achieved ($r \geq 0.9999$).

Table 43 Recoveries of dimethoate from plant matrices for QuEChERS method P-14.141

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%) 230 → 125	Dimethoate recoveries (%) 230 → 199
Wheat straw	0.001	75, 81, 82, 82, 83 (mean = 81, RSD = 4.0%)	79, 79, 82, 84, 84 (mean = 82 (RSD = 3.1%)
	0.01	81, 81, 84, 84, 90 (mean = 84, RSD = 4.4%)	79, 79, 85, 85, 89 (mean = 83, RSD = 5.2%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%) 230 → 125	Dimethoate recoveries (%) 230 → 199
Cabbage heads	0.001	86, 89, 89, 94, 96 (mean = 91, RSD = 4.5%)	90, 91, 91, 94, 95 (mean = 92, RSD = 2.4%)
	0.01	84, 87, 87, 87, 88 (mean = 87, RSD = 1.8%)	84, 85, 85, 86, 87 (mean = 86, RSD = 1.1%)
Lettuce	0.001	86, 86, 88, 91, 91 (mean = 88, RSD = 2.8%)	83, 86, 86, 89, 92 (mean = 87, RSD = 3.9%)
	0.01	84, 86, 86, 87, 87 (mean = 86, RSD = 1.4%)	84, 84, 86, 87, 88 (mean = 86, RSD = 2.1%)
Olive (without stones)	0.001	81, 82, 84, 85, 90 (mean = 84, RSD = 4.2%)	84, 84, 84, 92, 95 (mean = 88, RSD = 6.0%)
	0.01	78, 79, 81, 81, 83 (mean = 80, RSD = 2.4%)	78, 79, 80, 81, 83 (mean = 80, RSD = 2.4%)
Citrus (whole fruit)	0.001	68, 79, 83, 85, 89 (mean = 81, RSD = 9.9%)	75, 77, 83, 83, 85 (mean = 81, RSD = 5.4%)
	0.01	80, 81, 82, 82, 82 (mean = 81, RSD = 1.1%)	78, 79, 80, 80, 81 (mean = 80, RSD = 1.4%)
Citrus pulp	0.001	77, 77, 77, 81, 83 (mean = 79, RSD = 3.6%)	77, 79, 83, 86, 89 (mean = 83, RSD = 5.9%)
	0.01	81, 86, 87, 89, 92 (mean = 87, RSD = 4.7%)	80, 84, 84, 86, 88 (mean = 84, RSD = 3.5%)
Citrus peel	0.001	86, 89, 94 (mean = 90, RSD = 4.5%)	87, 88, 89 (mean = 88, RSD = 1.1%)
	0.01	86, 86, 88 (mean = 87, RSD = 1.3%)	87, 88, 90 (mean = 88, RSD = 1.7%)
Wheat grain	0.001	81, 81, 82, 85, 85 (mean = 83, RSD = 2.5%)	80, 83, 87, 89, 91 (mean = 86, RSD = 5.2%)
	0.01	82, 83, 87, 87, 87 (mean = 85, RSD = 2.9%)	83, 85, 85, 86, 88 (mean = 85, RSD = 2.1%)
Carrot roots	0.001	81, 82, 88, 91, 92 (mean = 87, RSD = 5.8%)	77, 81, 84, 85, 91 (mean = 84, RSD = 6.2%)
	0.01	80, 84, 85, 85, 86 (mean = 84, RSD = 2.8%)	83, 86, 86, 86, 88 (mean = 86, RSD = 2.1%)
Sugar beet roots	0.001	99, 99, 100, 101, 102 (mean = 100, RSD = 1.3%)	99, 102, 103, 106, 107 (mean = 103, RSD = 3.1%)
	0.01	90, 92, 92, 94, 95 (mean = 93, RSD = 2.1%)	93, 93, 94, 95, 95 (mean = 94, RSD = 1.1%)
Onion bulb	0.001	83, 86, 93, 95, 101 (mean = 92, RSD = 7.9%)	87, 89, 90, 90, 94 (mean = 90, RSD = 2.8%)
	0.01	83, 83, 83, 86, 88 (mean = 85, RSD = 2.7%)	83, 83, 85, 85, 85 (mean = 84, RSD = 1.3%)
Barley forage	0.001	105, 107, 107 (mean = 106, RSD = 1.1%)	99, 101, 104 (mean = 101, RSD = 2.5%)
	0.01	104, 105, 106 (mean = 105, RSD = 1.0%)	101, 104, 104 (mean = 103, RSD = 1.7%)
Cherries (without stones)	0.001	91, 91, 92 (mean = 91, RSD = 0.6%)	92, 94, 94 (mean = 93, RSD = 1.2%)
	0.01	93, 95, 97 (mean = 95, RSD = 2.1%)	94, 96, 98 (mean = 96, RSD = 2.1%)
Cauliflower heads	0.001	78, 85, 86 (mean = 83, RSD = 5.3%)	77, 83, 85 (mean = 82, RSD = 5.1%)
	0.01	81, 82, 86 (mean = 83, RSD = 3.2%)	80, 82, 84 (mean = 82, RSD = 2.4%)
Broccoli heads	0.001	86, 86, 88 (mean = 87, RSD = 1.3%)	81, 83, 88 (mean = 84, RSD = 4.3%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%) 230 → 125	Dimethoate recoveries (%) 230 → 199
	0.01	86, 86, 87 (mean = 86, RSD = 0.7%)	83, 86, 87 (mean = 85, RSD = 2.7%)
Brussels sprouts	0.001	87, 88, 99 (mean = 91, RSD = 7.3%)	84, 86, 91 (mean = 87, RSD = 4.1%)
	0.01	86, 88, 89 (mean = 88, RSD = 1.7%)	86, 87, 88 (mean = 87, RSD= 1.1%)

Table 44 Recoveries of omethoate from plant matrices for QuEChERS method P-14.141

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%) 214 → 125	Omethoate recoveries (%) 214 → 182
Wheat straw	0.001	69, 74, 76, 83, 83 (mean = 77, RSD = 7.8%)	72, 81, 85, 87, 91 (mean = 83, RSD = 8.7%)
	0.01	79, 80, 81, 82, 83 (mean = 81, RSD = 2.0%)	75, 78, 79, 86, 89 (mean = 81, RSD = 7.2%)
Cabbage heads	0.001	82, 82, 84, 86, 91 (mean = 85, RSD = 4.4%)	83, 83, 89, 90, 91 (mean = 87, RSD = 4.5%)
	0.01	78, 79, 79, 80, 83 (mean = 80, RSD = 2.4%)	79, 80, 81, 82, 85 (mean = 81, RSD= 2.8%)
Lettuce	0.001	81, 84, 86, 88, 89 (mean = 86, RSD = 3.7%)	83, 85, 85, 87, 88 (mean = 86, RSD = 2.3%)
	0.01	79, 81, 81, 84, 84 (mean = 82, RSD = 2.7%)	78, 81, 82, 85, 86 (mean = 82, RSD = 3.9%)
Olive (without stones)	0.001	73, 75, 83, 87, 88 (mean = 81, RSD = 8.5%)	74, 79, 81, 83, 83 (mean = 80, RSD = 4.7%)
	0.01	72, 75, 79, 80, 81 (mean = 77, RSD = 4.9%)	71, 72, 72, 75, 80 (mean = 774, RSD = 5.0%)
Citrus (whole fruit)	0.001	72, 74, 74, 77, 77 (mean = 75, RSD = 2.9%)	72, 73, 78, 81, 83 (mean = 77, RSD = 6.2%)
	0.01	69, 71, 72, 73, 73 (mean = 72, RSD = 2.3%)	70, 70, 73, 74, 77 (mean = 73, RSD = 4.1%)
Citrus pulp	0.001	74, 74, 77, 78, 83 (mean = 77, RSD = 4.8%)	69, 71, 71, 77, 78 (mean = 73, RSD = 5.5%)
	0.01	71, 74, 75, 78, 79 (mean = 75, RSD = 4.3%)	73, 74, 74, 75, 79 (mean = 775, RSD = 3.1%)
Citrus peel	0.001	81, 87, 100 (mean = 89, RSD = 11%)	89, 90, 91 (mean = 90, RSD = 1.1%)
	0.01	83, 84, 84 (mean = 84, RSD = 0.7%)	82, 86, 90 (mean = 86, RSD = 4.7%)
Wheat grain	0.001	74, 77, 84, 87, 91 (mean = 83, RSD = 8.5%)	67, 72, 72, 82, 86 (mean = 76, RSD = 10%)
	0.01	76, 76, 80, 81, 81 (mean = 79, RSD = 3.3%)	74, 79, 79, 80, 80 (mean = 78, RSD = 3.2%)
Carrot roots	0.001	71, 81, 87, 87, 88 (mean = 83, RSD = 8.6%)	81, 88, 89, 96, 99 (mean = 91, RSD = 7.8%)
	0.01	74, 77, 81, 85, 86 (mean = 81. RSD = 6.4%)	79, 79, 80, 81, 81 (mean = 80, RSD = 1.3%)
Sugar beet roots	0.001	99, 101, 102, 103, 104 (mean = 102, RSD = 1.9%)	80, 104, 109, 110, 133 (mean = 107, RSD = 18%)
	0.01	92, 92, 93, 94, 94 (mean = 93, RSD = 1.1%)	92, 92, 93, 95, 97 (mean = 94, RSD = 2.3%)
Onion bulb	0.001	81, 82, 90, 92, 103 (mean = 90, RSD = 9.9%)	72, 73, 74, 86, 89 (mean = 79, RSD = 10%)

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%) 214 → 125	Omethoate recoveries (%) 214 → 182
	0.01	74, 74, 75, 80, 81 (mean = 77, RSD = 4.5%)	72, 76, 78, 80, 82 (mean = 78, RSD = 5.0%)
Barley forage	0.001	99, 103, 103 (mean = 102, RSD = 2.3%)	106, 107, 109 (mean = 107, RSD = 1.4%)
	0.01	103, 105, 107 (mean = 105, RSD = 1.9%)	105, 107, 107 (mean = 106, RSD = 1.1%)
Cherries (without stones)	0.001	91, 93, 93 (mean = 92, RSD = 1.3%)	91, 96, 97 (mean = 95, RSD = 3.4%)
	0.01	92, 95, 99 (mean = 95, RSD= 3.7%)	95, 98, 99 (mean = 97, RSD = 2.1%)
Cauliflower heads	0.001	72, 73, 73 (mean = 73, RSD = 0.8%)	71, 76, 78 (mean = 75%, RSD = 4.8%)
	0.01	72, 77, 77 (mean = 75, RSD = 3.8%)	72, 76, 79 (mean = 76, RSD = 4.6%)
Broccoli heads	0.001	76, 77, 86 (mean = 80, RSD = 6.9%)	78, 87, 90 (mean = 85, RSD = 7.3%)
	0.01	70, 72, 78 (mean = 73, RSD = 5.7%)	70, 72, 73 (mean = 72, RSD = 2.1%)
Brussels sprouts	0.001	77, 84, 85 (mean = 82, RSD = 5.3%)	92, 96, 99 (mean = 96, RSD = 3.7%)
	0.01	86, 87, 92 (mean = 88, RSD = 3.6%)	86, 87, 90 (mean = 88, RSD = 2.4%)

Excellent recoveries were achieved, demonstrating validity of the method for analysis of dimethoate and omethoate in a wide range of plant matrices, covering low and high water content, high starch content, high acid, and high oil content matrices.

The QuEChERS method was independently validated by a second laboratory for analysis of dimethoate and omethoate in representative plant matrices (Garrigue – 2014a).

Samples of wheat grain, lettuce, olives, and orange (whole fruit), covering dry, high water content, high oil content, and high acid matrices were fortified with either dimethoate or omethoate standards at 0.001 (wheat grain only), 0.01, or 0.10 mg/kg.

No significant changes were made to the extraction, cleanup or instrumental phases of the method.

Table 45 Independent laboratory validation of QuEChERS method for dimethoate (Garrigue – 2014a)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%) 230 → 125	Dimethoate recoveries (%) 230 → 199
Wheat grain	0.001	102, 106, 106, 107, 108 (mean = 106, RSD = 2.2%)	110, 111, 114, 115, 121 (mean = 114, RSD = 3.8%)
	0.01	105, 105, 107, 109, 111 (mean = 107, RSD = 2.4%)	105, 105, 106, 106, 106 (mean = 106, RSD = 0.5%)
	0.10	101, 102, 103, 106, 111 (mean = 105, RSD = 3.9%)	100, 102, 102, 104, 106 (mean = 103, RSD = 2.2%)
Lettuce	0.01	114, 115, 115, 119, 122 (mean = 117, RSD = 2.9%)	111, 114, 115, 115, 115 (mean = 114, RSD = 1.5%)
	0.10	103, 105, 106, 106, 107 (mean = 105, RSD = 1.4%)	100, 102, 103, 105, 107 (mean = 103, RSD = 2.6%)
Olives	0.01	85, 90, 90, 97, 107 (mean = 94, RSD = 9.1%)	96, 97, 105, 106, 113 (mean = 103, RSD = 6.8%)
	0.10	98, 102, 103, 104, 107 (mean = 103, RSD = 3.2%)	101, 103, 103, 104, 105 (mean = 103, RSD = 1.4%)
Orange (whole fruit)	0.01	101, 101, 105, 107, 110 (mean = 105, RSD = 3.7%)	99, 101, 101, 105, 111 (mean = 103, RSD = 4.6%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%) 230 → 125	Dimethoate recoveries (%) 230 → 199
	0.10	101, 102, 102, 102, 103 (mean = 102, RSD = 0.7%)	101, 101, 101, 101, 103 (mean = 101, RSD = 0.9%)

Table 46 Independent laboratory validation of QuEChERS method for omethoate (Garrigue – 2014a)

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%) 214 → 125	Omethoate recoveries (%) 214 → 183
Wheat grain	0.001	92, 96, 97, 107, 109 (mean = 100, RSD = 7.4%)	106, 109, 110, 111, 113 (mean = 110, RSD = 2.4%)
	0.01	103, 104, 105, 105, 105 (mean = 104, RSD = 0.8%)	103, 103, 104, 107, 108 (mean = 105, RSD = 2.2%)
	0.10	98, 98, 100, 102, 103 (mean = 100, RSD = 2.3%)	92, 98, 98, 100, 103 (mean = 98, RSD = 4.1%)
Lettuce	0.01	118, 118, 119, 121, 123 (mean = 120, RSD = 1.8%)	117, 117, 118, 121, 124 (mean = 119, RSD = 2.6%)
	0.10	103, 103, 105, 106, 107 (mean = 105, RSD = 1.7%)	106, 107, 107, 108, 109 (mean = 107, RSD = 1.1%)
Olives	0.01	100, 104, 104, 106, 112 (mean = 105, RSD = 4.2%)	99, 101, 101, 102, 102 (mean = 101, RSD = 1.2%)
	0.10	97, 98, 100, 102, 104 (mean = 100, RSD = 2.9%)	103, 104, 104, 107, 108 (mean = 105, RSD = 2.1%)
Orange (whole fruit)	0.01	103, 104, 104, 105, 108 (mean = 105, RSD = 1.8%)	105, 106, 108, 110, 110 (mean = 108, RSD = 2.1%)
	0.10	94, 96, 99, 100, 100 (mean = 98, RSD = 2.7%)	97, 99, 100, 100, 101 (mean = 99, RSD = 1.5%)

As with the primary validation study, matrix effects were found to be insignificant, however matrix-matched standards were employed for quantification. The validated LOQ was 0.001 mg/kg for dimethoate and omethoate in wheat grain, and 0.01 mg/kg in olives, orange and lettuce. The LOD was 0.0003 mg/kg in wheat grain and 0.003 mg/kg in the other matrices. Sample extracts were shown to be stable for at least 9 days storage in a refrigerator. Method linearity was good ($r^2 > 0.99$). All mean recoveries were within the range 70–120%, and all RSD values were < 20%.

The extraction efficiency of the QuEChERS method was estimated (Sørensen – 2016a) by comparison of the extraction techniques and solvents used in the method, and those used in the potato (Cordon – 2000), wheat (Cordon-2001), and olive (Cordon – 2005) metabolism studies.

Table 47 Estimated extraction efficiency of the modified QuEChERS method for analysis of dimethoate and omethoate in plant matrices considered in metabolism studies

Matrix	Estimated extraction efficiency
Olive leaves (0DAA)	99.9%
Olives (28DALA)	80%
Wheat whole plant (14DAA)	83%
Ear and remaining plant (26 or 39 DAA)	91%
Grain/straw/hull (32DAA)	91%
Potato tubers (7-28 DAA)	88%

Estimated extraction efficiencies of the QuEChERS method were above 70% for a range of plant commodities, including high water, high starch, dry and high oil content matrices, supporting the suitability of the QuEChERS method as a monitoring method for dimethoate and omethoate residues.

Method AGR/MOA/DIMETHOATE-1 and Method AGR/MOA/DIMETHOATE-2

A validation study was conducted for analysis of olives for dimethoate, omethoate, and three further metabolites, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate (Amic – 2013).

Olive samples were homogenised without stones. For determination of recoveries, samples were separately fortified with the appropriate analyte at 0.01 or 0.10 mg/kg.

The first was a QuEChERS-based method for determination of dimethoate, omethoate, and dimethoate carboxylic acid. 5 g samples were extracted by shaking with 10 mL acetonitrile, followed by vortexing. Magnesium sulfate, sodium chloride, trisodium citrate dihydrate, and disodium hydrogen citrate sesquihydrate were added and shaken and vortexed. The sample was centrifuged for 5 minutes (3000 rpm) and an aliquot transferred to a centrifuge tube, to which PSA sorbent, C18EC, and magnesium sulfate were added, shaken, vortexed and centrifuged for 5 minutes (3000 rpm). An aliquot of the supernatant was diluted to volume with ultrapure water.

The second method, for determination of O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate involved extraction of samples by homogenisation with 1:1 v/v methanol/water, followed by centrifuging for 3 minutes (3500 rpm). An aliquot was diluted with dilute formic acid, then purified by solid phase extraction (Strata X-AW cartridge, washing with water and methanol, evaporation to dryness, elution with 99:1 v/v methanol/35% ammonia). The eluate was evaporated to dryness and reconstituted in dilute formic acid.

The extracts were analysed by LC-MS/MS, with some modification to parameters required for the different analytes. At least two transitions were monitored for each analyte.

Table 48 Recoveries of dimethoate, omethoate and dimethoate carboxylic acid from olives (minus stones)

Fortification level (mg/kg)	Dimethoate recoveries (%)		Omethoate recoveries (%)		Dimethoate carboxylic acid recoveries (%)	
	230 → 199	230 → 125	214 → 183	214 → 125	141 → 127	141 → 95
0.01	93, 95, 97, 100, 103 (mean = 98, RSD = 4.1%)	78, 87, 93, 95, 100 (mean = 91, RSD = 9.3%)	97, 97, 100, 104, 109 (mean = 101, RSD = 5.1%)	85, 89, 92, 94, 98 (mean = 92, RSD = 5.4%)	75, 80, 83, 84, 85 (mean = 81, RSD = 5.0%)	72, 81, 83, 95, 113 (mean = 89, RSD = 18%)
0.10	86, 87, 89, 94, 96 (mean = 90, RSD = 4.9%	83, 85, 88, 92, 96 (mean = 89, RSD = 5.9%)	90, 91, 91, 94, 96 (mean = 92, RSD = 2.7%)	91, 94, 95, 97, 98 (mean = 92, RSD = 2.9%)	74, 74, 75, 75, 78 (mean = 75, RSD = 2.2%)	70, 72, 72, 77, 86 (mean = 75, RSD = 8.6%)

Table 49 Recoveries of O-desmethyl omethoate carboxylic acid and O-desmethyl isodimethoate from olives minus stones

Fortification level (mg/kg	O-Desmethyl omethoate carboxylic acid recoveries (%)		O-desmethyl isodimethoate recoveries (%)	
	185 → 91	185 → 111	214 → 166	214 → 104
0.01	60, 66, 78, 80, 81 (mean = 73, RSD = 13%)	64, 64, 74, 77, 84 (mean = 73, RSD = 12%)	64, 65, 79, 80, 86 (mean = 75, RSD = 13%)	61, 67, 76, 81, 87 (mean = 74, RSD = 14%)
0.10	64, 67, 72, 75, 84 (mean = 72,	62, 66, 72, 74, 82	70, 71, 77, 80, 93	72, 73, 76, 80, 94

Fortification level (mg/kg	O-Desmethyl omethoate carboxylic acid recoveries (%)		O-desmethyl isodimethoate recoveries (%)	
	185 → 91	185 → 111	214 → 166	214 → 104
	RSD = 11%)	(mean = 71, RSD = 11%)	(mean = 78, RSD = 12%)	(mean = 79, RSD = 11%)

The method was also validated for the same analytes in wheat matrices (Amic – 2014a).

Table 50 Recoveries of dimethoate, omethoate and dimethoate carboxylic acid from wheat matrices

Fortification level (mg/kg)	Dimethoate recoveries (%)		Omethoate recoveries (%)		Dimethoate carboxylic acid recoveries (%)	
	230 → 199	230 → 125	214 → 183	214 → 125	141 → 127	141 → 95
Wheat grain						
0.01	89, 93, 96, 98, 99 (mean = 95, RSD = 4.3%)	89, 93, 94, 98, 103 (mean = 95, RSD = 5.6%)	88, 91, 93, 95, 97 (mean = 93, RSD = 3.8%)	83, 87, 89, 92, 94 (mean = 89, RSD = 4.8%)	86, 90, 90, 92, 96 (mean = 91, RSD = 4.0%)	78, 79, 83, 84, 91 (mean = 83, RSD = 6.2%)
0.10	88, 90, 90, 91, 92 (mean = 90, RSD = 1.6%)	89, 91, 94, 94, 96 (mean = 93, RSD = 3.0%)	84, 88, 89, 89, 90 (mean = 88, RSD = 2.7%)	81, 86, 89, 89, 90 (mean = 97, RSD = 4.2%)	90, 91, 92, 93, 94 (mean = 92, RSD = 1.7%)	88, 89, 94, 94, 94 (mean = 92, RSD = 3.3%)
Wheat straw						
0.01	93, 94, 99, 99, 99 (mean = 97, RSD = 3.1%)	89, 93, 98, 102, 102 (mean = 97, RSD = 5.9%)	86, 91, 94, 94, 96 (mean = 92, RSD = 4.2%)	80, 90, 92, 99, 101 (mean = 92, RSD = 9.0%)	91, 94, 95, 96, 98 (mean = 95, RSD = 2.7%)	75, 77, 82, 98, 102 (mean = 87, RSD = 14%)
0.10	73, 85, 86, 86, 88 (mean = 84, RSD = 7.2%)	77, 89, 89, 91, 92 (mean = 88, RSD = 6.9%)	75, 85, 85, 86, 86 (mean = 83, RSD = 5.7%)	72, 86, 90, 92, 96 (mean = 87, RSD = 11%)	88, 90, 91, 93, 95 (mean = 91, RSD = 3.0%)	89, 90, 90, 91, 95 (mean = 91, RSD = 2.6%)
Wheat whole plant						
0.01	97, 101, 105, 105, 105 (mean = 102, RSD = 3.3%)	99, 104, 107, 107, 108 (mean = 105, RSD = 3.5%)	80, 81, 83, 86, 93 (mean = 85, RSD = 6.2%)	83, 84, 86, 89, 93 (mean = 87, RSD = 4.7%)	92, 97, 98, 101, 103 (mean = 98, RSD = 4.3%)	84, 96, 99, 103, 113 (mean = 99, RSD = 11%)
0.10	97, 99, 99, 99, 100 (mean = 99, RSD = 1.1%)	102, 104, 105, 107, 109 (mean = 105, RSD = 2.6%)	83, 88, 90, 90, 93 (mean = 89, RSD = 4.2%)	90, 91, 94, 96, 98 (mean = 94, RSD = 3.6%)	85, 90, 90, 92, 93 (mean = 90, RSD = 3.4%)	86, 89, 90, 91, 92 (mean = 90, RSD = 2.6%)

Table 51 Recoveries of O-desmethyl omethoate carboxylic acid and O-desmethyl isodimethoate from wheat matrices

Fortification level (mg/kg	O-Desmethyl omethoate carboxylic acid recoveries (%)		O-desmethyl isodimethoate recoveries (%)	
	185 → 91	185 → 111	214 → 166	214 → 104
Wheat grain				
0.01	84, 86, 87, 90, 91 (mean = 88, RSD = 3.3%)	83, 84, 86, 91, 93 (mean = 87, RSD = 5.0%)	83, 83, 84, 84, 91 (mean = 85, RSD = 4.0%)	75, 80, 85, 88, 88 (mean = 83, RSD = 6.8%)
0.10	83, 85, 90, 90, 93 (mean = 88, RSD = 4.6%)	88, 89, 91, 93, 93 (mean = 91, RSD = 2.5%)	86, 87, 92, 93, 102 (mean = 92, RSD = 6.9%)	86, 90, 92, 95, 102 (mean = 93, RSD = 6.5%)
Wheat straw				

Fortification level (mg/kg	O-Desmethyl omethoate carboxylic acid recoveries (%)		O-desmethyl isodimethoate recoveries (%)	
	185 → 91	185 → 111	214 → 166	214 → 104
0.01	81, 82, 83, 86, 86 (mean = 84, RSD = 2.7%)	78, 81, 82, 85, 86 (mean = 82, RSD = 3.9%)	83, 84, 86, 87, 88 (mean = 86, RSD = 2.4%)	82, 84, 85, 85, 86 (mean = 84, RSD = 1.8%)
0.10	78, 79, 81, 81, 84 (mean = 81, 2.9%)	79, 79, 80, 81, 82 (mean = 80, RSD = 1.6%)	77, 78, 82, 84, 85 (mean = 81, RSD = 4.4%)	74, 76, 80, 81, 85 (mean = 89, RSD = 5.5%)
Wheat whole plant				
0.01	74, 79, 80, 84, 84 (mean = 80, RSD = 5.2%)	77, 79, 81, 83, 84 (mean = 81, RSD = 3.5%)	79, 80, 84, 84, 87 (mean = 83, RSD = 4.0%)	76, 81, 82, 83, 86 (mean = 82, RSD = 4.5%)
0.10	80, 82, 83, 85, 86 (mean = 83, RSD = 2.9%)	79, 82, 83, 84, 85 (mean = 83, RSD = 2.8%)	79, 81, 82, 83, 87 (mean = 82, RSD = 3.6%)	79, 81, 82, 83, 87 (mean = 82, RSD = 3.6%)

An LOQ of 0.01 mg/kg was achieved for all five analytes. Quantification was achieved using matrix-matched standards, as matrix effects were significant (>20%) for many combinations of analytes and matrices. Good linearity was achieved (r^2>0.99 for all analytes and transitions). All extracts were demonstrated to be stable for at least 7 days of storage in a refrigerator. Good accuracy and precision were achieved at the LOQ and 10 × LOQ for all five analytes, with all mean recoveries in the range 70–110% and all RSD <20%.

Method AGR/MOA/DIMETHOATE-1 was further extended (Method AGR/MOA/DIMETHOATE-2) and validated for the metabolites desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in olives and wheat matrices (Amic – 2014b and Amic – 2014c).

Samples were extracted and cleaned up as for O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate, using 1:1 v/v methanol/water, followed by centrifuging for 3 minutes (3500 rpm), dilution with dilute formic and clean up by solid phase extraction with a Strata X-AW cartridge (washing with water and methanol, evaporation to dryness, elution with 99:1 v/v methanol/35% ammonia). The eluate was evaporated to dryness and reconstituted in dilute formic acid.

The extracts were analysed by LC-MS/MS. Two transitions were monitored for each analyte, An LOQ of 0.01 mg/kg was achieved for the three additional analytes. Matrix matched standards were employed. Good linearity was achieved (r^2>0.99 for all analytes and transitions). Sample extracts were stable for 8 days refrigerated storage.

Table 52 Concurrent residue trial recoveries of desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate for olives (Amic – 2014b)

Fortification level (mg/kg)	O-desmethyl omethoate recoveries (%) 198 → 73	Desmethyl dimethoate recoveries (%) 214 → 104	O-desmethyl N-desmethyl omethoate recoveries (%) 184 → 90
0.01	80, 95, 101	77, 85, 97	85, 85, 88
0.10	86, 94	75, 96	87, 97
1.0	81	86	87

Table 53 Method validation recoveries for O-desmethyl omethoate from wheat and olive matrices (Amic – 2014c)

Matrix	Fortification level (mg/kg)	O-desmethyl omethoate recoveries (%)	
		198 → 73	198 → 79
Wheat grain	0.01	71, 72, 75, 78, 78 (mean = 75, RSD = 4.4%)	78, 82, 83, 86, 95 (mean = 85, RSD = 7.5%)
	0.10	76, 77, 80, 80, 80 (mean = 79, RSD = 2.5%)	73, 78, 79, 81, 81 (mean = 78, RSD = 4.2%)
Wheat straw	0.01	70, 72, 77, 79, 80 (mean = 76, RSD = 5.8%)	66, 69, 72, 91, 97 (mean = 79, RSD = 18%)
	0.10	82, 82, 83, 83, 86 (mean = 83, RSD = 2.0%)	82, 84, 85, 86, 86 (mean = 85, RSD = 2.0%)
Wheat whole plant	0.01	73, 76, 82, 83, 86 (mean = 80, RSD = 6.7%)	72, 79, 83, 85, 86 (mean = 81, RSD = 7.0%)
	0.10	77, 78, 79, 81, 82 (mean = 79, RSD = 2.6%)	79, 79, 81, 82, 82 (mean = 81, RSD = 1.9%)
Olives	0.01	72, 77, 77, 77, 81 (mean = 77, RSD = 4.2%)	66, 73, 77, 85, 88 (mean = 78, RSD = 11%)
	0.10	82, 86, 87, 87, 91 (mean = 87, RSD = 3.7%)	82, 83, 84, 90, 90 (mean = 86, RSD = 4.5%)

Table 54 Method validation recoveries for desmethyl dimethoate from wheat and olive matrices (Amic – 2014c)

Matrix	Fortification level (mg/kg)	Desmethyl dimethoate recoveries (%)	
		214 → 104	214 → 95
Wheat grain	0.01	83, 86, 89, 89, 96 (mean = 89, RSD = 5.4%)	83, 83, 90, 92, 93 (mean = 88, RSD = 5.5%)
	0.10	86, 87, 89, 95, 96 (mean = 91, RSD = 5.1%)	83, 91, 92, 92, 39 (mean = 91, RSD = 5.1%)
Wheat straw	0.01	81, 83, 84, 86, 93 (mean = 85, RSD = 5.4%)	79, 84, 90, 92, 93 (mean = 88, RSD = 6.8%)
	0.10	85, 86, 88, 94, 97 (mean = 90, RSD = 5.8%)	88, 89, 91, 94, 100 (mean = 92%, RSD = 5.2%)
Wheat whole plant	0.01	84, 84, 85, 90, 90 (mean = 87, RSD = 3.6%)	84, 84, 89, 91, 98 (mean = 89, RSD = 6.5%)
	0.10	85, 86, 87, 87, 88 (mean = 87, RSD = 1.3%)	87, 87, 90, 90, 95 (mean = 90, RSD = 3.6%)
Olives	0.01	84, 87, 89, 89, 94 (mean = = 89, RSD = 4.1%)	79, 81, 82, 86, 87 (mean = 83, RSD = 4.1%)
	0.10	80, 87, 87, 91, 94 (mean = 88, RSD = 6.0%)	73, 85, 86, 89, 93 (mean = 85, RSD = 8.8%)

Table 55 Method validation recoveries for O-desmethyl N-desmethyl omethoate from wheat and olive matrices (Amic – 2014c)

Matrix	Fortification level (mg/kg)	O-desmethyl N-desmethyl omethoate recoveries (%)	
		184 → 90	184 → 167
Wheat grain	0.01	75, 77, 79, 80, 88 (mean = 80, RSD = 6.2%)	76, 77, 77, 80, 83 (mean = 79, RSD = 3.7%)
	0.10	75, 76, 76, 78, 78 (mean = 77, RSD = 1.8%)	75, 77, 79, 81, 81 (mean = 79, RSD = 3.3%)
Wheat straw	0.01	83, 85, 89, 91, 93 (mean = 88, RSD = 4.7%)	75, 81, 82, 82, 85 (mean = 81, RSD = 4.5%)
	0.10	83, 85, 86, 87, 91 (mean = 86, RSD = 3.4%)	82, 82, 83, 83, 88 (mean = 84, RSD = 3.0%)
Wheat whole plant	0.01	79, 79, 82, 82, 86 (mean = 82, RSD = 3.5%)	78, 78, 82, 83, 84 (mean = 81, RSD = 3.5%)
	0.10	78, 78, 79, 80, 81 (mean = 79, RSD = 1.6%)	78, 79, 79, 80, 81 (mean = 79, RSD = 1.4%)
Olives	0.01	75, 80, 82, 83, 84 (mean = 81, RSD = 4.4%)	79, 80, 81, 91, 94 (mean = 85, RSD = 82.%)
	0.10	82, 84, 85, 91, 93 (mean = 87, RSD = 5.5%)	83, 83, 84, 90, 91 (mean = 86, RSD = 4.6%)

Good accuracy and precision were achieved at the LOQ and 10 × LOQ for all three analytes, with all mean recoveries in the range 70–110% and all RSD < 20%.

Finally, this suite of extraction and cleanup procedures and LC-MS/MS methods was validated for all eight analytes in sugar beet roots and tops (Amic – 2015a). Matrix effects were evident for dimethoate, omethoate, O-desmethyl isodimethoate, desmethyl dimehoate, and O-desmethyl N-desmethyl omethoate. As a result, both matrix matched and plain solvent standards were used for quantification as appropriate. Sample extracts were shown to be stable for at least 8 days of refrigerated storage.

Table 56 Method validation recoveries for dimethoate from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	
		230 → 199	230 → 125
Sugar beet roots	0.01	93, 96, 97, 100, 101 (mean = 97, RSD = 3.3%)	88, 91, 92, 94, 97 (mean = 92, RSD = 3.6%)
	0.10	91, 94, 96, 98, 101 (mean = 96, RSD = 4.0%)	90, 93, 96, 98, 99 (mean = 95, RSD = 3.9%)
Sugar beet tops	0.01	85, 85, 87, 95, 95 (mean = 89, RSD = 5.8%)	89, 90, 90, 91, 95 (mean = 91, RSD = 2.5%)
	0.10	83, 85, 85, 87, 91 (mean = 86, RSD = 3.5%)	80, 85, 86, 88, 92 (mean = 86, RSD = 5.1%)

Table 57 Method validation recoveries for omethoate from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%)	
		214 → 183	214 → 125
Sugar beet roots	0.01	87, 90, 94, 95, 97 (mean = 93, RSD = 4.4%)	88, 91, 91, 94, 97 (mean = 92, RSD = 3.7%)
	0.10	84, 85, 85, 87, 88 (mean = 86, RSD = 1.9%)	84, 85, 86, 86, 90 (mean = 86, RSD = 2.6%)
Sugar beet tops	0.01	83, 92, 96, 100, 106 (mean = 95, RSD = 9.1%)	90, 93, 103, 111, 111 (mean = 102, RSD = 9.7%)
	0.10	93, 94, 94, 95, 96 (mean = 94, RSD = 1.2%)	93, 93, 94, 96, 102 (mean = 96, RSD = 4.0%)

Table 58 Method validation recoveries for dimethoate carboxylic acid from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	Dimethoate carboxylic acid recoveries (%)	
		141 → 126	141 → 95
Sugar beet roots	0.01	61, 68, 71, 79, 82 (mean = 72, RSD = 12%)	63, 67, 70, 78, 86 (mean = 73, RSD = 13%)
	0.10	69, 74, 74, 77, 85 (mean = 76, RSD = 7.8%)	69, 74, 75, 77, 85 (mean = 76, RSD = 7.7%)
Sugar beet tops	0.01	67, 71, 76, 80, 81 (mean = 75, RSD = 7.9%)	65, 70, 75, 81, 84 (mean = 75, RSD = 10%)
	0.10	62, 70, 74, 76, 77 (mean = 72, RSD = 8.5%)	62, 68, 72, 76, 78 (mean = 71, RSD = 9.0%)

Table 59 Method validation recoveries for O-desmethyl omethoate carboxylic acid from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	O-Desmethyl omethoate carboxylic acid recoveries (%)	
		185 → 91	185 → 111
Sugar beet roots	0.01	69, 80, 80, 84, 90 (mean = 81, RSD = 9.5%)	80, 84, 87, 87, 92 (mean = 86, RSD = 5.1%)
	0.10	70, 78, 78, 79, 79 (mean = 77, RSD = 5.0%)	72, 78, 78, 79, 80 (mean = 77, RSD = 4.0%)
Sugar beet tops	0.01	71, 72, 74, 76, 82 (mean = 75, RSD = 5.8%)	66, 72, 77, 78, 81 (mean = 75, RSD = 7.9%)
	0.10	75, 76, 77, 77, 78 (mean = 77, RSD = 1.5%)	74, 75, 76, 77, 78 (mean = 76, RSD = 2.1%)

Table 60 Method validation recoveries for O-desmethyl isodimethoate from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	O-Desmethyl isodimethoate recoveries (%)	
		214 → 166	214 → 104
Sugar beet roots	0.01	83, 89, 90, 93, 94 (mean = 90, RSD = 4.8%)	83, 89, 90, 93, 95 (mean = 90, RSD = 5.1%)
	0.10	79, 87, 88, 89, 90 (mean = 87, RSD = 5.1%)	81, 87, 89, 90, 92 (mean = 88, RSD = 4.8%)

Matrix	Fortification level (mg/kg)	O-Desmethyl isodimethoate recoveries (%)	
		214 → 166	214 → 104
Sugar beet tops	0.01	103, 105, 106, 107, 114 (mean = 107, RSD = 3.9%)	101, 103, 105, 109, 119 (mean = 107, RSD = 6.6%)
	0.10	98, 99, 101, 103, 103 (mean = 101, RSD = 2.3%)	97, 97, 98, 101, 104 (mean = 99, RSD = 3.1%)

Table 61 Method validation recoveries for O-desmethyl omethoate from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	O-Desmethyl omethoate recoveries (%)	
		198 → 73	198 → 79
Sugar beet roots	0.01	76, 78, 86, 93, 99 (mean = 86, RSD = 11%)	61, 69, 72, 87, 88 (mean = 75, RSD = 16%)
	0.10	73, 80, 80, 80, 81 (mean = 79, RSD = 4.2%)	71, 72, 73, 74, 77 (mean = 73, RSD = 3.1%)
Sugar beet tops	0.01	76, 78, 79, 81, 94 (mean = 82, RSD = 8.8%)	77, 81, 90, 92, 95 (mean = 87, RSD = 8.8%)
	0.10	95, 96, 100, 100, 104 (mean = 99, RSD = 3.6%)	89, 90, 90, 93, 96 (mean = 92, RSD = 3.1%)

Table 62 Method validation recoveries for desmethyl dimethoate from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	Desmethyl dimethoate recoveries (%)	
		214 → 104	214 → 95
Sugar beet roots	0.01	71, 77, 77, 87, 87 (mean = 80, RSD = 8.8%)	72, 77, 80, 86, 88 (mean = 81, RSD = 8.1%)
	0.10	74, 81, 82, 82, 87 (mean = 81, RSD = 5.7%)	74, 79, 80, 81, 84 (mean = 80, RSD = 4.6%)
Sugar beet tops	0.01	87, 88, 89, 90, 91 (mean = 89, RSD = 1.8%)	89, 90, 91, 91, 98 (mean = 92, RSD = 3.9%)
	0.10	85, 86, 89, 90, 91 (mean = 88, RSD = 2.9%)	86, 89, 92, 92, 93 (mean = 90, RSD = 3.2%)

Table 63 Method validation recoveries for O-desmethyl N-desmethyl omethoate from sugar beet roots and tops (Amic – 2015a)

Matrix	Fortification level (mg/kg)	O-Desmethyl N-desmethyl omethoate recoveries (%)	
		184 → 90	184 → 167
Sugar beet roots	0.01	77, 82, 83, 84, 93 (mean = 84, RSD = 6.9%)	77, 78, 79, 84, 88 (mean = 81, RSD = 5.7%)
	0.10	69, 75, 76, 76, 77 (mean = 75, RSD = 4.3%)	68, 75, 76, 76, 78 (mean = 75, RSD = 5.2%)
Sugar beet tops	0.01	75, 75, 82, 82, 84 (mean = 80, RSD = 5.4%)	70, 77, 79, 84, 97 (mean = 81, RSD = 12%)
	0.10	73, 75, 76, 77, 78 (mean = 76, RSD = 2.5%)	75, 75, 77, 77, 79 (mean = 77, RSD = 2.2%)

Again, good accuracy and precision were achieved at the LOQ and 10 × LOQ for all eight analytes in sugar beet roots and tops, with all mean recoveries in the range 70–110% and all RSD <20%. Matrix effects were evident for dimethoate, omethoate, O-desmethyl isodimethoate, desmethyl dimehoate, and O-desmethyl N-desmethyl omethoate.

Animal matrices

A QuEChERS-based method was developed and validated for determination of dimethoate and omethoate residues in cattle liver, kidney, muscle, fat and milk and chicken eggs (Arndt – 2010).

For recovery determinations, 10 g milk samples and 5 g tissue and egg samples were fortified with dimethoate or omethoate at 0.001, 0.005, or 0.5 mg/kg.

Samples were extracted by hand shaking and/or sonication with acetonitrile, with water being added for fat samples. Magnesium sulfate and sodium chloride, and with the exception of egg samples, disodium and trisodium citrate as well, were added to create a partition; the separation into two phases was aided by centrifuging. An aliquot of the organic phase was cleaned up by dispersive solid phase extraction (PSA/ENVI-Carb sorbent/magnesium sulfate). The samples were centrifuged and the supernatant filtered and analysed by LC-MS/MS. The primary quantification transitions were 230 → 199 and 214 → 183 for dimethoate and omethoate respectively, with qualification transitions of 230 → 125 and 214 → 155 respectively.

Good linearity was achieved ($r > 0.99$ for all sample data sets). LOQs of 0.001 mg/kg were validated for both dimethoate and omethoate for all matrices, with LODs of 0.0005 mg/kg for tissues and eggs and 0.00025 mg/kg for milk. Matrix matching was not used.

Table 64 Method validation recoveries for dimethoate from animal matrices for a QuEChERS method (Arndt – 2010)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	
		230 → 199	230 → 125
Milk	0.001	97, 98, 99, 100, 100 (mean = 99, RSD = 1.3%)	100, 103, 103, 103, 107 (mean = 103, RSD = 2.4%)
	0.005	101, 101, 104, 105, 106 (mean = 103, RSD = 2.2%)	102, 104, 104, 104, 106 (mean = 104, RSD = 1.4%)
	0.5	100, 103, 104, 104, 105 (mean = 103, RSD = 1.9%)	102, 105, 106, 106, 106 (mean = 105, RSD = 1.6%)
Bovine liver	0.001	95, 96, 97, 98, 99 (mean = 97, RSD = 1.6%)	91, 94, 95, 97, 98 (mean = 95, RSD = 2.9%)
	0.005	95, 98, 99, 101 (mean = 98, RSD = 2.5%)	95, 98, 99, 99 (mean = 98, RSD = 1.9%)
	0.5	98, 100, 100, 100, 105 (mean = 101, RSD = 2.6%)	98, 98, 99, 100, 106 (mean = 100, RSD = 3.3%)
Bovine kidney	0.001	93, 94, 94, 96, 98 (mean = 95, RSD = 2.1%)	90, 90, 94, 95, 95 (mean = 93, RSD = 2.8%)
	0.005	100, 100, 100, 101, 102 (mean = 101, RSD = 1.1%)	99, 101, 102, 104, 104 (mean = 102, RSD = 2.1%)
	0.5	100, 100, 100, 102, 103 (mean = 101, RSD = 1.4%)	99, 100, 101, 101, 103 (mean = 101, RSD = 1.5%)
Bovine muscle	0.001	98, 100, 104, 106, 106 (mean = 103, RSD = 3.5%)	96, 97, 98, 102, 106 (mean = 100, RSD = 4.2%)
	0.005	100, 100, 101, 103, 104 (mean = 102, RSD = 1.8%)	102, 103, 107, 109, 110 (mean = 106, RSD = 3.4%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	
		230 → 199	230 → 125
	0.5	102, 103, 104, 105, 111 (mean = 105, RSD = 3.4%)	102, 103, 104, 104, 108 (mean = 104, RSD = 2.2%)
Bovine fat	0.001	86, 89, 92, 95, 100 (mean = 92, RSD = 5.9%)	89, 91, 97, 98, 100 (mean = 95, RSD = 5.0%)
	0.005	107, 110, 110, 110, 111 (mean = 110, RSD = 1.4%)	108, 110, 110, 110, 112 (mean = 110, RSD = 1.3%)
	0.5	102, 104, 104, 108, 109 (mean = 105, RSD = 2.8%)	103, 104, 104, 108, 109 (mean = 106, RSD = 2.6%)
Egg	0.001	90, 91, 93, 95, 95 (mean = 93, RSD = 2.5%)	80, 86, 89, 91, 93 (mean = 88, RSD = 5.8%)
	0.005	98, 100, 100, 103, 103 (mean = 101, RSD = 2.2%)	96, 99, 100, 101, 103 (mean = 100, RSD = 2.6%)
	0.5	98, 106, 108, 110, 110 (mean = 106, RSD = 4.7%)	100, 104, 108, 110, 110 (mean = 106, RSD = 4.1%)

Table 65 Method validation recoveries for omethoate from animal matrices for a QuEChERS method (Arndt – 2010)

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%)	
		214 → 183	214 → 155
Milk	0.001	91, 93, 93, 94, 96 (mean = 94, RSD = 1.9%)	85, 86, 87, 87, 87 (mean = 87, RSD = 1.3%)
	0.005	88, 92, 92, 93, 95 (mean = 92, RSD = 2.8%)	90, 92, 95, 96, 96 (mean = 94, RSD = 2.9%)
	0.5	93, 93, 95, 95, 96 (mean = 94, RSD = 1.4%)	93, 94, 95, 96, 97 (mean = 95, RSD = 1.7%)
Bovine liver	0.001	86, 86, 91, 94, 99 (mean = 91, RSD = 6.1%)	80, 83, 86, 91, 92 (mean = 86, RSD = 5.9%)
	0.005	82, 84, 84, 85 (mean = 84, RSD = 1.5%)	80, 83, 85, 85 (mean = 83, RSD = 2.8%)
	0.5	88, 89, 91, 91, 95 (mean = 91, RSD = 3.0%)	86, 88, 89, 91, 96 (mean = 90, RSD = 4.2%)
Bovine kidney	0.001	82, 83, 84, 89, 93 (mean = 86, RSD = 5.4%)	81, 85, 92, 94, 95 (mean = 89, RSD = 6.8%)
	0.005	91, 91, 92, 93, 95 (mean = 92, RSD = 1.8%)	89, 90, 93, 94, 96 (mean = 92, RSD = 3.1%)
	0.5	92, 92, 93, 93, 95 (mean = 93, RSD = 1.3%)	90, 91, 91, 92, 94 (mean = 92, RSD = 1.7%)
Bovine muscle	0.001	91, 94, 96, 97, 99 (mean = 95, RSD = 3.2%)	86, 86, 91, 92, 92 (mean = 89, RSD = 3.5%)
	0.005	91, 94, 96, 97, 98 (mean = 95, RSD = 2.9%)	89, 95, 97, 100, 101 (mean = 96, RSD = 5.0%)
	0.5	93, 96, 96, 98, 102 (mean = 96, RSD = 3.9%)	93, 95, 95, 97, 100 (mean = 96, RSD = 2.8%)
Bovine fat	0.001	96, 100, 101, 102, 106 (mean = 101, RSD = 3.2%)	92, 93, 94, 96, 100 (mean = 95, RSD = 3.3%)
	0.005	99, 102, 103, 103, 104 (mean =	101, 101, 102, 102, 102 (mean =

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%)	
		214 → 183	214 → 155
		102, RSD = 1.9%)	102, RSD =0.5%)
	0.5	100, 100, 101, 102, 102 (mean = 101, RSD = 1.0%)	98, 99, 99, 101, 102 (mean = 100, RSD =1.6%)
Egg	0.001	80, 89, 92, 92, 94 (mean = 89, RSD = 6.2%)	84, 84, 89, 92, 96 (mean = 89, RSD = 5.8%)
	0.005	92, 92, 93, 93, 97 (mean = 93, RSD = 2.2%)	91, 91, 93, 95, 96 (mean = 93, RSD = 2.4%)
	0.5	89, 94, 94, 98, 102 (mean = 95, RSD = 5.1%)	91, 95, 96, 96, 102 (mean = 96, RSD = 4.1%)

Excellent accuracy and precision was demonstrated for both dimethoate and omethoate in milk, eggs, and bovine liver, kidney, muscle and fat (all mean recoveries and most individual recoveries in the 70–110%, and all RSD values < 20%). The method is considered validated for analysis of dimethoate and omethoate in mammalian tissues, as well as milk and eggs.

The QuEChERS method was independently validated for determination of dimethoate and omethoate in milk, eggs and bovine tissues (Garrigue – 2014b). No significant changes were made to the method. Samples of bovine fat, muscle, liver and kidney, and milk and eggs were fortified with dimethoate or omethoate at 0.001, 0.01, or 0.10 mg/kg.

Table 66 Independent validation recoveries for dimethoate from animal matrices for a QuEChERS method (Garrigue – 2014b)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	
		230 → 199	230 → 125
Bovine fat	0.001	101, 102, 104, 106, 106 (mean = 104, RSD = 2.2%)	103, 104, 105, 109, 113 (mean = 107, RSD = 3.9%)
	0.01	90, 96, 96, 98, 101 (mean = 96, RSD = 4.2%)	90, 94, 97, 99, 100 (mean = 96, RSD = 4.2%)
	0.1	93, 95, 97, 99, 102 (mean = 97, RSD = 3.6%)	93, 96, 97, 99, 101 (mean = 97, RSD = 3.1%)
Bovine muscle	0.001	85, 95, 102, 105, 105 (mean = 98, RSD = 8.7%)	86, 96, 105, 108, 110 (mean = 101, RSD = 9.9%)
	0.01	105, 107, 108, 109, 109 (mean = 108, RSD = 1.6%)	108, 108, 108, 109, 110 (mean = 109, RSD = 0.8%)
	0.1	104, 109, 110, 110, 112 (mean = 109, RSD = 2.8%)	107, 108, 111, 112, 116 (mean = 111, RSD = 3.2%)
Bovine liver	0.001	100, 104, 105, 106, 107 (mean = 104, RSD = 2.6%)	100, 100, 101, 107, 109 (mean = 103, RSD = 4.1%)
	0.01	106, 107, 111, 111, 112 (mean = 109, RSD = 2.5%)	108, 111, 113, 113, 115 (mean = 112, RSD = 2.4%)
	0.1	104, 108, 110, 112, 112 (mean = 109, RSD = 3.1%)	106, 110, 112, 112, 113 (mean = 111, RSD = 2.5%)
Bovine kidney	0.001	93, 94, 96, 99, 105 (mean = 97, RSD = 5.0%)	94, 97, 98, 99, 104 (mean = 98, RSD = 3.7%)
	0.01	94, 103, 105, 105, 107 (mean = 103, RSD = 5.0%)	93, 101, 104, 105, 105 (mean = 102, RSD = 5.0%)
	0.1	95, 97, 98, 100, 100 (mean = 98, RSD = 2.2%)	93, 97, 97, 98, 99 (mean = 97, RSD = 2.4%)

Matrix	Fortification level (mg/kg)	Dimethoate recoveries (%)	
		230 → 199	230 → 125
Milk	0.001	105, 107, 108, 108, 108 (mean = 107, RSD = 1.2%)	105, 107, 107, 109, 111 (mean = 108, RSD = 2.1%)
	0.01	104, 108, 109, 109, 111 (mean = 108, RSD = 2.4%)	106, 108, 109, 109, 111 (mean = 109, RSD = 1.8%)
	0.1	106, 106, 107, 110, 111 (mean = 108, RSD = 2.2%)	106, 108, 109, 110, 111 (mean = 109, RSD = 1.8%)
Egg	0.001	102, 104, 104, 108, 109 (mean = 105, RSD = 2.8%)	102, 104, 104, 105, 111 (mean = 105, RSD = 3.3%)
	0.01	100, 102, 105, 105, 109 (mean = 104, RSD = 3.3%)	102, 103, 104, 106, 108 (mean = 105, RSD = 2.3%)
	0.1	101, 105, 105, 105, 105 (mean = 104, RSD = 1.7%)	103, 103, 105, 106, 107 (mean = 105, RSD = 1.7%)

Table 67 Method validation recoveries for omethoate from animal matrices for a QuEChERS method (Garrigue – 2014b)

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%)	
		214 → 183	214 → 155
Bovine fat	0.001	94, 98, 102, 102, 105 (mean = 100, RSD = 4.3%)	97, 98, 100, 101, 102 (mean = 100, RSD = 2.1%)
	0.01	92, 92, 94, 96, 99 (mean = 95, RSD = 3.1%)	89, 94, 94, 95, 99 (mean = 94, RSD = 3.8%)
	0.1	85, 88, 88, 92, 97 (mean = 90, RSD = 5.2%)	84, 87, 89, 91, 96 (mean = 89, RSD = 5.0%)
Bovine muscle	0.001	75, 89, 91, 93, 95 (mean = 89, RSD = 8.9%)	77, 86, 96, 97, 102 (mean = 92, RSD = 11%)
	0.01	100, 101, 101, 103, 104 (mean = 102, RSD = 1.6%)	100, 101, 103, 103, 116 (mean = 105, RSD = 6.2%)
	0.1	98, 106, 106, 107, 110 (mean = 105, RSD = 4.2%)	100, 107, 108, 108, 109 (mean = 106, RSD = 3.4%)
Bovine liver	0.001	95, 95, 97, 97, 98 (mean = 96, RSD = 1.4%)	96, 99, 99, 100, 100 (mean = 99, RSD = 1.7%)
	0.01	99, 100, 103, 103, 106 (mean = 102, RSD = 2.7%)	99, 101, 102, 104, 106 (mean = 102, RSD = 2.6%)
	0.1	98, 100, 101, 103, 106 (mean = 102, RSD = 3.0%)	99, 100, 102, 102, 103 (mean = 101, RSD = 1.6%)
Bovine kidney	0.001	85, 89, 92, 98, 99 (mean = 93, RSD = 6.4%)	86, 95, 97, 97, 99 (mean = 95, RSD = 5.4%)
	0.01	89, 94, 95, 95, 96 (mean = 94, RSD = 3.0%)	87, 93, 93, 95, 96 (mean = 93, RSD = 3.8%)
	0.1	89, 91, 91, 94, 97 (mean = 92, RSD = 3.4%)	89, 90, 91, 92, 93 (mean = 91, RSD = 1.7%)
Milk	0.001	92, 92, 96, 97, 98 (mean = 95, RSD = 3.0%)	83, 88, 91, 91, 98 (mean = 90, RSD = 6.0%)
	0.01	96, 100, 101, 102, 103 (mean = 100, RSD = 2.7%)	95, 100, 100, 101, 103 (mean = 100, RSD = 3.0%)
	0.1	96, 98, 99, 100, 105 (mean =	95, 96, 98, 101, 105 (mean = 99,

Matrix	Fortification level (mg/kg)	Omethoate recoveries (%)	
		214 → 183	214 → 155
		100, RSD = 3.4%)	RSD = 4.1%)
Egg	0.001	89, 89, 89, 93, 97 (mean = 90, RSD = 3.9%)	84, 87, 89, 91, 94 (mean = 89, RSD = 4.3%)
	0.01	87, 91, 91, 93, 97 (mean = 92, RSD = 4.0%)	87, 89, 91, 91, 94 (mean = 90, RSD = 2.9%)
	0.1	89, 90, 90, 91, 93 (mean = 91, RSD = 1.7%)	90, 90, 90, 91, 91 (mean = 90, RSD = 0.6%)

Again, excellent accuracy and precision was demonstrated for both dimethoate and omethoate in milk, eggs, and bovine liver, kidney, muscle and fat (all mean recoveries and individual recoveries in the 70–120%, and all RSD values < 20%). The method was successfully independently validated (with an LOQ of 0.001 mg/kg for both dimethoate and omethoate) in mammalian tissues, as well as milk and eggs. Sample extracts of kidney, muscle, fat, eggs and milk were determined to be stable for at least 7 dayys of freezer storage (-20 °C). Liver extracts were not stable for this period however and should be analysed within 24 hours of extraction.

The extraction efficiency of the modified QuEChERS method was discussed (Sørensen – 2016b). By comparison of the extraction methods and solvents of the QuEChERS method with those used in the goat and hen metabolism studies (Jalali – 1995a and Jalali – 1995b), and noting the fractions of radioactivity extracted, the expected extraction efficiency of the QuEChERS method was estimated.

Table 68 Estimated extraction efficiency of the modified QuEChERS method for analysis of dimethoate and omethoate in animal tissues, milk and eggs

Matrix	Estimated extraction efficiency
Liver	46%
Kidney	63%
Muscle	56%
Fat	58%
Milk	90%
Eggs	74%

The estimated extraction efficiencies are above 70% of the total incurred residue for milk and eggs. Although the levels of extraction for tissues are lower, it is noted that recoveries of dimethoate and omethoate were well within the range of 70–120% for both the primary and independent validation of the QuEChERS method.

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

Plant matrices

The stability of dimethoate and omethoate residues in cherries over 6 months frozen storage was investigated (Harper – 2001b). Homogenised cherry samples were fortified with either dimethoate or omethoate at 0.1 mg/kg and stored deep frozen (-18 °C). Control samples were frozen for determination of concurrent recoveries. Samples were extracted with dichloromethane, evaporated to dryness, cleaned up by partitioning between hexane and water, and the aqueous phase taken for analysis by LC-MS.

Table 69 Stability of dimethoate and omethoate residues in frozen cherry samples (Harper – 2001)

Time interval (months)	Dimethoate		Omethoate	
	Storage recovery (%)	Concurrent recovery (%)	Storage recovery (%)	Concurrent recovery (%)
0	104, 104	-	94, 95	-
6	103, 106	95, 97	84, 92	93, 96

Residues of dimethoate and omethoate were observed to be stable for at least 6 months frozen storage.

The stability of dimethoate and omethoate residues in olives over 12 months frozen storage was investigated (Lindner – 2009b). Homogenised green olive flesh samples were fortified with either dimethoate or omethoate at 0.1 mg/kg and stored deep frozen (-18 °C). Samples were withdrawn from storage at intervals of 1, 3, 6 and 12 months and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile, followed by cleanup by liquid-liquid partition by addition of sodium chloride, magnesium sulfate and sodium citrate, and dispersive solid phase extraction (primary-secondary amine (PSA) sorbent and magnesium sulfate). Cleaned up extracts were analysed using LC-MS/MS. Concurrent recoveries were determined at each sampling interval using freshly fortified olive samples.

Table 70 Stability of dimethoate and omethoate residues in frozen olive samples (Lindner – 2009)

Time interval (months)	Dimethoate		Omethoate	
	Storage recovery (%)	Concurrent recovery (%)	Storage recovery (%)	Concurrent recovery (%)
0	86, 98, 99 (94)	-	91, 93, 103 (96)	-
1	87, 90	90	81, 84	89
3	89, 92	101	84, 88	95
6	90, 98	96	77, 77	77
12	76, 77	79	70, 73	74

Acceptable concurrent recoveries were achieved for both dimethoate and omethoate at all time intervals. Recoveries of dimethoate and omethoate from stored olive samples were within 70–110% at all time intervals, and were consistent with the concurrent recoveries, indicating that residues of dimethoate and omethoate are stable in olive samples for at least 12 months when stored deep frozen (-18 °C).

As part of a residue study, freezer storage stability of dimethoate residues in dry peas was investigated (Samoil-2002). Homogenised dry pea seed samples were fortified with dimethoate at 5 mg/kg and stored frozen (-18 °C) for 462 days (15 months). Samples were extracted with 5:95 v/v ethanol/ethyl acetate, extracts were dried with sodium sulfate, and cleaned up using 4:1 Celite/charcoal columns prior to analysis by GC-FPD (operating in phosphorus mode).

Table 71 Stability of dimethoate residues in frozen dry pea samples (Samoil – 2002)

Time interval (months)	Dimethoate	
	Storage recovery (%)	Concurrent recovery (%)
15	71, 77, 82 (76)	1 mg/kg fortification: 81, 82 6 mg/kg fortification: 87, 87

Based on this data, residues of dimethoate are stable in deep frozen samples of dry peas for at least 15 months.

Freezer storage stability of dimethoate residues in podded fresh peas was investigated as part of a field residue study (Samoil – 2000). Homogenised pea seed plus pod samples were fortified with dimethoate at 5 mg/kg and stored frozen (-18 °C) for 739 days (24 months). Samples were extracted with 5:95 v/v ethanol/ethyl acetate, extracts were dried with sodium sulfate, and cleaned up using 4:1 Celite/charcoal columns prior to analysis by GC-FPD (operating in phosphorus mode).

Table 72 Stability of dimethoate residues in frozen podded pea samples (Samoil – 2000)

Time interval (months)	Dimethoate	
	Storage recovery (%)	Concurrent recovery (%)
24	82, 84, 89 (85)	0.06 mg/kg fortification: 95, 99 5 mg/kg fortification: 93, 96

Based on this data, residues of dimethoate are stable in deep frozen samples of fresh peas with pods for at least 24 months.

The stability of dimethoate and omethoate residues in a range of representative raw agricultural commodities was investigated over a period of 27 months (Williams – 1994 and two subsequent amendments extending the storage periods). The commodities selected were potato tuber, orange whole fruit, sorghum grain, sorghum forage, and cottonseed, representing high water content, high water/high acid content, dry non-oily, and high oil content matrices. Homogenised samples were fortified with either dimethoate at 1 mg/kg or omethoate at 0.5 mg/kg in frozen storage (-20 to -10 °C) for up to 27 months, with samples withdrawn for analysis at intervals of 1, 2, 4, 6, 20, and 27 month intervals. All matrices except cottonseed were extracted with acetone (with the addition of water in the case of sorghum grain), filtered, and partitioned with dichloromethane with the addition of sodium chloride to aid the partitioning of water. The organic phase was dried and further cleaned up by column chromatography (Celite/charcoal 4:1 v/v). Cottonseed samples were placed in Soxhlet thimbles with Celite and Soxhlet extracted for 8–24 hours with ethyl acetate at a cycle rate of 4–5 cycles per hour. The extracts were evaporated to oily residue and made up to volume with 15:85 v/v dichloromethane/cyclohexane then cleaned up using gel permeation chromatography. All extracts were analysed by GC-FDP operating in phosphorus mode. At each sampling interval, duplicate stored samples and a single freshly fortified control sample were analysed.

Table 73 Stability of dimethoate and omethoate residues on frozen storage in a range of plant commodities (Williams – 1994)

Matrix	Storage interval (days)	Dimethoate		Omethoate	
		Stored recoveries (%)	Concurrent recoveries (%)	Stored recoveries (%)	Concurrent recoveries (%)
Potato	0	85, 92, 94	-	84, 84, 93	-
	39	77, 81	87	76, 78	83
	70	91, 92	96	91, 92	95
	137	85, 86	95	84, 89	95
	188	88, 89	94	88, 89	93
	620	76, 80	88	87, 87	93
	799	83, 86	96	83, 86	94
Orange	0	79, 81, 84	-	81, 83, 84	-
	39	87, 87	93	80, 88	89
	70	95, 110	97	97, 99	94

Matrix	Storage interval (days)	Dimethoate		Omethoate	
		Stored recoveries (%)	Concurrent recoveries (%)	Stored recoveries (%)	Concurrent recoveries (%)
	137	86, 91	99	98, 102	101
	188	95, 95	97	102, 103	102
	620	88, 92	89	107, 112	100
	799	89, 95	98	92, 97	95
Sorghum grain	0	85, 88, 91	-	83, 85, 91	-
	34	86, 88	85	33, 84	75
	67	72, 74	91	86, 90	97
	137	89, 90	94	84, 88	94
	185	92, 98	97	92, 93	97
	620	77, 79	100	68, 71	106
	801	87, 90	96	83, 83	92
Sorghum forage	0	73, 74, 76	-	81, 85, 87	-
	36	73, 79	85	73, 74	71
	69	94, 98	101	97, 98	104
	139	78, 81	86	80, 81	91
	187	82, 84	86	86, 86	86
	622	65, 72	94	84, 96	103
	803	63, 63	84	67, 72	81
Cottonseed	82	93, 93	97	44, 50	53
	126	88, 91	95	46, 52	62
	189	87, 87	98	48, 53	67
	623	91, 92	103	78, 82	101
	804	92, 93	103	61, 62	81

Residues of dimethoate and omethoate are, based on the data in this study, stable for at least 27 months in deep frozen sorghum seed, orange, cottonseed, and potato. In sorghum forage, some degradation of dimethoate is noticeable, particularly at the 27 month time point and to a lesser extent at 20 months. Both stored and concurrent recoveries of omethoate from cottonseed were poor at the 3-6 month intervals, but were consistent between stored and freshly fortified samples. Better recoveries were achieved after 20 and 27 months due to a modification to the extraction process for cottonseed.

The stability of the dimethoate metabolites dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in olive matrices was investigated (Amic – 2014d). Homogenised olive samples (minus stones) were separately fortified with one of the metabolites at 0.10 mg/kg and stored at -20 °C. Samples were withdrawn at intervals up to 12 months and analysed alongside freshly fortified samples of defrosted control homogenised olive flesh using method AGR/MOA/DIMETHOATE-1 (Amic-2013).

Table 74 Frozen storage stability of dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in olives (Amic – 2014d)

Time interval (months)	Dimethoate carboxylic acid		O-desmethyl omethoate carboxylic acid		O-desmethyl isodimethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	77, 83, 94	-	73, 84, 80	-	97, 105, 113	-
1	82, 117	85, 87	99, 105	85, 110	62, 86	72, 85
3	73, 74	76, 79	79, 81	82, 85	81, 87	81, 83
6	91, 94	90, 91	55, 59	75, 77	67, 68	73, 74
8	86, 88	86, 88	49, 53	77, 87	62, 64	91, 93
12	68, 68	78, 82	48, 62	95, 97	81, 87	79, 87

Dimethoate carboxylic acid and O-desmethyl isodimethoate were observed to be stable in olives samples on frozen storage for at least 12 months while O-desmethyl omethoate carboxylic acid was stable for 3-6 months.

The stability of the dimethoate metabolites dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in wheat whole plant, grain and straw was investigated (Amic – 2015b). Homogenised samples were separately fortified with one of the metabolites at 0.10 mg/kg and stored at -20 °C. Samples were withdrawn at intervals up to 12 months and analysed alongside freshly fortified samples of defrosted control samples using method AGR/MOA/DIMETHOATE-1 (Amic-2013).

Table 75 Frozen storage stability of dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in wheat whole plant (Amic – 2015b)

Time interval (months)	Dimethoate carboxylic acid		O-desmethyl omethoate carboxylic acid		O-desmethyl isodimethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	86, 88, 89	-	80, 82, 83	-	84, 85, 86	-
1	75, 75	79, 81	99, 107	105, 105	82, 93	99, 103
3	91, 92	91, 97	84, 97	90, 103	85, 100	92, 103
6	88, 89	91, 92	27, 29	78, 78	32, 36	74, 77
12	82, 82	88, 111	19, 19	81, 81	23, 24	87, 90

Table 76 Frozen storage stability of dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in wheat grain (Amic – 2015b)

Time interval (months)	Dimethoate carboxylic acid		O-desmethyl omethoate carboxylic acid		O-desmethyl isodimethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	89, 89, 90	-	82, 83, 83	-	83, 84, 88	-
1	81, 85	88, 89	94, 94	99, 102	69, 79	74, 78
3	90, 91	88, 90	94, 97	84, 97	90, 98	84, 99
6	87, 91	85, 86	40, 46	75, 77	74, 76	79, 81
12	85, 95	84, 88	26, 37	73, 78	71, 79	89, 89

Table 77 Frozen storage stability of dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in wheat straw (Amic – 2015b)

Time interval (months)	Dimethoate carboxylic acid		O-desmethyl omethoate carboxylic acid		O-desmethyl isodimethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	85, 87, 93	-	85, 85, 86	-	83, 83, 83	-
1	71, 72	77, 84	81, 85	83, 96	70, 84	82, 85
3	90, 92	91, 92	95, 96	90, 91	84, 98	93, 94
6	89, 95	90, 96	70, 73	97, 105	76, 93	96, 97
12	92, 96	91, 93	103, 113	87, 88	76, 85	91, 95

Dimethoate carboxylic acid was stable in wheat whole plant, grain and straw for at least 12 months frozen storage, O-desmethyl omethoate carboxylic acid was stable for 3 months in wheat whole plant and grain, and 12 months in straw, while O-desmethyl isodimethoate was stable for 3 months in whole plant, and 12 months in grain and straw.

Stability of O-desmethyl omethoate, desmethyl dimethoate, and O-desmethyl N-desmethyl omethoate in wheat grain and olive matrices was investigated (Amic – 2015c). Homogenised samples were fortified separately with one of the metabolites at 0.10 mg/kg and at -20 °C. Samples were withdrawn at intervals up to 12 months and analysed alongside freshly fortified samples of defrosted control homogenised olive flesh or wheat grain using method AGR/MOA/DIMETHOATE-2 (Amic – 2014b and Amic – 2014c).

Table 78 Frozen storage stability of O-desmethyl omethoate, desmethyl dimethoate, and O-desmethyl N-desmethyl omethoate in wheat grain (Amic – 2015c)

Time interval (months)	O-desmethyl omethoate		Desmethyl dimethoate		O-desmethyl N-desmethyl omethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	77, 79, 80	-	108, 111, 112	-	95, 96, 103	-
1	85, 87	90, 90	86, 91	87, 88	98, 102	90, 93
3	91, 91	93, 95	102	97, 103	43, 44	93, 95
3.3	-	-	-	-	21, 22	80, 81
6	72, 73	89, 90	61, 61	79, 82	28, 30	79, 79
12	74, 88	60, 63	70, 73	87, 91	37, 38	57, 61
14	77, 81	69, 70	85, 86	76, 78	31, 41	71, 76

Table 79 Frozen storage stability of O-desmethyl omethoate, desmethyl dimethoate, and O-desmethyl N-desmethyl omethoate in olives (Amic – 2015c)

Time interval (months)	O-desmethyl omethoate		Desmethyl dimethoate		O-desmethyl N-desmethyl omethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	75, 77, 80	-	92, 94, 97	-	88, 92, 92	-
1	90, 90	92, 93	87, 91	92, 93	94, 102	91, 92

Time interval (months)	O-desmethyl omethoate		Desmethyl dimethoate		O-desmethyl N-desmethyl omethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
3	90, 92	92, 94	95, 96	98, 102	42, 43	92, 96
3.3	-	-	-	-	29, 30	80, 80
6	58, 60	80, 81	51, 53	74, 78	26, 29	71, 74
12	32, 40	83, 86	39, 40	78, 82	18, 20	85, 87

O-desmethyl omethoate residues were observed to be stable on frozen storage for 14 months in wheat grain, and 3-6 months in olives. Desmethyl dimethoate residues were stable for 14 months in wheat grain and 3 months in olives, while O-desmethyl N-desmethyl residues were stable for only 1 month of frozen storage in both olives and wheat grain.

An additional study for O-desmethyl N-desmethyl omethoate in olives and wheat whole plant, grain and straw was conducted over 8 weeks frozen storage (Amic – 2015d).

Table 80 Frozen storage stability of O-desmethyl N-desmethyl omethoate in olives and wheat matrices (Amic – 2015d)

Time interval (weeks)	Wheat whole plant		Wheat grain		Wheat straw		Olives	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	80, 86, 88	-	77, 77, 81	-	76, 80, 81	-	80, 81, 85	-
2	71, 75	76, 77	19, 27	75, 76	66, 73	76, 77	89, 90	78, 78
4	47, 51	74, 75	29, 37	63, 66	61, 69	80, 81	69, 75	72, 84
5	48, 49	88, 92	35, 44	80, 82	62, 63	94, 96	68, 75	98, 99
6	42, 43	78, 79	29, 30	67, 78	58, 64	73, 79	65, 68	67, 67
7	32, 42	89, 93	21, 23	67, 76	50, 51	87, 88	66, 66	90, 96
8	39, 43	89, 90	25, 32	93, 95	56, 61	89, 90	78, 97	86, 87

O-Desmethyl N-desmethyl omethoate residues were observed to be stable on frozen storage for 8 weeks in olives, between 4 and 8 weeks in wheat straw, and 2 weeks in wheat whole plant. Residues were not stable in wheat grain.

The frozen storage stability of O-desmethyl omethoate and desmethyl dimethoate in wheat straw was investigated over a 6-month storage period (Amic – 2015e).

Table 81 Frozen storage stability of O-desmethyl omethoate and desmethyl dimethoate in olives and wheat matrices (Amic – 2015e)

Time interval	O-desmethyl omethoate		Desmethyl dimethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	79, 81, 81	-	102, 102, 103	-
2 weeks	60, 71	101, 102	83, 87	94, 97
4 weeks	59, 75	103, 107	93, 95	105, 106
6 weeks	55, 60	85, 87	78, 84	90, 93

Time interval	O-desmethyl omethoate		Desmethyl dimethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
8 weeks	50, 53	93, 97	72, 74	94, 97
3 months	47, 50	80, 86	72, 80	75, 76
6 months	40, 41	103, 106	97, 98	104, 104

Residues of desmethyl dimethoate were stable in wheat straw on freezer storage for at least 6 months, while residues of O-desmethyl omethoate were only stable for 4-6 weeks.

Finally, stability of dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, O-desmethyl omethoate, desmethyl dimethoate, and O-desmethyl N-desmethyl omethoate residues in sugar beet roots and tops was investigated over 12 months frozen storage (Amic – 2015f).

Homogenised samples were fortified separately with one of the compounds at 0.10 mg/kg and at -20 °C. Samples were withdrawn at intervals up to 12 months and analysed alongside freshly fortified samples of defrosted control homogenised sugar beet root or tops using methods AGR/MOA/DIMETHOATE-1 and AGR/MOA/DIMETHOATE-2 (Amic – 2013, Amic – 2014b and Amic – 2014c).

Residues of dimethoate, omethoate, and dimethoate carboxylic acid in sugar beet roots and tops are stable to frozen storage for at least 12 months. Residues of O-desmethyl omethoate carboxylic acid in sugar beet roots were stable for 1 month of frozen storage, but were not stable in sugar beet tops. Residues of O-desmethyl isodimethoate were stable in sugar beet roots for at 4 months, and in sugar beet tops for 2 months. Residues of O-desmethyl omethoate were stable in sugar beet roots for 2 months, but were not stable sugar beet tops. Residues of desmethyl dimethoate were stable in sugar beet roots for 12 months, but were not shown to be stable in sugar beet tops. Residues of O-desmethyl N-desmethyl omethoate were not stable in either sugar beet roots or tops.

Table 82 Stability of residues of dimethoate, omethoate and six other metabolites in sugar beet roots on frozen storage (Amic – 2015f)

Time interval (months)	Dimethoate		Omethoate		Dimethoate carboxylic acid		O-desmethyl omethoate carboxylic acid		O-desmethyl isodimethoate		O-desmethyl omethoate		Desmethyl dimethoate		O-desmethyl N-desmethyl omethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	84, 84, 89	-	87, 88, 91	-	77, 80, 87	-	78, 79, 80	-	89, 90, 90	-	73, 74, 79	-	97, 98, 106	-	77, 78, 80	-
1	102, 108	79, 86	64, 64	77, 77	64, 64	78, 78	64, 70	108, 120	85, 89	97, 101	85, 102	127, 132	76, 100	104, 107	46, 47	93, 96
2	129, 130	108, 109	83, 87	93, 95	109, 114	100, 105	25, 26	80, 84	78, 78	89, 94	51, 71	93, 94	69, 69	55, 57	29, 33	83, 87
3	-	-	-	-	-	-	-	-	-	-	-	-	65, 71	50, 68	-	-
3.5	-	-	-	-	-	-	32, 36	72, 85	71, 71	79, 82	-	-	73, 77	83, 90	-	-
4	111, 112	108, 108	64, 68	90, 92	76, 89	97, 108	29, 30	91, 92	74, 76	90, 91	25, 28	93, 96	93, 105	81, 89	17, 25	90, 90
6	124, 125	99, 102	67, 71	88, 91	96, 103	84, 86	26, 30	77, 83	51, 53	81, 85	12, 25	89, 92	72, 74	44, 72	< 10, 16	85, 88
12	90, 97	92, 97	63, 64	92, 97	78, 79	71, 72	< 10, < 10	85, 87	64, 65	85, 87	< 10, 22	92, 93	65, 88	78, 82	< 10, < 10	86, 86

Table 83 Stability of residues of dimethoate, omethoate and six other metabolites in sugar beet tops on frozen storage (Amic – 2015f)

Time interval (months)	Dimethoate		Omethoate		Dimethoate carboxylic acid		O-desmethyl omethoate carboxylic acid		O-desmethyl isodimethoate		O-desmethyl omethoate		Desmethyl dimethoate		O-desmethyl N-desmethyl omethoate	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	94, 98, 104	-	85, 92, 98	-	97, 102, 103	-	88, 90, 93	-	82, 92, 93	-	79, 81, 86	-	93, 98, 105	-	79, 83, 84	-
1	93, 97	92, 104	83, 88	95, 110	66, 68	73, 90	32, 36	55, 57	55, 66	61, 63	10, 15	59, 60	62, 65	52, 56	< 10, < 10	54, 54
1.5	-	-	-	-	-	-	33, 45	79, 86	65, 66	83, 85	14, 15	80, 81	52, 55	82, 86	< 10, 15	78, 83
2	99, 105	105, 106	67, 77	97, 99	74, 78	80, 81	29, 32	69, 76	73, 74	90, 96	< 10, < 10	71, 80	68, 108	70, 81	< 10, < 10	69, 78

4	105, 106	100, 102	81, 85	88, 89	64, 69	66, 67	12, 13	101, 102	33, 47	73, 75	< 10, < 10	92, 92	18, 54	74, 77	< 10, < 10	86, 89
6	101, 108	98, 99	65, 73	87, 93	91, 100	88, 94	< 10, < 10	85, 89	26, 38	90, 98	< 10, < 10	84, 87	60, 63	62, 75	< 10, < 10	74, 78
12	119, 127	80, 85	64, 78	80, 84	78, 79	69, 70	< 10, < 10	84, 85	13, 13	90, 99	< 10, < 10	78, 79	35, 35	75, 90	< 10, < 10	90, 91

Plant extracts

The stability of cleaned up final extracts of plant samples fortified with dimethoate or omethoate on storage in a refrigerator (~4 °C) was investigated as part of an analytical method validation study (Harper – 2001a). The samples were extracted after homogenisation using dichloromethane. The extract was evaporated to dryness, reconstituted in hexane and partitioned into water before analysis by LC-MS. Extracts were either analysed immediately or stored for 14 days in a refrigerator before analysis.

Table 84 Stability of dimethoate and omethoate residues in plant matrix extract on refrigerated storage (Harper – 2001)

Matrix	Storage time (days)	Dimethoate		Omethoate	
		Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
Apple	0	93, 94	-	89, 91	-
	14	86, 88	93, 97	90, 92	92, 93
Artichoke	0	88, 94	-	91, 92	-
	14	84, 91	84, 85	90, 93	91, 92
Celery	0	88, 91	-	90, 92	-
	14	92, 92	88, 90	90, 92	94, 94
Cherry	0	88, 89	-	92, 94	-
	14	87, 92	90, 95	91, 92	92, 93
Lettuce	0	90, 92	-	91, 92	-
	14	92, 92	90, 92	90, 91	90, 91
Tomato	0	83, 86	-	83, 84	-
	14	83, 85	88, 88	82, 84	83, 88
Wheat grain	0	90, 90	-	98, 99	-
	14	98, 101	95, 98	100, 100	96, 98
Wheat whole green plant	0	85, 94	-	103, 107	-
	14	83, 92	85, 85	99, 99	97, 99
Wheat straw	0	99, 100	-	98, 101	-
	14	100, 103	97, 102	95, 95	92, 93
Sugar beet roots	0	88, 88	-	90, 91	-
	14	82, 85	80, 81	83, 85	83, 85
Sugar beet tops	0	89, 94	-	90, 93	-
	14	83, 85	83, 84	93, 98	84, 95
Asparagus	0	92, 92	-	90, 94	-
	14	87, 89	88, 88	91, 92	89, 92
Melon peel	0	87, 89	-	90, 92	-
	14	89, 91	86, 87	87, 90	89, 91
Melon pulp	0	93, 96	-	91, 93	-
	14	88, 89	87, 88	89, 89	87, 87

Sample extracts for dimethoate and omethoate residues extracted from a wide range of plant matrices are stable for at least 14 days of refrigerated storage.

Animal matrices

The stability of dimethoate and omethoate in frozen (-18 °C) bovine liver, kidney, muscle, and fat, milk, and eggs was investigated over 12-14 months (Arndt – 2012a). Matrices were homogenised and fortified with either dimethoate or omethoate at 0.05 mg/kg and frozen. Triplicate treated samples were withdrawn for analysis at each sampling interval, along with duplicate control samples for fortification and determination of concurrent recoveries. Samples were analysed using a QuEChERS based method (PTRL study no. 2080W), involving extraction with acetonitrile, with the addition of water in the case of fat, with cleanup by partitioning (by addition of salts to create an organic/aqueous partition) and dispersive solid phase extraction, followed by LC-MS/MS analysis (LOQ = 0.001 mg/kg).

Table 85 Storage stability of dimethoate residues in animal matrices (Arndt – 2012a)

Matrix	Time interval (days)	Storage recovery (%)		Concurrent recovery (%)	
		230 → 199	230 → 125	230 → 199	230 → 125
Milk	0	85, 88, 96 (90)	94, 99, 99 (97)	-	-
	14	99, 104, 105 (103)	92, 95, 96 (94)	96, 112 (104)	88, 105 (97)
	30	96, 108, 110 (105)	98, 108, 109 (105)	100, 110 (105)	107, 109 (108)
	59	74, 101, 102 (92)	96, 99, 113 (103)	92, 109 (10)	112, 113 (113)
	91	81, 88, 88 (86)	85, 87, 87 (86)	95, 96 (96)	92, 92 (92)
	189	97, 97, 100 (98)	99, 101, 101 (100)	105, 107 (106)	108, 110 (109)
	275	96, 101, 113 (103)	98, 109, 109 (103)	101, 108 (105)	102, 108 (105)
	367	96, 102, 103 (100)	96, 104, 105 (102)	83, 97 (90)	81, 93 (87)
Liver	0	99, 101, 105 (102)	100, 105, 105 (103)	-	-
	7	96, 97, 98 (97)	95, 98, 98 (97)	100, 107 (104)	103, 103 (103)
	15	94, 98, 98 (97)	96, 97, 99 (97)	100, 101 (101)	100, 100 (100)
	30	91, 95, 97 (94)	93, 98, 100 (97)	98, 105 (102)	98, 107 (103)
	59	93, 96, 98 (96)	94, 96, 97 (96)	103, 106 (105)	104, 106 (105)
	87	86, 87, 87 (87)	89, 89, 90 (89)	104, 111 (108)	105, 109 (107)
	177	88, 90, 98 (92)	88, 90, 95 (91)	104, 106 (105)	104, 104 (104)
	428	63, 69, 70 (67)	63, 69, 72 (68)	90, 92 (91)	90, 93 (92)
Kidney	0	103, 105, 107 (105)	107, 107, 108 (107)	-	-
	7	88, 91, 98 (92)	83, 86, 96 (92)	93, 96 (95)	91, 96 (94)
	14	91, 93, 96 (93)	91, 94, 96 (94)	105, 109 (107)	107, 108 (108)
	30	83, 90, 95 (89)	81, 86, 95 (87)	102, 103 (103)	96, 100 (98)
	69	63, 69, 72 (68)	61, 72, 77 (70)	102, 102 (102)	104, 108 (106)
	90	66, 72, 78 (72)	67, 74, 80 (74)	105, 108 (107)	106, 109 (108)
	177	60, 74, 75 (70)	60, 73, 79 (71)	98, 103 (101)	99, 104 (102)
	427	41, 47, 65 (51)	41, 47, 64 (51)	94, 95 (95)	94, 96 (95)

Matrix	Time interval (days)	Storage recovery (%)		Concurrent recovery (%)	
		230 → 199	230 → 125	230 → 199	230 → 125
Muscle	0	106, 107, 108 (107)	106, 108, 114 (109)	-	-
	14	95, 97, 98 (97)	96, 96, 98 (97)	104, 104 (104)	104, 105 (105)
	30	90, 102, 103 (98)	97, 100, 100 (99)	94, 94 (94)	102, 104 (103)
	69	86, 87, 92 (88)	84, 88, 90 (87)	93, 99 (96)	96, 101 (99)
	90	89, 97, 100 (95)	91, 99, 100 (97)	102, 109 (106)	105, 108 (107)
	177	97, 98, 99 (98)	99, 100, 102 (100)	104, 108 (106)	102, 108 (105)
	281	98, 99, 100 (99)	101, 104, 105 (103)	101, 103 (102)	104, 105 (105)
	363	94, 97 (96)	95, 95 (95)	103, 106 (105)	103, 108 (106)
Fat	0	103, 104, 107 (105)	106, 106, 107 (106)	-	-
	16	100, 100, 105 (102)	98, 100, 106 (101)	106, 112 (109)	104, 105 (105)
	31	101, 103, 107 (104)	100, 100, 106 (102)	95, 108 (102)	100, 107 (104)
	60	101, 101, 103 (102)	99, 101, 103 (101)	105, 110 (108)	102, 108 (105)
	88	95, 99, 102 (99)	95, 98, 100 (98)	101, 107 (104)	102, 108 (105)
	178	102, 109, 111 (107)	103, 106, 107 (105)	107, 112 (110)	106, 109 (108)
	283	94, 98, 102 (98)	96, 97, 104 (99)	96, 97 (97)	100, 101 (101)
	365	90, 91, 94 (92)	89, 91, 92 (91)	104, 107 (106)	104, 106 (105)
Egg	0	92, 92, 102 (95)	91, 97, 102 (97)	-	-
	14	96, 103, 106 (102)	101, 101, 104 (102)	017, 109 (108)	103, 107 (105)
	30	99, 103, 104 (102)	98, 101, 105 (101)	107, 109 (108)	109, 110 (110)
	58	80, 90, 96 (89)	95, 95, 95 (95)	81, 86 (84)	99, 100 (100)
	90	87, 93, 95 (92)	89, 95, 95 (93)	95, 96 (96)	95, 99 (97)
	188	89, 92, 93 (91)	90, 92, 94 (92)	101, 102 (102)	100, 101 (101)
	274	90, 94, 95 (93)	93, 95, 95 (94)	96, 104 (100)	99, 106 (103)
	366	81, 91, 93 (88)	84, 96, 99 (93)	97, 101 (99)	100, 110 (105)

Table 86 Storage stability of omethoate residues in animal matrices (Arndt – 2012a)

Matrix	Time interval (days)	Storage recovery (%)		Concurrent recovery (%)	
		214 → 183	214 → 155	214 → 183	214 → 155
Milk	0	87, 87, 90 (88)	86, 88, 92 (89)	-	-
	14	99, 99, 99 (99)	98, 99, 100 (99)	91, 98 (95)	93, 97 (95)
	30	91, 91, 94 (92)	91, 92, 94 (92)	102, 105 (104)	100, 103 (102)
	59	88, 93, 96 (92)	87, 97, 98 (94)	107, 108 (108)	105, 106 (106)
	91	75, 82, 83 (80)	77, 84, 84 (82)	87, 88 (88)	91, 92 (92)

Matrix	Time interval (days)	Storage recovery (%)		Concurrent recovery (%)	
		214 → 183	214 → 155	214 → 183	214 → 155
	189	82, 83, 84 (83)	82, 83, 83 (83)	95, 95 (95)	94, 96, (95)
	275	82, 87, 88 (86)	84, 87, 88 (86)	97, 104 (101)	96, 101 (99)
	367	80, 85, 85 (83)	80, 83, 84 (82)	80, 81 (81)	84, 85 (85)
Liver	0	94, 95, 96 (95)	94, 96, 98 (96)	-	-
	7	69, 76, 77 (74)	70, 77, 79 (75)	94, 99 (97)	94, 99 (97)
	15	62, 64, 65 (64)	60, 64, 65 (63)	87, 91 (89)	90, 92 (91)
	30	36, 43, 44 (41)	36, 43, 45 (41)	89, 96 (93)	91, 94 (93)
	59	26, 28, 30 (28)	26, 28, 32 (29)	96, 99 (98)	97, 97 (97)
	87	16, 16, 18 (17)	16, 16, 16 (16)	100, 102 (101)	99, 102 (101)
	177	10, 16, 17 (14)	10, 16, 17 (14)	90, 91 (91)	90, 91 (91)
	428	0, 2, 4 (2)	0, 2, 4 (2)	84, 84 (84)	85, 85 (85)
Kidney	0	96, 97, 99 (97)	97, 97, 10 (98)	-	-
	7	56, 56, 60 (57)	53, 55, 61 (56)	91, 94 (93)	87, 93 (90)
	14	40, 42, 47 (43)	40, 42, 49 (44)	96, 104 (100)	98, 103 (101)
	30	17, 24, 28 (23)	17, 25, 28 (23)	96, 97 (97)	94, 96 (95)
	69	4, 4, 10 (6)	3, 4, 10 (6)	86, 86 (86)	86, 89 (88)
	90	3, 4, 6 (4)	3, 4, 6 (4)	98, 98 (98)	95, 99 (97)
	177	1, 1, 8 (3)	1, 1, 8 (3)	87, 91 (89)	88, 92 (90)
	427	0, 0, 0 (0)	0, 0, 0 (0)	83, 83, (83)	84, 84 (84)
Muscle	0	97, 98, 99 (98)	97, 98, 98 (98)	-	-
	14	81, 83, 85 (83)	78, 83, 87 (83)	94, 98 (96)	93, 97 (95)
	30	88, 88, 88 (88)	86, 88, 88 (87)	94, 94 (94)	93, 94 (94)
	69	76, 78, 79 (78)	77, 78, 80 (78)	87, 88 (88)	85, 89 (87)
	90	83, 84, 90 (86)	81, 88, 88 (86)	99, 101 (100)	98, 100 (99)
	177	74, 75, 80 (76)	75, 77, 78 (77)	94, 95 (95)	92, 95 (94)
	281	72, 73, 74 (73)	72, 72, 73 (72)	89, 90 (90)	88, 93 (91)
	363	63, 66, 68 (66)	64, 66, 68 (66)	89, 92 (91)	90, 95 (93)
Fat	0	95, 98, 101 (98)	96, 98, 101 (98)	-	-
	16	92, 99, 100 (97)	95, 101, 104 (101)	93, 104 (99)	94, 107 (101)
	31	94, 96, 100 (97)	95, 96, 101 (97)	96, 100 (98)	93, 100 (97)
	60	94, 94, 96 (95)	93, 95, 95 (94)	100, 101 (101)	101, 104 (103)
	88	91, 92, 96 (93)	90, 91, 94 (92)	99, 105 (102)	101, 105 (103)
	178	84, 89, 91 (88)	84, 88, 89 (87)	101, 104 (103)	98, 100 (99)
	283	77, 78, 78 (78)	77, 80, 80 (79)	89, 90 (90)	90, 93 (92)
	365	73, 75, 76 (75)	75, 76, 77 (76)	92, 94 (93)	93, 96 (95)
Egg	0	85, 88, 97 (90)	79, 83, 90 (84)	-	-
	14	90, 91, 92 (91)	84, 86, 89 (86)	95, 97 (96)	93, 93 (93)
	30	84, 85, 89 (86)	85, 86, 89 (87)	100, 101 (101)	102, 102 (102)
	58	76, 81, 82 (80)	77, 79, 82 (79)	90, 94 (92)	89, 95 (92)
	90	64, 67, 70 (67)	66, 69, 69 (68)	86, 88 (87)	85, 86 (86)

Matrix	Time interval (days)	Storage recovery (%)		Concurrent recovery (%)	
		214 → 183	214 → 155	214 → 183	214 → 155
	188	56, 59, 60 (58)	56, 58, 62 (59)	83, 84 (84)	82, 82 (82)
	274	54, 56, 64, (61)	57, 61, 63 (60)	91, 98 (95)	93, 98 (96)
	366	47, 50, 54 (50)	47, 48 (53 (49)	86, 100 (93)	88, 94 (91)

Acceptable concurrent recoveries were achieved at all time points for both dimethoate and omethoate.

Residues of dimethoate were stable for at least 12 months in milk, muscle, fat and egg, while stability was more limited in liver and kidney. In liver, dimethoate was stable for at least 6 months, with reduced mean recoveries of 67–68% at 14 months. In kidney, dimethoate residues were stable for 6 months, with marked declines to only around 51% mean recovery at 14 months.

Residues of omethoate were stable for at least 12 months in milk and fat. Lesser periods of stability were observed for omethoate in all other matrices. In muscle, residues were stable for 9 months, with a noticeable decline thereafter (mean recoveries at 66% at 12 months). In egg, recoveries remained acceptable for at least 2 months, with a steady decline noted and mean recoveries reaching only 49–50% at 12 months. In liver and kidney, omethoate stability was poor, with mean recoveries declining below 70% by 2 weeks storage for liver and even by 1 week in kidney, and dropping to essentially zero by 14 months.

USE PATTERNS

Current labels for dimethoate products registered in Australia, Brazil, the Czech Republic, Estonia, Greece and the USA were provided to the Meeting. Relevant critical GAPs are tabulated below.

Table 87 GAP table

Crop	Country	Formulation	Application						Comments
			Method	No. (RTI – days)	Rate (kg ai/ha)	Conc. (kg ai/100 L)	Spray volume (L/ha)	PHI (days)	
Citrus fruit									
Citrus fruit (including oranges, lemons, mandarins, limes) (except Meyer lemons, Seville oranges and kumquats)	Australia	400 g/L EC	Foliar	3 (14)	-	0.03	Apply to the point of run-off.	7	
Citrus fruit (except edible peel crops, e.g. kumquats)	Australia	400 g/L EC	Post-harvest dip or flood spray (ensure fruit remains wet for at least 1 minute)	1	-	0.04 kg ai/100 L (400 mg ai/L)		Not required.	DO NOT use on fruit that has received pre-harvest treatment with dimethoate.
Citrus crops	Brazil	400 g/L EC	Foliar	Apply when infestation reaches economic		0.04	1000-1500	3	.

Crop	Country	Formulation	Application						Comments
			Method	No. (RTI – days)	Rate (kg ai/ha)	Conc. (kg ai/100 L)	Spray volume (L/ha)	PHI (days)	
				levels of damage.					
Citrus (orange, lemon, mandarin, grapefruit)	Greece	400 g/L EC	Foliar	2	0.48-0.576	0.04-0.048	1200	Apply first spray at end of bloom and the second at 40% of final fruit size	
Citrus (grapefruit, lemon, orange, tangerine)	USA	480 g/L EC	Foliar	1	1.12	-	-	15	
Stone fruit									
Cherries (sweet and sour)	Czech Republic	400 g/L EC	Foliar	1	0.4			28	
Cherries	USA	480 g/L EC	Foliar	1	1.49	-	467	21	
Tropical fruit, inedible peel									
Avocados	Australia	400 g/L EC	Foliar	Apply as pest populations indicate.	-	0.03		7	product per hectare and adjust the spray concentration accordingly.
Mangoes	Australia	400 g/L EC	Foliar	As required	-	0.03		3	
Avocados, Kiwifruit (inedible peel varieties only), Lychees, Custard apple, Mangoes, Pawpaw, Passion fruit	Australia	400 g/L EC	Post-harvest dipping	1		0.04 kg ai/100 L (400 mg ai/L)		Not required	Dip for 1 minute and allow to drain before packing
Tropical fruit, edible peel									
Olives	Greece	400 g/L EC	Bait spraying	1	0.05	0.25	20	28 days	Apply once per season as a bait spray with 0.65 L/100 L protein hydrolysate. Apply 0.2 L of spraying liquid per tree on 10 trees per stremma (100 trees per hectare). Can be combined with

Crop	Country	Formulation	Application						Comments
			Method	No. (RTI – days)	Rate (kg ai/ha)	Conc. (kg ai/100 L)	Spray volume (L/ha)	PHI (days)	
									a foliar spray with an interval of at least 10 days between applications.
Olives	Greece	400 g/L EC	Foliar	2 (14)	0.384-0.48	0.032-0.04	1200	28 days	
Bulb vegetables									
Onions	Australia	400 g/L EC	Foliar	As required	0.3	0.03	-	7 days	
Onions	Czech Republic	400 g/L EC	Foliar	2	0.24			14 days	
Onions	Greece	400 g/L EC	Foliar	2 (10)	0.20-0.24	0.067-0.08	300	14 days	
Brassica vegetables									
Broccoli	Czech Republic	400 g/L EC	Foliar	2	0.24			21 days	
Broccoli	Estonia	400 g/L EC	Foliar	2 (7)	0.24	-	200-400	21 days	
Broccoli	USA	480 g/L EC	Foliar	3 (7)	0.28-0.56	-	-	7 days	
Brussels sprouts	Czech Republic	400 g/L EC	Foliar	2	0.24			21 days	
Brussels sprouts	USA	480 g/L EC	Foliar	3 (7)	0.28-0.56	-	-	10 days	
Cauliflower	Czech Republic	400 g/L EC	Foliar	2	0.24			21 days	
Cauliflower	Estonia	400 g/L EC	Foliar	2 (7)	0.24	-	200-400	21 days	
Cauliflower	USA	480 g/L EC	Foliar	3 (7)	0.28-0.56	-	-	7 days	
Cabbage	Czech Republic	400 g/L EC	Foliar	Not specified	0.24			14 days	
Cabbage	Estonia	400 g/L EC	Foliar	2 (10)	0.24	-	200-400	14 days	
Fruiting vegetables, Cucurbits									
Melons	Australia	400 g/L EC	Foliar	As required	0.3	0.03	-	7 days	
Melons, water melons, squash	Greece	400 g/L EC	Foliar	2 (10)	0.20-0.24	0.067-0.08	300	Not required	Apply before flowering
Melons (except watermelons)	USA	480 g/L EC	Foliar	2 (7)	0.56	-	-	3 days	
Watermelons	USA	480 g/L EC	Foliar	2 (7)	0.28-0.56	-	-	3 days	
Fruiting vegetables, other than Cucurbits									
Capsicum	Australia	400 g/L EC	Foliar	As required	0.3	0.03	-	3 days	
Capsicum	USA	480 g/L EC	Foliar	5 (7)	0.28-0.37	-	-	0 days	
Tomatoes	Australia	400 g/L EC	Foliar	2 (14)	0.3	0.03		21 days	DO NOT use in

Crop	Country	Formulation	Application						Comments
			Method	No. (RTI – days)	Rate (kg ai/ha)	Conc. (kg ai/100 L)	Spray volume (L/ha)	PHI (days)	
(processing)									protected crops.
Tomatoes (fresh)	Australia	400 g/L EC	Foliar	Not stated		0.024		Not required	DO NOT apply after commencement of flowering.
Tomato	Brazil	400 g/L EC	Foliar	As required	-	0.04	400-700	14 days	
Tomato, eggplant (field grown)	Greece	400 g/L EC	Foliar	2 (14)	0.20-0.24	0.067-0.08	300	21 days	
Tomato	USA	480 g/L EC	Foliar	2 (6)	0.28-0.56	-	-	7 days	
Leafy vegetables									
Lettuce	Czech Republic	400 g/L EC	Foliar	2	0.24			21 days	
Leaf lettuce,	USA	480 g/L EC	Foliar	3 (7)	0.28	-	-	14 days	
Turnip greens	USA	480 g/L EC	Foliar	7 (3)	0.28	-	-	14 days	
Legume vegetables									
Beans, peas (not snow or sugar snap peas)	Australia	400 g/L EC	Foliar	As required	0.3	0.03		7 days (harvest, grazing)	
Peas	USA	480 g/L EC	Foliar	2 (7)	0.18-0.36 (max. 0.56 per season)	-	-	0 days	
Peas with pods	USA	480 g/L EC	Foliar	3 (14)	0.18	-	-	0 days	
Yard long bean	Thailand	400 g/L EC	Foliar	4 (7)	0.3-0.6	0.04-0.08	750	7 days	
Pulses									
Adzuki beans, Cowpeas, Mung beans, Navy beans, Pigeon peas, chickpeas, lupins, Borlotti beans	Australia	400 g/L EC	Foliar	As required, minimum RTI 14 days	0.32	0.03		14 (harvest) 14 (grazing)	
Field peas and beans	Australia	400 g/L EC	Foliar	As required, minimum RTI 14 days	0.32	0.03		14 (harvest) 14 (grazing)	
Peas (dry)	USA	480 g/L EC	Foliar	2 (7)	0.18-0.36 (max. 0.56 per season)	-	-	0 days	

Crop	Country	Formulation	Application						Comments
			Method	No. (RTI – days)	Rate (kg ai/ha)	Conc. (kg ai/100 L)	Spray volume (L/ha)	PHI (days)	
Soy beans	Australia	400 g/L EC	Foliar	As required, minimum RTI 14 days	0.136			14 (harvest) 14 (grazing)	
Soy beans	USA	480 g/L EC	Foliar	2 (7)	0.56	-	-	28 days (harvest) 5 days (grazing)	
Root and tuber vegetables									
Carrot, Parsnip	Czech Republic	400 g/L EC	Foliar	2	0.24			28 days	
Carrot, Parsnip, Parsley root	Estonia	400 g/L EC	Foliar	3 (7)	0.24	-	200-400	28 days	
Carrot, Parsnip, Parsley root	Greece	400 g/L EC	Foliar	3 (7)	0.20-0.24	0.067-0.08	300	35 days	
Sugar beet, fodder beet	Czech Republic	400 g/L EC	Foliar	2	0.24			28 days	
Sugar beet, beetroot	Estonia	400 g/L EC	Foliar	1	0.2	-	200-400	28 days	
Sugar beet, beetroot, turnip	Greece	400 g/L EC	Foliar	2 (21)	0.20-0.24	0.10-0.12	200	28 days	
Turnip	Australia	400 g/L EC	Foliar	As required	0.3	0.03		14 days	
Turnip	Estonia	400 g/L EC	Foliar	1	0.2	-	200-400	28 days	
Turnip	USA	480 g/L EC	Foliar	7 (3)	0.28	-	-	14 days	
Stalk and stem vegetables									
Asparagus	Australia	400 g/L EC	Foliar	As required	0.3	0.03	-	7 days	
Asparagus	Czech Republic	400 g/L EC	Foliar	2	0.2			Not required	
Asparagus	USA	480 g/L EC	Foliar	2 (14)	0.56	-	-	180 days	
Celery	USA	480 g/L EC	Foliar	3 (7)	0.28-0.56	-	-	7 days	
Cereal grains									
Cereals (wheat, barley, oats, triticale)	Australia	400 g/L EC	Foliar	As required	0.3	-	Ground: 50-100 Aerial: 20-40	Harvest: 4 weeks Grazing: 14 days	
Wheat	Brazil	400 g/L EC	Foliar	As required	0.252	-	150-200	28 days	
Winter barley, winter wheat, rye, triticale	Czech Republic	400 g/L EC	Foliar	1	0.2			Not required	Apply up to BBCH 69
Barley, oat	Estonia	400 g/L EC	Foliar	1	0.2	-	200-400	Not required	Apply up to BBCH 59
Wheat, winter	Estonia	400 g/L EC	Foliar	1	0.2	-	200-400	Not	Apply up to

Crop	Country	Formulation	Application						Comments
			Method	No. (RTI – days)	Rate (kg ai/ha)	Conc. (kg ai/100 L)	Spray volume (L/ha)	PHI (days)	
rye, triticale								required	BBCH 69
Cereals (wheat, rye, triticale, durum wheat)	Greece	400 g/L EC	Foliar	1	0.20	0.1	200	Not required	Apply up to end of flowering
Wheat	USA	480 g/L EC	Foliar	1	0.56	-	-	35 days (harvest) 14 days (grazing)	
Oilseeds									
Oilseeds (except peanuts and cotton (including mustard, linseed, poppy, canola, sunflower and safflower)	Australia	400 g/L EC	Foliar	1	0.14	-	Ground: 50-100 Aerial: 20-40	7 days (harvest) 7 days (grazing)	

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The residue trial tables below include values for the sum of dimethoate and omethoate used for livestock dietary burden calculation (where applicable).

Where residues were reported below the LOQ, the following conventions were adopted for summing residues:

Table 88 Convention adopted for summing of residues

Dimethoate (mg/kg)	Omethoate (mg/kg)	Sum of dimethoate and omethoate (mg/kg)
0.30	0.04	0.34
0.30	< 0.01	0.31
< 0.01	< 0.01	< 0.02

Citrus fruits – post harvest treatment

Mandarins

Mandarins from four sites in Australia were harvested and subjected to a post harvest dip treatment with a 400 g/L EC formulation of dimethoate at a concentration of 40 g ai/100 L (Melville – 2007). The fruit was immersed in the liquid for approximately 60 seconds before draining and allowing to air dry for 20 minutes. Control samples were immersed the same volume of tap water in place of the dipping solution. Samples were placed in freezer storage immediately after treatment.

Samples were separated into peel and flesh, then homogenised and subjected to extraction with acetonitrile, then magnesium sulfate and sodium chloride were added to create a partition. An

aliquot of the acetonitrile layer was concentrated and cleaned up using primary-secondary amine sorbent and magnesium sulfate, prior to analysis by LC-MS/MS.

Results were reported separately for peel and flesh. No weight data for the peel and flesh fractions were available in the study, so values were calculated for the whole fruit based on a default ratio of 30% peel to 70% flesh.

Table 89 Residues of dimethoate and omethoate in mandarins after post harvest dip treatment with 400 g/L EC dimethoate (Melville -2007)

Crop, variety, location, year	Treatment (g ai/100 L)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	Sum (mg/kg)
Mandarins, Imperial, Peats Ridge, NSW, Australia, 2007	40	0	Peel	2.3 (2.2, 2.4)	0.012 (0.015, 0.009)	2.3 (2.2, 2.4)
			Flesh	0.014 (0.017, 0.012)	< 0.01 (< 0.01, < 0.01)	0.024 (0.027, 0.022)
			Whole fruit (calculated)	0.70 (0.67, 0.73)	≤ 0.01 (< 0.01, < 0.01)	0.71 (0.68, 0.74)
Mandarins, Unique, Wallaville, QLD, Australia, 2007	40	0	Peel	1.8 (1.8, 1.8)	< 0.01 (< 0.01, < 0.01)	1.8 (1.8, 1.8)
			Flesh	0.056 (0.060, 0.051)	< 0.01 (< 0.01, < 0.01)	0.066 (0.070, 0.071)
			Whole fruit (calculated)	0.58 (0.58, 0.58)	≤ 0.01 (< 0.01, < 0.005)	0.58 (0.59, 0.58)
Mandarins, Imperial, Leeton, NSW, Australia, 2007	40	0	Peel	2.6 (2.5, 2.8)	< 0.01 (< 0.01, < 0.01)	2.6 (2.5, 2.8)
			Flesh	0.056 (0.057, 0.055)	< 0.01 (< 0.01, < 0.01)	0.066 (0.067, 0.065)
			Whole fruit (calculated)	0.82 (0.79, 0.88)	≤ 0.01 (< 0.01, < 0.01)	0.83 (0.80, 0.89)
Mandarins, Ellendale, Dareton, NSW, Australia, 2007	40	0	Peel	2.2 (2.3, 2.1)	< 0.01 (< 0.01, < 0.01)	2.2 (2.3, 2.1)
			Flesh	0.076 (0.062, 0.089)	< 0.01 (< 0.01, < 0.01)	0.086 (0.072, 0.099)
			Whole fruit (calculated)	0.71 (0.73, 0.69)	≤ 0.01 (< 0.01, < 0.01)	0.72 (0.74, 0.70)

No residues were detected in any of the untreated control samples.

Oranges

Oranges from six Australian test sites were collected at commercial harvest and subjected to a dip treatment of a 400 g/L EC formulation of dimethoate at 100 mL/100 L (40 g ai/100 L). The oranges were immersed in the dip liquid (50 L) for 60 seconds, before being drained and allowed to air dry under ambient conditions. The treated fruit were stored frozen until analysis which was completed within 3 months.

Dimethoate and omethoate residues were extracted from the homogenised orange peel and flesh samples with 1% acetic acid in acetonitrile. Sodium chloride and magnesium sulphate were added and the extract was vigorously mixed. The acetonitrile layer was cleaned up by addition of PSA (primary secondary amine) and magnesium sulphate prior to analysis by LC-MS/MS. The LOQ for the method was 0.001 mg/kg for each of dimethoate and omethoate in orange flesh and 0.003 mg/kg for each of dimethoate and omethoate in orange peel. Acceptable recoveries in the range were observed for both the method validation and concurrent recovery testing.

Table 90 Residues of dimethoate and omethoate in oranges after post-harvest dip treatment (Lean – 2012)

Crop, variety, location, year	Treatment (g ai/100 L)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	Sum (mg/kg)
Oranges, Joppa, Mundubbera, Qld, 2012	40	0	Peel	1.20	0.008	1.2
			Flesh	0.38	0.002	0.38
			Whole fruit (calculated)	0.66	0.004	0.66
Oranges, Washington navel, Gayndah, Qld, 2012	40	0	Peel	1.10	0.006	1.1
			Flesh	0.34	0.002	0.34
			Whole fruit (calculated)	0.59	0.003	0.59
Blood orange, Gayndah, Qld, 2012	40	0	Peel	1.30	0.009	1.3
			Flesh	0.37	0.003	0.37
			Whole fruit (calculated)	0.67	0.005	0.68
Oranges, Washington navel, Red Cliffs, Vic, 2012	40	0	Peel	1.100	0.008	1.1
			Flesh	0.260	0.002	0.26
			Whole fruit (calculated)	0.600	0.004	0.60
Oranges, Navel, Autumn gold, Lyrup, SA, 2012	40	0	Peel	1.100	0.007	1.1
			Flesh	0.190	0.001	0.19
			Whole fruit (calculated)	0.510	0.003	0.51
Blood orange, Gol Gol, NSW, 2012	40	0	Peel	1.450	0.009	1.5
			Flesh	0.275	0.001	0.28

Crop, variety, location, year	Treatment (g ai/100 L)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	Sum (mg/kg)
			Whole fruit (calculated)	0.630	0.003	0.63

The concentration of dimethoate and omethoate in the dip solutions was also measured as summarized below:

Study site	Sample	Days after treatment	Dimethoate (mg/kg)	Omethoate (mg/kg)
1	Dip liquid	0	277.8	2.1
2	Dip liquid	0	285.7	2.5
3	Dip liquid	0	302.7	2.3
4	Dip liquid	0	271.8	2.8
5	Dip liquid	0	280.3	2.8
6	Dip liquid	0	270.4	2.7

Citrus fruit – foliar application

Oranges – foliar treatment

A series of trials was conducted in Brazil (de Toledo – 2002a, b and c). Dimethoate was applied as a 400 g/L EC formulation using an airblast sprayer. Three applications were made at approximately 30-day intervals as a dilute foliar spray (spray volume = 2000 L/ha), with spray concentration of either 0.04 or 0.08 kg ai/100 L (1× or 2× respectively). At all sites, samples were collected from the 1× and 2× treated plots and an untreated control plot 3 days after the last application, and frozen until processed at the laboratory. At one site, a decline trial was conducted on the 1× plot and samples were collected from 0 to 21 days after the last application. At the laboratory, samples were separated into peel and flesh. Samples were extracted by homogenisation, cleaned up by addition of dichlormethane/hexane to create an organic/aqueous partition, concentrated and made up to volume in iso-octane/toluene (9:1 v/v) before analysis by GC-FPD. Concurrent recoveries ranged from 70-100% for dimethoate and 73-118% for omethoate. Analyses were completed within 5.5 months of sample collection.

Table 91 Residues of dimethoate and omethoate in oranges after high volume foliar application of a 400 g/L EC dimethoate product (de Toledo – 2002)

Crop, variety, location, year	No. (RTI, days)	Spray concentration (kg ai/100 L)	Spray volume	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Oranges, Pear, Santa Rita do Passa Quatro, Sao Paolo, Brazil, 2002	Untreated control			-	Peel	< 0.02	< 0.30
				-	Pulp	< 0.02	< 0.30
				-	Whole fruit	< 0.02	< 0.30
	3 (31, 30)	0.04	2000	3	Peel	0.77	< 0.30
				3	Pulp	0.13	< 0.30

Crop, variety, location, year	No. (RTI, days)	Spray concentration (kg ai/100 L)	Spray volume	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
				3	Whole fruit	0.15	< 0.30
	3 (31, 30)	0.08	2000	3	Peel	2.4	< 0.30
				3	Pulp	0.59	< 0.30
				3	Whole fruit	0.77	< 0.30
Oranges, Pear, Tatui, Sao Paolo, Brazil, 2002	Untreated control			-	Peel	< 0.02	< 0.30
				-	Pulp	< 0.02	< 0.30
				-	Whole fruit	< 0.02	< 0.30
	3 (31, 30)	0.04	2000	3	Peel	0.71	< 0.30
				3	Pulp	0.09	< 0.30
				3	Whole fruit	0.48	< 0.30
	3 (31, 30)	0.08	2000	3	Peel	2.3	< 0.30
				3	Pulp	0.71	< 0.30
				3	Whole fruit	1.3	< 0.30
Oranges, Pear, Plantec, Iracemapolis, Sao Paolo, Brazil, 2002	Untreated control			-	Peel	< 0.02	< 0.30
				-	Pulp	< 0.02	< 0.30
				-	Whole fruit	< 0.02	< 0.30
	3 (31, 31)	0.04	2000	0	Peel	0.67	< 0.30
				3	Peel	0.56	< 0.30
				7	Peel	0.53	< 0.30
				14	Peel	0.54	< 0.30
				21	Peel	1.7	< 0.30
				0	Pulp	0.05	< 0.30
				3	Pulp	0.08	< 0.30
				7	Pulp	0.09	< 0.30
				14	Pulp	0.03	< 0.30
				21	Pulp	0.04	< 0.30
				0	Whole fruit	0.23	< 0.30
				3	Whole fruit	0.20	< 0.30
				7	Whole fruit	0.24	< 0.30
				14	Whole fruit	0.45	< 0.30
				21	Whole fruit	0.11	< 0.30
	3 (31, 31)	0.08	2000	3	Peel	1.8	< 0.30

Crop, variety, location, year	No. (RTI, days)	Spray concentration (kg ai/100 L)	Spray volume	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
				3	Pulp	0.32	< 0.30
				3	Whole fruit	0.97	< 0.30

Mandarins – foliar treatment

Residue trials were conducted using a 400 g/L EC formulation in mandarins at a single site in Italy in the 2002 growing season (Wilson – 2003. One untreated control and three treated plots were established at the site, with plots receiving one, two or three applications using a backpack sprayer and a hand lance at a target per-hectare rate of 0.75 kg ai/ha in a target spray volume of 2000 L/ha (nominal spray concentration of 0.0375 kg/100 L). Samples of whole fruit, peel and pulp were collected and frozen on the days of sample collection. Samples were extracted by maceration with dichloromethane, cleaned up by partition with hexane and water, and the aqueous phase was analysed for imethoate and omethoate by LC-MS (method LOQ = 0.01 mg/kg for both analytes). Concurrent recoveries were in the range 70–110%. Samples were analysed within 3 months of collection.

Table 92 Residues of dimethoate and omethoate in mandarins in Italy after foliar treatment with a 400 g/L EC formulation (Wilson – 2003)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Mandarins, Clementino Liscio di Paterno, Bernalda, Italy, 2002	3 (38, 108)	71, 74, 89	0.79, 0.78, 0.78	2028, 2004, 2000	0.039, 0.039, 0.039	0	Whole fruit	1.8	0.01
						21	Whole fruit	0.31	0.06
						21	Peel	1.6	0.10
						21	Pulp	< 0.01 (0.002)	< 0.01 (0.006)
	2 (38)	71, 74	0.80, 0.78	2050, 2004	0.039, 0.039	129	Whole fruit	< 0.01 (ND)	< 0.01 (0.003)
						129	Peel	< 0.01 (ND)	< 0.01 (ND)
						129	Pulp	< 0.01 (ND)	< 0.01 (ND)
	1	71	0.80	2041	0.039	167	Whole fruit	< 0.01 (ND)	< 0.01 (ND)
						167	Peel	< 0.01 (ND)	< 0.01 (ND)
						167	Pulp	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the control samples.

Three residue trials in citrus (one in mandarins and two in oranges) were conducted in Spain in the 2003 growing season (Wilson – 2004). Two applications of a 400 g/L EC formulation were made to the treated plots at each site, at BBCH growth stages 69 and 73–74, at a target rate of 0.75 kg

ai/ha in either 2000 or 3000 L/ha (mandarins and oranges respectively). Samples of whole fruit, peel and pulp were collected on the day of the last application after the spray had dried, and at earliest commercial harvest. Samples were frozen on the day of collection and kept frozen until analysis. Samples were extracted by maceration with dichloromethane, cleaned up by partition with hexane and water, and the aqueous phase was analysed for imethoate and omethoate by LC-MS (method LOQ = 0.01 mg/kg for both analytes). Concurrent recoveries were in the range 70–83%. Samples were analysed within 5 months of collection.

Table 93 Residues of dimethoate and omethoate in mandarins in Spain after foliar treatment with a 400 g/L EC formulation (Wilson – 2004)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Mandarins, Clementina de Nules, Betera, Valencia, Spain, 2003	2 (98)	69, 73/74	0.79, 0.79	2025, 2021	0.039, 0.039	0	Whole immature fruit	2.0	0.02
						91	Whole mature fruit	< 0.01	< 0.01
						91	Peel	0.03	< 0.01 (ND)
						91	Pulp	< 0.01 (ND)	< 0.01 (ND)

No residues were found above the LOQ in any of the control samples.

Table 94 Residues of dimethoate and omethoate in oranges in Spain after foliar treatment with a 400 g/L EC formulation (Wilson – 2004)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Oranges, Newhall, Benimamet, Valencia, Spain, 2003	2 (55)	69, 74	0.78, 0.79	2996, 3022	0.026, 0.026	0	Whole immature fruit	0.98	0.02
						106	Whole mature fruit	< 0.01 (ND)	< 0.01 (ND)
						106	Peel	< 0.01 (ND)	< 0.01 (ND)
						106	Pulp	< 0.01 (ND)	< 0.01 (ND)
Oranges, Navelina, Benimamet, Valencia, Spain, 2003	2 (55)	69, 74	0.79, 0.79	3033, 3026	0.026, 0.026	0	Whole immature fruit	1.0	0.01
						106	Whole mature	< 0.01 (ND)	< 0.01 (ND)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
							fruit		
						106	Peel	< 0.01 (ND)	< 0.01 (ND)
						106	Pulp	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the control samples.

Residue trials were conducted using a 400 g/L EC formulation in mandarins and oranges (two trials each) at sites in Italy and Spain the 2004 growing season (Wilson – 2005). Two applications of a 400 g/L EC formulation were made to the treated plots at each site, at BBCH growth stages 69 and 73–74, at a target rate of 0.75 kg ai/ha in either 2000 L/ha. Samples of whole fruit, peel and pulp were collected on the day of the last application after the spray had dried, and at earliest commercial harvest. Samples were frozen on the day of collection and kept frozen until analysis. Samples were extracted by maceration with dichloromethane, cleaned up by partition with hexane and water, and the aqueous phase was analysed for imethoate and omethoate by LC-MS (method LOQ = 0.01 mg/kg for both analytes). Concurrent recoveries were in the range 82–103%. Samples were analysed within 5 months of collection.

Table 95 Residues of dimethoate and omethoate in mandarins in Italy and Spain after foliar treatment with a 400 g/L EC formulation (Wilson – 2005)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Mandarins, Clementina de Nules, Betera, Spain, 2004	2 (65)	69, 73-74	0.77, 0.79	2037, 2093	0.038, 0.038	0	Whole immature fruit	2.2	0.01
						103	Whole mature fruit	< 0.01 (ND)	< 0.01 (ND)
						103	Peel	< 0.01 (ND)	< 0.01 (ND)
						103	Pulp	< 0.01 (ND)	< 0.01 (ND)
MandarinsClementine Monreal, Lentini, Italy, 2004	2 (67)	69, 74	0.75, 0.76	1973, 1997	0.038, 0.038	0	Whole immature fruit	2.4	0.01
						123	Whole mature fruit	< 0.01 (ND)	< 0.01 (ND)
						123	Peel	< 0.01 (ND)	< 0.01 (ND)
						123	Pulp	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the control samples.

Table 96 Residues of dimethoate and omethoate in oranges in Italy after foliar treatment with a 400 g/L EC formulation (Wilson – 2005)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Oranges, Tarocco, Catania, Sicily, Italy, 2004	2 (73)	69, 74	0.75, 0.75	1967, 1983	0.038, 0.038	0	Whole immature fruit	1.0	< 0.01 (0.008)
						127	Whole mature fruit	< 0.01 (ND)	< 0.01 (ND)
						127	Peel	< 0.01 (ND)	< 0.01 (ND)
						127	Pulp	< 0.01 (ND)	< 0.01 (ND)
Oranges, Tarocco Scire, Scordia, Sicily, Italy, 2004	2 (73)	69, 74	0.75, 0.76	1973, 1999	0.038, 0.038	0	Whole immature fruit	1.8	0.01
						133	Whole mature fruit	< 0.01 (ND)	< 0.01 (ND)
						133	Peel	< 0.01 (ND)	< 0.01 (ND)
						133	Pulp	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the control samples.

Cherries

Trials were conducted in sweet cherries at three sites in northern Europe during the 2008 growing season (Raufer – 2009a) and at one site during the 2009 growing season (Raufer – 2009b). A single foliar application of a 400 g/L dimethoate EC formulation was made using a knapsack sprayer late in the season (target growth stage BBCH 81, target rate and spray concentration 0.4 kg ai/ha and 0.0267 kg ai/100 L), with fruit being sample at intervals from 0 to 28 days after application. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Less mature fruit was homogenised whole (i.e. including the stones) before extraction, while the more mature fruit samples were separated into flesh and stones, with just the flesh extracted, but residues calculated on a whole fruit basis. Recoveries of dimethoate and omethoate from fortified control samples ranged from 75–95%. Samples were analysed within 5 months of collection.

Table 97 Residues of dimethoate and omethoate in sweet cherries in northern Europe after a single foliar application of a 400 g/L EC formulation (Raufer – 2009a, 2009b)

Crop, variety, location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
Cherries, Altenberger Melonenkirsche, 15345, Altlandsberg OT Wesendahl, Brandenburg, Germany, 2008	81	0.40	1516	0.027	0	Whole fruit	1.0	< 0.01	1.01
					7	Whole fruit	0.52	0.12	0.65
					10	Whole fruit	0.20	0.08	0.29
					14	Flesh[a]	0.10	0.09	0.20
					21	Flesh[a]	0.02	0.04	0.06
					28	Flesh[a]	0.01	0.05	0.06
Cherries, Regina, 71735, Eberdingen, Baden-Württemberg, Germany, 2008	81-85	0.39	1472	0.027	0	Whole fruit	0.62	< 0.01	0.63
					7	Whole fruit	0.45	0.08	0.54
					10	Whole fruit	0.18	0.07	0.26
					14	Flesh[a]	0.12	0.06	0.18
					21	Flesh [a]	0.03	0.05	0.08
					28	Flesh [a]	< 0.01	0.04	0.05
Cherries, Kordia, 64530, Gaj Wielki, Wielkopolska, Poland, 2008	81	0.40	1491	0.027	0	Whole fruit	1.0	< 0.01	1.01
					7	Whole fruit	0.19	0.08	0.28
					10	Whole fruit	0.09	0.09	0.19
					14	Flesh [a]	0.02	0.04	0.06
					21	Flesh [a]	< 0.01	0.03	0.04
					28	Flesh [a]	< 0.01	< 0.01	< 0.02
Cherries, Kordia, 21635, Jork, Niedersachsen, Germany, 2009	81	0.42	1569	0.027	0	Whole fruit	1.7	0.03	1.73
					7	Whole fruit	0.86	0.21	1.09

Crop, variety, location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
					10	Whole fruit	0.51	0.29	0.82
					14	Flesh[a]	0.27	0.27	0.56
					21	Flesh[a]	0.03	0.11	0.15
					28	Flesh[a]	< 0.01	0.08	0.10

[a] Stones separated and weighed and residues calculated on a whole fruit basis.

No residues were detected in any of the control samples.

A second series of trials was conducted in sweet cherries at four sites in northern Europe during the 2008 growing season (Raufer – 2009c). 2 × 0.4 kg ai/ha (0.0267 kg ai/100 L) foliar applications of a 400 g/L dimethoate EC formulation was made using a knapsack sprayer late in the season (target growth stage BBCH 81), with fruit being sample at intervals from 0 to 28 days after application. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Less mature fruit was homogenised whole (i.e. including the stones) before extraction, while the more mature fruit samples were separated into flesh and stones, with just the flesh extracted, but residues calculated on a whole fruit basis. Recoveries of dimethoate and omethoate from fortified control samples ranged from 84–96%. Samples were analysed within 5 months of collection.

Table 98 Residues of dimethoate and omethoate in sweet cherries in northern Europe after two foliar applications of a 400 g/L EC formulation (Raufer – 2009c)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Cherries, Altenberger Melonenkirsche, 15345, Altlandsberg, Brandenburg, Germany, 2008	2 (7)	79-81, 81	0.40, 0.41	1502, 1553	0.027, 0.027	0	Whole fruit	0.82	0.07
						7	Whole fruit	0.32	0.15
						10	Whole fruit	0.11	0.11
						14	Flesh[a]	0.05	0.10
						21	Flesh[a]	0.02	0.08
						28	Flesh[a]	< 0.01	0.06
Cherries, Kordia, 21635, Jork, Niedersachsen, Germany, 2008	2 (7)	81-83, 81-83	0.39, 0.38	1458, 1426	0.027, 0.027	0	Whole fruit	2.5	0.19
						7	Whole fruit	1.3	0.38

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
						10	Whole fruit	0.63	0.33
						14	Flesh[a]	0.51	0.38
						21	Flesh[a]	0.10	0.22
						28	Flesh[a]	0.08	0.29
Cherries, Regina, 71735, Eberdingen, Baden-Württemberg, Germany, 2008	2 (7)	81, 81-85	0.39, 0.40	1466, 1491	0.027, 0.027	0	Whole fruit	1.0	0.11
						7	Whole fruit	0.97	0.16
						10	Whole fruit	0.63	0.16
						14	Flesh[a]	0.28	0.14
						21	Flesh[a]	0.15	0.12
						28	Flesh[a]	0.12	0.16
Cherries, Kordia, 64500, Szamotuly, Wielkopolska, Poland, 2008	2 (7)	79, 82	0.41, 0.40	1524, 1486	0.027, 0.027	0	Whole fruit	2.5	0.19
						7	Whole fruit	0.61	0.28
						10	Whole fruit	0.15	0.16
						14	Flesh[a]	0.06	0.13
						21	Flesh[a]	0.04	0.17
						28	Flesh[a]	< 0.01	0.08

[a] Stones separated and weighed and residues calculated on a whole fruit basis.

No residues were detected in any of the control samples.

Olives

A series of trials was conducted in olives at sites in southern Europe during the 2008 (Raufer – 2009d), 2009 (Raufer – 2010a), 2010 (Raufer – 2011a), and 2011 (Raufer – 2012a) growing seasons. 2 × 0.48 kg ai/ha (0.04 kg ai/100 L) foliar applications of a 400 g/L dimethoate EC formulation was made using a knapsack sprayer at a re-treatment interval of 10 days late in the season (target growth stage BBCH 80–87), with fruit being sample at intervals from 0 to 42 days after application. Samples were frozen within 9 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Less mature fruit was homogenised whole (i.e. including the stones) before extraction, while the more mature fruit samples were separated into flesh and stones, with just the flesh extracted,

but residues calculated on a whole fruit basis. Recoveries of dimethoate and omethoate from fortified control samples ranged from 67–112%. Samples were analysed within 7 months of collection.

Table 99 Residues of dimethoate and omethoate in olives in southern Europe after 2 × 0.48 kg ai/ha (0.04 kg ai/100 L) foliar applications of a 400 g/L EC formulation (Raufer – 2009d and Raufer-2010a)

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
								DM	OM	DM + OM
Olives, Piwal, 46830, Beniganim, Valencia, Spain, 2008	2 (10)	80, 80	0.52, 0.52	1232, 1240	0.042, 0.042	0	Whole fruit	4.1	0.23	4.33
						7	Whole fruit	2.4	0.36	2.76
						14	Whole fruit	2.4	0.44	2.84
						21	Flesh [a]	1.6	0.34	1.94
						28	Flesh [a]	1.2	0.35	1.55
						35	Flesh [a]	0.88	0.31	1.19
						42	Flesh [a]	0.48	0.27	0.75
Olives, Piwal, 46814, Llanera de Ranes, Valencia, Spain, 2008	2 (10)	80, 81	0.50, 0.52	1178, 1227	0.042, 0.042	0	Whole fruit	3.9	0.29	4.2
						7	Whole fruit	2.0	0.35	2.35
						14	Whole fruit	0.88	0.27	1.15
						21	Flesh [a]	0.53	0.28	0.81
						28	Flesh [a]	0.41	0.35	0.76
						35	Flesh [a]	0.30	0.28	0.58
						42	Flesh [a]	0.16	0.18	0.34
Olives, Megariti, 64200, Zarkadia, Karala, Greece, 2008	2 (10)	81, 81	0.48, 0.46	1202, 1156	0.04, 0.04	0	Whole fruit	0.67	< 0.01	0.68
						7	Whole fruit	0.15	0.02	0.17
						14	Whole fruit	0.14	0.01	0.15
						21	Flesh [a]	0.02	< 0.01	0.03
						28	Flesh [a]	< 0.01	< 0.01	< 0.02
						35	Flesh [a]	0.01	< 0.01	0.02
						42	Flesh [a]	< 0.01	< 0.01	< 0.02

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
								DM	OM	DM + OM
Olives, Frantolo, 50060, Pelago, Toscana, Italy, 2008	2 (10)	87-89, 87-89	0.51, 0.47	1264, 1178	0.04, 0.04	0	Whole fruit	6.2	0.31	6.51
						7	Whole fruit	2.6	0.38	3.0
						14	Whole fruit	2.1	0.49	2.6
						21	Flesh [a]	0.93	0.23	1.2
						28	Flesh [a]	0.63	0.20	0.83
						35	Flesh [a]	0.55	0.22	0.77
						42	Flesh [a]	0.38	0.16	0.54
Olives, Picholine, 66300, Saint-Jean Lasseille, Languedoc-Roussillon, France, 2009	2 (10)	85, 87	0.49, 0.50	1226, 1241	0.04, 0.04	0	Whole fruit	2.3	0.08	2.4
						28	Flesh	0.55	0.24	0.79
						35	Flesh [a]	0.34	0.16	0.49
						42	Flesh [a]	0.27	0.14	0.41
Olives, Megaritiki, 64008, Kariani, Kavala, Greece, 2009	2 (10)	85-87, 87	0.47, 0.47	1188, 1189	0.04, 0.04	0	Whole fruit	2.2	0.04	2.24
						28	Flesh [a]	0.11	0.29	0.40
						35	Flesh [a]	0.03	0.17	0.20
						42	Flesh [a]	< 0.01	0.13	0.14
Olives, Chalkidikis, 64200, Zarkadia, Kavala, Greece, 2009	2 (10)	87, 87	0.48, 0.48	1203, 1199	0.04, 0.04	0	Whole fruit	2.0	0.47	2.5
						28	Flesh [a]	0.11	0.30	0.41
						35	Flesh [a]	0.02	0.17	0.19
						42	Flesh [a]	0.02	0.13	0.15
Olives, Manzanillo, 41600, Arahal, Andalusia,	2 (10)	80, 80	0.48, 0.48	1194, 1202	0.04, 0.04	0	Whole fruit	5.9	0.92	6.8

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
								DM	OM	DM + OM
Spain, 2009										
						7	Whole fruit	1.9	1.1	3.0
						14	Whole fruit	0.47	1.2	1.7
						21	Whole fruit	0.17	1.2	1.4
						28	Flesh [a]	0.06	0.69	0.75
						35	Flesh [a]	0.06	0.38	0.44
						42	Flesh [a]	< 0.01	0.25	0.26
Olives, Hojiblanca, 14548, Montalban de Cordoba, Andalusia, Spain, 2009	2 (10)	80, 80	0.48, 0.48	1209, 1200	0.04, 0.04	0	Whole fruit	3.5	0.82	4.3
						7	Whole fruit	1.5	0.99	2.5
						14	Whole fruit	0.12	0.48	0.60
						21	Whole fruit	0.37	0.55	0.92
						28	Flesh [a]	0.07	0.28	0.35
						35	Flesh [a]	0.01	0.12	0.13
						42	Flesh [a]	0.02	0.19	0.21
Olives, Picholine, 66690, Saint Andre, Pyrenees-Orientales, France, 2010	2 (11)	81, 85	0.49, 0.49	1224, 1221	0.04, 0.04	0	Whole fruit	1.9	0.19	2.1
						27	Flesh [a]	0.02	0.15	0.17
						34	Flesh [a]	< 0.01	0.11	0.12
						41	Flesh [a]	0.01	0.08	0.09
Olives, Megaritiki, 64008, Kariani, Kavala, Greece, 2010	2 (10)	75-79, 79	0.48, 0.48	1207, 1198	0.04, 0.04	0	Whole fruit	1.2	0.37	1.6
						28	Flesh [a]	< 0.01	0.29	0.30
						35	Flesh [a]	< 0.01	0.13	0.14
						42	Flesh [a]	< 0.01	0.10	0.11

Crop, variety, location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
								DM	OM	DM + OM
Olives, Aglando, 13590, Meyreuil, Provence-Alpes-Cotes d'Azur, France, 2011	2 (10)	80, 81	0.48, 0.49	1204, 1219	0.04, 0.04	0	Whole fruit	3.4	0.53	3.9
						28	Flesh [a]	< 0.01	0.21	0.22
						35	Flesh [a]	< 0.01	0.14	0.15
						42	Flesh [a]	< 0.01	0.09	0.10

[a] Stones separated and weighed and residues calculated on a whole fruit basis.

No residues were detected in any of the control samples.

A series of trials for olives grown in southern Europe during the 2012 growing season included analysis for three additional dimethoate metabolites (Martos – 2014). 2 × 0.48 kg ai/ha (0.04 kg ai/100 L) foliar applications of a 400 g/L dimethoate EC formulation was made using a knapsack sprayer at a re-treatment interval of 10 days late in the season (target growth stage BBCH 80–87), with treated and control fruit samples being collected at 0, 28, 35 and 42 days after the last application. Samples were frozen within 9 hours of collection and analysed for dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate. The 0-day samples were analysed whole, while the later, more mature fruit samples were separated into flesh and stones, with only the flesh being analysed and the residues being calculated on a whole fruit basis using the flesh/stone weight ratios. Dimethoate, omethoate, and dimethoate carboxylic acid were analysed using a QuEChERS-based method, involving extraction with acetonitrile, cleanup by partition through addition of magnesium sulfate, sodium chloride, sodium citrate dibasic and sodium citrate tribasic, further cleanup of an aliquot of the organic phase with PSA sorbent, C18EC and magnesium sulfate, followed by analysis with LC-MS/MS. For determination of O-desmethyl omethoate carboxylic acid and O-desmethyl isodimethoate, samples were extracted with methanol/water (1:1 v/v), purified by solid phase extraction (Strata AX-W column, elution with 99:1 v/v methanol/35% ammonia), evaporated to dryness and reconstituted in dilute formic acid prior to LC-MS/MS analysis. The LOQs were 0.01 mg/kg for each analyte. Concurrent method recoveries were within the range 72–107% for all analytes. Extractions and analysed were completed within 5 months of sample collection.

The samples from the Martos – 2014 study were later analysed for further metabolites – desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate (Amic – 2014a). The three additional metabolites were analysed using the same method as for O-desmethyl isodimethoate and O-desmethyl omethoate carboxylic acid – extraction with methanol/water (1:1 v/v), purified by solid phase extraction (Strata AX-W cartridge, elution with 99:1 v/v methanol/35% ammonia), evaporated to dryness and reconstituted in dilute formic acid prior to LC-MS/MS analysis. The LOQs were 0.01 mg/kg for each analyte. Concurrent method recoveries were within the range 75–101% for all analytes. Samples were stored for up to 13 months before analysis.

Table 100 Residues of dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in olives in southern Europe during the 2012 growing season after 2 × 0.48 kg ai/ha (0.04 kg ai/100 L) foliar applications of a 400 g/L EC formulation (Martos-2014)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Conc. (kg ai/100 L)	DALA	Residues (mg/kg)					
							DM	OM	DCA	ODI	OCA	DM + OM
Olives (Coratina), 75010, Metaponto, Matera, Italy, 2012	2 (11)	75, 79	0.50, 0.50	1213, 1227	0.041, 0.041	0	2.0	0.34	< 0.01 (ND)	< 0.01 (ND)	< 0.01	2.3
						28	0.06	0.22	< 0.01 (ND)	< 0.01 (ND)	0.01	0.28
						35	< 0.01	0.20	< 0.01 (ND)	< 0.01 (ND)	< 0.01	0.21
						43	< 0.01 (ND)	0.11	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	0.12
Olives (Frantoio), 71045, Orta Nova, Foggia, Italy, 2012	2 (10)	75, 79	0.49, 0.49	1201, 1201	0.041, 0.041	0	0.93	0.12	< 0.01	< 0.01 (ND)	0.03	1.0
						28	0.12	0.07	< 0.01 (ND)	< 0.01 (ND)	0.02	0.19
						35	0.01	0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01	0.02
						42	0.04	0.04	< 0.01 (ND)	< 0.01 (ND)	0.02	0.08
Olives (Chondroelia Chalkidikis), 63085, Nea Skioni, Chalkidiki, Greece, 2012	2 (9)	79-81, 81-85	0.50, 0.49	1205, 1199	0.041, 0.041	0	3.4	0.41	< 0.01	< 0.01 (ND)	0.08	3.8
						28	0.16	0.28	< 0.01 (ND)	< 0.01 (ND)	0.11	0.44
						35	0.22	0.24	< 0.01 (ND)	< 0.01 (ND)	0.11	0.46
						42	0.02	0.23	< 0.01 (ND)	< 0.01 (ND)	0.12	0.25
Olives (Kalamon), 63200, Agios Mamas, Chalkidiki, Greece, 2012	2 (9)	79-81, 81	0.49, 0.49	1200, 1198	0.041, 0.041	0	3.5	0.48	< 0.01	< 0.01 (ND)	0.05	4.0
						28	0.74	0.78	< 0.01	< 0.01 (ND)	0.13	1.5
						35	0.43	0.33	< 0.01	< 0.01 (ND)	0.11	0.76
						40	0.16	0.33	< 0.01	< 0.01	0.09	0.49

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Conc. (kg ai/100 L)	DALA	Residues (mg/kg)					
							DM	OM	DCA	ODI	OCA	DM + OM
									(ND)	(ND)		
Olives (Arbequina), 46821, Chella, Valencia, Spain, 2012	2 (10)	81, 81-85	0.48, 0.50	1174, 1216	0.041, 0.041	0	3.7	0.31	< 0.01	< 0.01 (ND)	0.02	4.0
						27	0.34	0.50	< 0.01	< 0.01 (ND)	0.08	0.84
						35	0.07	0.33	< 0.01 (ND)	< 0.01 (ND)	0.05	0.40
						41	0.06	0.27	< 0.01 (ND)	< 0.01 (ND)	0.04	0.33
Olives (Alfafarenca), 03837, Agres, Alicante, Spain, 2012	2 (10)	81, 81	0.50, 0.48	1225, 1168	0.041, 0.041	0	3.7	0.15	< 0.01	< 0.01 (ND)	0.01	3.8
						28	0.76	0.26	< 0.01	< 0.01 (ND)	0.10	1.0
						35	0.58	0.23	< 0.01	< 0.01 (ND)	0.09	0.81
						42	0.55	0.27	< 0.01	< 0.01 (ND)	0.11	0.82
Olives (Picual), 46630, La Font de la Figuera, Valencia, Spain, 2012	2 (11)	80, 81-85	0.49, 0.50	1187, 1205	0.041, 0.041	0	4.8	0.31	< 0.01	< 0.01 (ND)	0.03	5.1
						29	1.5	0.36	< 0.01	< 0.01 (ND)	0.13	1.9
						35	1.1	0.34	< 0.01	< 0.01 (ND)	0.16	1.4
						42	0.25	0.26	< 0.01 (ND)	< 0.01 (ND)	0.10	0.51
Olives (Villalonga), 12580, Benicarlo, Castellon, Spain, 2012	2 (10)	81, 81	0.49, 0.47	1193, 1142	0.041, 0.041	0	6.3	0.32	0.02	< 0.01 (ND)	0.08	6.6
						29	1.5	0.88	0.01	< 0.01 (ND)	0.35	2.4
						36	0.85	0.75	< 0.01	< 0.01 (ND)	0.30	1.6
						41	0.26	0.35	< 0.01	< 0.01 (ND)	0.29	0.61

No residues were found above the LOQ in any of the untreated control samples, and the overwhelming majority contained no detectable residues.

Day 0 samples homogenised and analysed whole. Day 28-42 samples were separated into flesh and stones, flesh was homogenised and analysed and residues calculated on a whole fruit basis using the fractional weight ratios.

DM = dimethoate; OM = omethoate; DCA = dimethoate carboxylic acid; ODI = O-desmethyl isodimethoate; OCA = O-

desmethyl omethoate carboxylic acid.

Table 101 Residues of desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in olives in southern Europe during the 2012 growing season after 2 × 0.48 kg ai/ha (0.04 kg ai/100 L) foliar applications of a 400 g/L EC formulation (Amic – 2014a)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Conc. (kg ai/100 L)	DALA	Residues (mg/kg)		
							DMD	ODO	ONO
Olives (Coratina), 75010, Metaponto, Matera, Italy, 2012	2 (11)	75, 79	0.50, 0.50	1213, 1227	0.041, 0.041	0	0.04	0.03	< 0.01 (ND)
						28	0.06	0.04	< 0.01 (ND)
						35	0.06	0.03	< 0.01 (ND)
						43	0.04	0.02	< 0.01 (ND)
Olives (Frantoio), 71045, Orta Nova, Foggia, Italy, 2012	2 (10)	75, 79	0.49, 0.49	1201, 1201	0.041, 0.041	0	0.03	0.02	< 0.01 (ND)
						28	0.03	0.01	< 0.01 (ND)
						35	0.03	< 0.01	< 0.01 (ND)
						42	0.02	< 0.01	< 0.01 (ND)
Olives (Chondroelia Chalkidikis), 63085, Nea Skioni, Chalkidiki, Greece, 2012	2 (9)	79-81, 81-85	0.50, 0.49	1205, 1199	0.041, 0.041	0	0.05	0.06	< 0.01 (ND)
						28	0.05	0.08	< 0.01 (ND)
						35	0.06	0.07	< 0.01 (ND)
						42	0.04	0.07	< 0.01 (ND)
Olives (Kalamon), 63200, Agios Mamas, Chalkidiki, Greece, 2012	2 (9)	79-81, 81	0.49, 0.49	1200, 1198	0.041, 0.041	0	0.05	0.03	< 0.01 (ND)
						28	0.03	0.06	< 0.01 (ND)
						35	0.01	0.05	< 0.01 (ND)
						40	0.04	0.06	< 0.01

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Conc. (kg ai/100 L)	DALA	Residues (mg/kg)		
							DMD	ODO	ONO
									(ND)
Olives (Arbequina), 46821, Chella, Valencia, Spain, 2012	2 (10)	81, 81-85	0.48, 0.50	1174, 1216	0.041, 0.041	0	0.03	0.03	< 0.01 (ND)
						27	0.10	0.06	< 0.01 (ND)
						35	0.05	0.03	< 0.01 (ND)
						41	0.06	0.04	< 0.01 (ND)
Olives (Alfafarenca), 03837, Agres, Alicante, Spain, 2012	2 (10)	81, 81	0.50, 0.48	1225, 1168	0.041, 0.041	0	0.01	0.02	< 0.01 (ND)
						28	0.09	0.05	< 0.01 (ND)
						35	0.03	0.06	< 0.01 (ND)
						42	0.04	0.06	< 0.01 (ND)
Olives (Picual), 46630, La Font de la Figuera, Valencia, Spain, 2012	2 (11)	80, 81-85	0.49, 0.50	1187, 1205	0.041, 0.041	0	0.03	0.02	< 0.01 (ND)
						29	0.19	0.06	< 0.01 (ND)
						35	0.13	0.06	< 0.01 (ND)
						42	0.10	0.05	< 0.01 (ND)
Olives (Villalonga), 12580, Benicarlo, Castellon, Spain, 2012	2 (10)	81, 81	0.49, 0.47	1193, 1142	0.041, 0.041	0	0.04	0.04	< 0.01 (ND)
						29	0.13	0.13	< 0.01 (ND)
						36	0.10	0.13	< 0.01 (ND)
						41	0.09	0.12	< 0.01 (ND)

No residues were found above the LOQ in any of the untreated control samples, and the overwhelming majority contained no detectable residues.

DMD = desmethyl dimethoate, ODO = O-desmethyl omethoate, ONO = O-desmethyl N-desmethyl omethoate.

A further series of trials was conducted during the 2017 growing season and including testing of the full suite of metabolites (Gemrot-2018). At each trial two applications of a 400 g/L EC dimethoate formulation were made at a nominal rate of 480g a.i./ha and water volume of 1200 L/ha. Applications were made with a 10 day retreatment interval with the final application being made 28 days before commercial harvest.

In the decline curves, RAC specimens (whole fruit) were collected at 0, 7–8, 13–14, 21–22 and 28 DALA (commercial harvest). In the harvest trials, RAC specimens (whole fruit) were collected at the time of commercial harvest, 28 DALA.

Dimethoate and its metabolites omethoate and dimethoate carboxylic acid were analysed using methods described in the report and amendment of study S12-04027, except for crude and refined oil for which the method was modified and validated during this study.

Desmethyl dimethoate, O-desmethyl omethoate, O-desmethyl iso-dimethoate, O-desmethyl omethoate carboxylic acid and O-desmethyl N-desmethyl omethoate were analysed using methods described in the report and related amendment of studies S12-04027 and S13-04322. The limit of quantification for dimethoate and its metabolites in all olive matrices was set at 0.01 mg/kg. Samples were analysed within 5 months of collection.

Table 102 Residues of dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in olives in southern Europe during the 2017 growing season after 2 × 0.48 kg ai/ha (0.04 kg ai/100 L) foliar applications of a 400 g/L EC formulation (Gemrot-2018)

Trial No., Location, Year, (Variety)	No. (RTI, days)	Growth stage (BBCH)	Application rate (g a.i./ha)	Spray volume (L/ha)	Spray concentration (g a.i./100L)	DALA	Residues (mg/kg)							
							Dimethoate	Omethoate	DM + OM	DCA	ODOCA	DD	ODO	ODNDO
17-00467-01 Nea Skioni, Central Macedonia, Greece 2017 (Chondroelia Chalkidikis)	2 (9)	28 37	480 480	1200 1199	40.0 40.0	0	3.1	0.99	4.1	< 0.01	0.05	0.14	0.04	< 0.01
						7	0.05	1.10	1.2	< 0.01	0.07	0.16	0.03	< 0.01
						14	< 0.01	0.55	0.56	< 0.01	0.06	0.17	0.03	< 0.01
						21	< 0.01	0.36	0.37	< 0.01	0.05	0.24	0.04	< 0.01
						28	< 0.01	0.11	0.12	< 0.01	0.03	0.14	0.02	< 0.01
17-00467-02 Manzanilla, Huelva/Andalucia, Spain 2017 (Manzanillo)	2 (9)	28 37	485 502	1213 1254	40.0 40.0	0	1.4	0.11	1.5	< 0.01	< 0.01	0.03 (*c 0.01*)	0.04	< 0.01
						8	0.05	0.15	0.20	< 0.01	0.01	0.04	0.04	< 0.01
						14	< 0.01	0.08	0.09	< 0.01	0.02	0.04	0.04	< 0.01
						22	< 0.01	0.01	0.02	< 0.01	0.01	0.02	0.01	< 0.01
						28	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01	0.01	< 0.01	< 0.01
17-00467-03 Alcaucin, Malaga/Andalucia, Spain 2017 (Hojiblanca)	2 (10)	28 38	471 484	1178 1210	40.0 40.0	0	3.11	0.86	4.0	< 0.01	0.03	0.11	0.05	< 0.01
						7	1.07	1.04	2.1	< 0.01	0.05	0.11	0.04	< 0.01
						13	0.61	1.08	1.7	< 0.01	0.06	0.16	0.06	< 0.01
						21	0.11	0.57	0.68	< 0.01	0.05	0.13	0.05	< 0.01
						28	0.04	0.27	0.31	< 0.01	0.03	0.11	0.03	< 0.01
17-00467-04 Orta Nova, Puglia, Italy 2017 (Frantoio)	2 (10)	28 38	1340 1410	1114 1173	??	0	2.27	0.56	2.8	0.01	0.03	0.25	0.03	< 0.01
						7	0.83	0.80	1.6	< 0.01	0.04	0.33	0.03	< 0.01
						14	0.13	0.48	0.61	< 0.01	0.03	0.29	0.03	< 0.01
						21	0.02	0.27	0.29	< 0.01	0.03	0.29	0.03	< 0.01
						28	< 0.01	0.05	0.06	< 0.01	< 0.01	0.06	< 0.01	< 0.01

Trial No., Location, Year, (Variety)	No. (RTI, days)	Growth stage (BBCH)	Application rate (g a.i./ha)	Spray volume (L/ha)	Spray concentration (g a.i./100L)	DALA	Residues (mg/kg)							
							Dimethoate	Omethoate	DM + OM	DCA	ODOCA	DD	ODO	ODNDO
17-00467-05 S. Arcangelo Magione, Umbria, Italy 2017 (Frantoio)	2 (11)	28 39	491 478	1228 1195	40.0 40.0	28	0.05	0.34	0.39	< 0.01	0.06	0.07	0.06	< 0.01
17-00467-06 Andora, Liguria, Italy 2017 (Colombaia)	2 (11)	28 39	477 480	1193 1199	40.0 40.0	28	< 0.01	0.03	0.04	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
17-00467-07 Saint Sauveur de Cruzieres, Auvergne Rhone Alpes, France 2017 (Picholine)	2 (10)	27 37	486 484	1214 1211	40.0 40.0	27	< 0.01	0.21	0.22	< 0.01	0.02	0.10	0.03	< 0.01
17-00467-08 Zafarraya, Granada/Andalucia, Spain 2017 (Picual)	2 (10)	28 38	495 478	1238 1195	40.0 40.0	28	0.04	0.27	0.31	< 0.01	0.06	0.08	0.04	< 0.01

LOQ of dimethoate and metabolites = 0.01 mg/kg

DCA = Dimethoate carboxylic acid

ODOCA = O-desmethyl omethoate carboxylic acid

DD = Desmethyl dimethoate

ODO = O-desmethyl omethoate

ODNDO = O-desmethyl N-desmethyl omethoate

Note: Residues of O-desmethyl iso-dimethoate were < 0.01 mg/kg in all samples

Two trials were conducted in southern Europe during the 2009 growing season (Raufer - 2010b), this time with only a single application (0.48 kg ai/ha, 0.04 kg ai/100 L) being made at BBCH ~84, and samples of olives being collected from 0 to 42 days after application. Samples were frozen within 7 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Less mature fruit was homogenised whole (i.e. including the stones) before extraction, while the more mature fruit samples were separated into flesh and stones, with just the flesh extracted, but residues calculated on a whole fruit basis. Recoveries of dimethoate and omethoate from fortified control samples ranged from 67–88%. Samples were analysed within 3 months of collection.

Table 103 Residues of dimethoate and omethoate in olives in southern Europe after a single 0.48 kg ai/ha (0.04 kg ai/100 L) foliar application of a 400 g/L EC formulation (Raufer – 2010b)

Crop, variety, location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Spray concentration (kg ai/100 L)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Olives, Hojiblanca, 14548, Montalban de Cordoba, Andalusia, Spain, 2009	80	0.48	1200	0.04	0	Whole fruit	5.8	0.20
					28	Flesh [a]	0.04	0.14
					35	Flesh [a]	0.02	0.14
					42	Flesh [a]	< 0.01	0.10
Olives, Megaritiki, 64008, Kariani, Kavala, Greece, 2009	87	0.48	1196	0.04	0	Whole fruit	1.9	0.28
					28	Flesh [a]	0.11	0.22
					35	Flesh [a]	0.02	0.11
					42	Flesh [a]	0.01	0.08

[a] Stones separated and weighed and residues calculated on a whole fruit basis.

No residues were detected in any of the control samples.

A further two trials were conducted in southern Europe during the 2009 growing season (Raufer – 2010c), involving a bait spray treatment applied to branches, with approximately 12–25% of the tree surface being covered, using a hand sprayer equipped with a flat fan nozzle. A single application of a 400 g/L EC formulation was made, tank mixed with a protein bait, at target application rate of 0.06 kg ai/ha. Samples of olives were collected at 0, 28, 35 and 42 days of application. Samples were frozen within 7 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Less mature fruit was homogenised whole (i.e. including the stones) before extraction, while the more mature fruit samples were separated into flesh and stones, with just the flesh extracted, but residues calculated on a whole fruit basis. Recoveries of dimethoate and

omethoate from fortified control samples ranged from 65–91%. Samples were analysed within 3 months of collection.

Table 104 Residues of dimethoate and omethoate in olives in southern Europe after a single 0.06 kg ai/ha (0.30 kg ai/100 L) foliar bait spray application of a 400 g/L EC formulation (Raufer – 2010c)

Crop, variety, location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Spray volume (L/ha)	Spray conc. (kg ai/100 L)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
Olives, Hojiblanca, 14548, Montalban de Cordoba, Andalusia, Spain, 2009	80	0.061	20	0.30	0	Whole fruit	1.4	0.06	1.5
					28	Flesh [a]	0.05	0.03	0.08
					35	Flesh [a]	< 0.01	0.03	0.04
					42	Flesh [a]	< 0.01	0.01	0.02
Olives, Megaritiki, 64008, Kariani, Kavala, Greece, 2009	87	0.060	20	0.30	0	Whole fruit	< 0.01	< 0.01	< 0.02
					28	Flesh [a]	< 0.01	< 0.01	< 0.02
					35	Flesh [a]	< 0.01	< 0.01	< 0.02
					42	Flesh [a]	< 0.01	< 0.01	< 0.02

[a] Stones separated and weighed and residues calculated on a whole fruit basis.

No residues were detected in any of the control samples.

Assorted tropical and subtropical fruits – inedible peel

Avocados – foliar and post-harvest dipping treatment

A GLP residue trial was conducted on avocados at 6 test sites in Australia in 2013. Avocados at each site were treated with 3 pre-harvest foliar sprays of a 400 g/L EC formulation at 75 mL/100 L (30 g ai/100 L). The applications were made at 35, 15–21 and 7 days before harvest. Samples of avocados were collected at 0, 7 and 14 days after the last spray application. A sample collected at 7 days after the last spray application was also treated with a post-harvest dip of a 400 g/L EC product at 100 mL/100 L (40 g ai/100 L). Samples were stored frozen until analysis which was completed within approximately 6 months.

Samples were homogenised and extracted with acetonitrile containing 1% acetic acid. The extract was cooled in a freezer before the addition of magnesium sulphate and sodium acetate. The mixture was shaken, centrifuged and an aliquot of the supernatant cleaned up by addition of primary secondary amine and magnesium sulphate. The mixture was centrifuged and an aliquot of the supernatant diluted and analysed by LC-MS/MS. The LOQ for the method was 0.01 mg/kg for each of dimethoate and omethoate. Recoveries of dimethoate and omethoate from fortified control samples of avocado flesh and peel were within acceptable limits (70–110%).

Table 105 Residues of dimethoate and omethoate in avocodos after foliar and post-harvest dipping treatments (Lean – 2013)

Crop, variety, location year	Application rate (g ai/100 L)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Avocado, Hass, Alloway, Queensland, 2013	30 (foliar) + 40 (dip)	3 foliar (14) + 1 dip (7)		0	Flesh	0.17	0.032	0.17
					Peel	3.1	0.18	3.3
					Whole fruit (calculated)	0.75	0.067	0.82
Avocado, Sharwell, Alloway, Queensland, 2013	30 (foliar) + 40 (dip)	3 foliar (14) + 1 dip (7)		0	Flesh	0.067	< 0.01	0.078
					Peel	3.2	0.085	3.3
					Whole fruit (calculated)	0.49	0.019	0.51
Avocado, Hass, Nangiloc, Victoria, 2013	30 (foliar) + 40 (dip)	3 foliar (20, 8) + 1 dip (7)		0	Flesh	0.042	< 0.01	0.05
					Peel	2.3	0.047	2.4
					Whole fruit (calculated)	0.32	< 0.01	0.33
Avocado, Reed, Nangiloc, Victoria, 2013	30 (foliar) + 40 (dip)	3 foliar (20, 8) + 1 dip (7)		0	Flesh	0.062	< 0.01	0.073
					Peel	2.7	0.12	2.9
					Whole fruit (calculated)	0.44	0.025	0.46
Avocado, Bacon, Colignan, Victoria, 2013	30 (foliar) + 40 (dip)	3 foliar (14) + 1 dip (7)		0	Flesh	0.11	0.010	0.12
					Peel	2.2	0.060	2.2
					Whole fruit (calculated)	0.41	0.016	0.43
Avocado, Hass, Ravensbourne, Queensland, 2013	30 (foliar) + 40 (dip)	3 foliar (14) + 1 dip (7)		0	Flesh	0.062	< 0.01	0.073
					Peel	3.3 c0.015	0.173	3.5
					Whole fruit (calculated)	0.71	0.042	0.76

Except where noted otherwise, no residues were detected in any of the untreated control samples.

Table 106 Residues of dimethoate and omethoate in avocodos after foliar treatment (Lean – 2013)

Crop, variety, location year	Application rate (g ai/100 L)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Avocado, Hass, Alloway, Queensland, 2013	30 (foliar)	3 (14)		0	Flesh	0.11	0.017	0.13
					Peel	1.8	0.10	1.9
					Whole fruit (calculated)	0.40	0.032	0.44
				0 (duplicate)	Flesh	0.10	0.016	0.12
					Peel	1.6	0.10	1.7
					Whole fruit (calculated)	0.36	0.031	0.40
				7	Flesh	0.042	0.012	0.054
					Peel	0.079	0.023	0.10
					Whole fruit (calculated)	0.043	0.012	0.055
				14	Flesh	0.016	< 0.01	0.026
					Peel	0.062	0.025	0.087
					Whole fruit (calculated)	0.023	0.012	0.035
Avocado, Sharwell, Alloway, Queensland, 2013	30 (foliar)	3 (14)		0	Flesh	0.064	< 0.01	0.074
					Peel	1.2	0.053	1.24
					Whole fruit (calculated)	0.22	< 0.01	0.22
				0 (duplicate)	Flesh	0.059	< 0.01	0.069
					Peel	1.2	0.050	1.3
					Whole fruit (calculated)	0.21	< 0.01	0.22
				7	Flesh	< 0.01	< 0.01	< 0.02
					Peel	0.072	0.027	0.099
					Whole fruit (calculated)	0.015	< 0.01	0.025
				14	Flesh	< 0.01	< 0.01	< 0.02

Crop, variety, location year	Application rate (g ai/100 L)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					Peel	0.082	0.016	0.098
					Whole fruit (calculated)	0.016	< 0.01	0.026
Avocado, Reed, Nangiloc, Victoria, 2013	30 (foliar)	3 (20, 8)		0	Flesh	< 0.01	< 0.01	< 0.02
					Peel	0.46	0.27	0.73
					Whole fruit (calculated)	0.44	0.042	0.11
				0 (duplicate)	Flesh	< 0.01	< 0.01	< 0.02
					Peel	0.48	0.28	0.75
					Whole fruit (calculated)	0.074	0.043	0.12
				7	Flesh	< 0.01	< 0.01	< 0.02
					Peel	0.21	0.16	0.36
					Whole fruit (calculated)	0.031	0.023	0.054
				14	Flesh	< 0.01	< 0.01	< 0.02
					Peel	0.31	0.21	0.52
					Whole fruit (calculated)	0.054	0.037	0.091
Avocado, Hass, Nangiloc, Victoria, 2013	30 (foliar)	3 (20, 8)		0	Flesh	0.026	< 0.005	0.026
					Peel	1.3	0.11	1.4
					Whole fruit (calculated)	0.24	0.019	0.26
				0 (duplicate)	Flesh	0.026	< 0.01	0.036
					Peel	1.2	0.11	1.3
					Whole fruit (calculated)	0.22	0.019	0.24
				7	Flesh	0.015	< 0.01	0.025
					Peel	0.21	0.040	0.25

Crop, variety, location year	Application rate (g ai/100 L)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					Whole fruit (calculated)	0.040	< 0.01	0.046
				14	Flesh	< 0.01	< 0.01	< 0.02
					Peel	0.082	0.053	0.14
					Whole fruit (calculated)	0.009	0.008	0.016
Avocado, Bacon, Colignan, Victoria, 2013	30 (foliar)	3 (14)		0	Flesh	0.021	0.018	0.039
					Peel	0.32	0.03	0.35
					Whole fruit (calculated)	0.097	0.021	0.12
				0 (duplicate)	Flesh	0.021	0.018	0.039
					Peel	0.31	0.065	0.37
					Whole fruit (calculated)	0.085	0.028	0.11
				7	Flesh	< 0.01	< 0.01	< 0.02
					Peel	< 0.01	< 0.01	< 0.02
					Whole fruit (calculated)	< 0.01	< 0.01	< 0.02
				14	Flesh	< 0.01	0.014	0.024
					Peel	< 0.01	0.045	0.055
					Whole fruit (calculated)	< 0.01	0.018	0.019
Avocado, Hass, Ravensbourne, Queensland, 2013	30 (foliar)	3 (14)		0	Flesh	0.076	0.010	0.086
					Peel	2.5 c0.015	0.18	2.7
					Whole fruit (calculated)	0.60	0.047	0.64
				0 (duplicate)	Flesh	0.076	0.011	0.087

Crop, variety, location year	Application rate (g ai/100 L)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after treatment	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					Peel	2.4	0.18	2.5
					Whole fruit (calculated)	0.57	0.048	0.62
				7	Flesh	0.087	0.018	0.10
					Peel	0.93	0.16	1.1
					Whole fruit (calculated)	0.30	0.055	0.35
				14	Flesh	0.072	0.014	0.086
					Peel	0.33	0.13	0.45
					Whole fruit (calculated)	0.12	0.036	0.16

Except where noted otherwise, no residues were detected in any of the untreated control samples.

Mangoes – foliar and post-harvest dipping treatment

Five trials were conducted in mangoes in Queensland in the 2001/02 growing season (Clark – 2002). Mangoes were treated by foliar spray (3 × 0.03 kg ai/100 L dilute foliar sprays at 7-day intervals), post-harvest dipping at 0.04 kg/100 L, or both.

Stones, peel and flesh were separated and weighed. Samples were extracted by homogenisation with acetone, filtered and hexane and dichloromethane were added. The layers were separated and the aqueous layer was re-extracted with dichloromethane after addition of saturated sodium chloride. The organic layers were combined and dried in a rotary evaporate, reconstituted in acetone, dried again and made up to volume in acetone for analysis by GC-FPD. Concurrent recoveries were variable, with values for dimethoate ranging from 54–112% (mean = 84%, RSD = 19%) and for omethoate ranging from 60–129% (mean = 102%, RSD = 23%).

Table 107 Residues of dimethoate and omethoate in mangoes after foliar and post-harvest dipping treatments (Clark – 2002)

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Mango, Kensington Pride, Walkamin QLD, 2001	0.03 (foliar) + 0.04 (dip)	3 foliar (7, 7) + 1 dip (3)	2250	0	Peel	1.22	0.19	1.4
					Flesh	0.06	0.03	0.09
					Whole fruit (calculated)	0.34	0.06	0.40
Mango, Kensington	0.03 (foliar)	3 foliar (7, 7)	1920	0	Peel	0.90	< 0.02	0.92

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Pride, Walkamin, QLD, 2001	+ 0.04 (dip)	+ 1 dip (3)						
					Flesh	0.07	< 0.02	0.09
					Whole fruit (calculated)	0.26	< 0.02	0.28
Mango, Palmer, Tolga QLD, 2001/02	0.03 (foliar) + 0.04 (dip)	3 foliar (7, 7) + 1 dip (3)	333	0	Peel	1.1	0.20	1.3
					Flesh	0.04	< 0.02	0.06
					Whole fruit (calculated)	0.24	0.05	0.29
Mango, Kensington Pride, Tolga, QLD, 2001	0.03 (foliar) + 0.04 (dip)	3 foliar (7, 7) + 1 dip (3)	1920	0	Peel	0.70	< 0.02	0.72
					Flesh	0.48	0.02	0.50
					Whole fruit (calculated)	0.43	< 0.02	0.45
Mango, Palmer, Tolga, QLD, 2002	0.03 (foliar) + 0.04 (dip)	3 foliar (7, 7) + 1 dip (3)	333	0	Peel	0.47	0.04	0.51
					Flesh	0.09	0.03	0.12
					Whole fruit (calculated)	0.17	0.03	0.20

No residues were found above the LOQ in any of the untreated control samples.

Table 108 Residues of dimethoate and omethoate in mangoes after foliar treatments (Clark – 2002)

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Mango, Kensington Pride, Walkamin QLD, 2001	0.03 (foliar)	3 (7, 7)	2250	0	Peel	0.60	0.07	0.67
					Flesh	0.15	0.04	0.19
					Whole fruit (calculated)	0.27	0.04	0.31
				1	Peel	0.40	0.04	0.44

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					Flesh	0.10	0.03	0.13
					Whole fruit (calculated)	0.17	0.03	0.20
				3	Peel	0.24	0.07	0.31
					Flesh	0.09	0.04	0.13
					Whole fruit (calculated)	0.12	0.04	0.16
				7	Peel	0.06	0.04	0.10
					Flesh	0.05	0.03	0.08
					Whole fruit (calculated)	0.04	0.03	0.12
Mango, Kensington Pride, Walkamin, QLD, 2001	0.03 (foliar)	3 (7, 7)	1920	3	Peel	0.15	0.05	0.20
					Flesh	0.04	< 0.02	0.06
					Whole fruit (calculated)	0.06	0.02	0.08
Mango, Palmer, Tolga QLD, 2001/02	0.03 (foliar)	3 (7, 7)	333	0	Peel	< 0.02	< 0.02	< 0.04
					Flesh	< 0.02	< 0.02	< 0.04
					Whole fruit (calculated)	< 0.02	< 0.02	< 0.04
				1	Peel	0.10	0.16	0.26
					Flesh	< 0.02	< 0.02	< 0.04
					Whole fruit (calculated)	0.03	0.04	0.12
				3	Peel	0.03	< 0.02	0.05
					Flesh	< 0.02	< 0.02	< 0.04
					Whole fruit (calculated)	< 0.02	< 0.02	< 0.04
				7	Peel	< 0.02	< 0.02	< 0.04

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					Flesh	< 0.02	< 0.02	< 0.04
					Whole fruit (calculated)	< 0.02	< 0.02	< 0.04
Mango, Kensington Pride, Tolga, QLD, 2001	0.03 (foliar)	3 (7, 7)	1920	3	Peel	0.51	0.03	0.54
					Flesh	0.22	0.02	0.24
					Whole fruit (calculated)	0.25	0.02	0.27
Mango, Palmer, Tolga, QLD, 2002	0.03 (foliar)	3 (7, 7)	333	3	Peel	< 0.02	0.09	0.11
					Flesh	< 0.02	0.03	0.05
					Whole fruit (calculated)	< 0.02	0.04	0.06

No residues were found above the LOQ in any of the untreated control samples.

Table 109 Residues of dimethoate and omethoate in mangoes after post-harvest dipping treatment (Clark – 2002)

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Mango, Kensington Pride, Walkamin QLD, 2001	0.04 (dip)	1		0	Peel	0.89	0.09	0.98
					Flesh	0.02	< 0.02	0.04
					Whole fruit (calculated)	0.25	0.03	0.28
Mango, Kensington Pride, Walkamin, QLD, 2001	0.04 (dip)	1		0	Peel	0.56	< 0.02	0.58
					Flesh	0.09	< 0.02	0.11
					Whole fruit (calculated)	0.21	< 0.02	0.23
Mango, Palmer, Tolga QLD,	0.04 (dip)	1		0	Peel	0.16	< 0.02	0.18

Crop, variety, location year	Rate (kg ai/100 L)	No., (RTI, days)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
2001/02								
					Flesh	0.04	< 0.02	0.06
					Whole fruit (calculated)	0.06	< 0.02	0.08
Mango, Kensington Pride, Tolga, QLD, 2001	0.04 (dip)	1		0	Peel	0.31	< 0.02	0.33
					Flesh	0.17	< 0.02	0.19
					Whole fruit (calculated)	0.18	< 0.02	0.20
Mango, Palmer, Tolga, QLD, 2002	0.04 (dip)	1		0	Peel	0.22	< 0.02	0.24
					Flesh	0.15	< 0.02	0.17
					Whole fruit (calculated)	0.15	< 0.02	0.17

No residues were found above the LOQ in any of the untreated control samples.

Onions, bulb

A series of trials was conducted in bulb onions at sites in northern and southern Europe during the 2008 and 2009 growing seasons (Raufer – 2009e, Raufer – 2009f, and Raufer – 2010d). 2 × 0.24 kg ai/ha foliar applications of a 400 g/L dimethoate EC formulation were made using a knapsack sprayer at a re-treatment interval of 10 days late in the season (with the final application timed for 2 weeks before harvest maturity), with whole plants or harvest mature bulbs (depending on the sampling interval) being collected at intervals from 0 to 21 days after application. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method. Recoveries of dimethoate and omethoate from fortified control samples ranged from 76–103%. Samples were analysed within 7 months of collection.

Table 110 Residues of dimethoate and omethoate in bulb onions in Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009e)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
Onions (Hyfield), 45300, Enzanville, Centre Val de Loire, France, 2008	2 (10)	41-42, 44-45	0.25, 0.24	300, 291	0	Whole plant	0.84	0.02	0.86
					3	Whole plant	0.15	0.03	0.18
					7	Whole	0.08	0.03	0.11

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
						plant			
					10	Whole plant	0.04	0.02	0.06
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002
Onions (Rijnsburger Gold), WS14, Brownhills, Staffordshire, UK, 2008	2 (9)	47-48, 47-48	0.24, 0.24	302, 302	0	Whole plant	0.50	< 0.01	0.51
					3	Whole plant	0.21	0.02	0.23
					7	Whole plant	0.02	< 0.01	0.03
					10	Whole plant	< 0.01	< 0.01	< 0.02
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002
Onions (Takmark), 69124, Heidelberg, Baden-Württemberg, Germany, 2008	2 (11)	45, 46	0.26, 0.25	320, 313	0	Whole plant	0.57	< 0.01	0.58
					3	Whole plant	0.19	0.03	0.22
					7	Whole plant	0.03	< 0.01	0.04
					10	Whole plant	0.02	< 0.01	0.03
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002
Onions (Red Baron), 27449, Kutenholz, Niedersachsen, Germany, 2008	2 (10)	48, 49	0.25, 0.24	309, 302	0	Whole plant	1.6	0.07	1.68
					3	Whole plant	0.23	0.08	0.32
					7	Whole plant	0.16	0.04	0.20
					10	Whole plant	0.07	0.03	0.10
					14	Bulb	0.005	0.001	0.006
					21	Bulb	0.002	< 0.001	< 0.003
Onions (Moranta), 57012,	2 (10)	45-47, 47	0.24, 0.25	302, 307	0	Whole plant	0.36	0.02	0.38

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
Marathoussa, Chalkidiki, Greece, 2008									
					3	Whole plant	0.02	< 0.01	0.03
					7	Whole plant	< 0.01	< 0.01	< 0.02
					10	Whole plant	< 0.01	< 0.01	< 0.02
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002
Onions (Derek), 40057, Granarolo dell Emilia, Emilia-Romagna, Italy, 2008	2 (10)	42, 45-47	0.23, 0.25	285, 308	0	Whole plant	0.66	0.01	0.67
					3	Whole plant	0.03	0.01	0.04
					7	Whole plant	< 0.01	< 0.01	< 0.02
					10	Whole plant	< 0.01	< 0.01	< 0.02
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002
Onions (Cometa), 40023, Castel Guelfo di Bologna, Emilia-Romagna, Italy, 2008	2 (10)	42, 45	0.24, 0.24	300, 302	0	Whole plant	0.87	0.02	0.89
					3	Whole plant	0.12	0.02	0.14
					7	Whole plant	0.02	< 0.01	< 0.03
					10	Whole plant	< 0.01	< 0.01	< 0.02
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002
Onions (Furia), 66250, Saint-Laurent-de-la-Salanque, Languedoc-Roussillon, France, 2009	2 (10)	19-41, 42	0.25, 0.23	313, 293	0	Whole plant	1.8	0.04	1.84
					3	Whole plant	0.14	0.03	0.17
					7	Whole plant	0.05	0.02	0.07
					10	Whole	0.02	0.01	0.03

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Residues (mg/kg)		
							DM	OM	DM + OM
						plant			
					14	Bulb	< 0.001	< 0.001	< 0.002
					21	Bulb	< 0.001	< 0.001	< 0.002

No residues were detected in any of the untreated control samples.

Brassica vegetables

A series of trials was conducted in Brussels sprouts (Raufer – 2009g, Raufer – 2010e and Raufer – 2011b), broccoli (Raufer-2009h and Raufer – 2010f), head cabbage (Raufer – 2009i) and cauliflower (Raufer – 2009k, Raufer – 2010h, and Raufer – 2010h) at sites in northern Europe during the 2008, 2009 and 2010 growing seasons. 2 × 0.24 kg ai/ha foliar applications of a 400 g/L dimethoate EC formulation were made using a knapsack sprayer at a re-treatment interval of 7–10 days late in the season (with the final application timed for 2–3 weeks before harvest maturity), with sprouts (Brussels sprouts), whole plants or inflorescences depending on maturity (broccoli and cauliflower), or whole plants or heads (cabbage) being collected at intervals from 0 to up to 28 days after application. Samples were frozen within 7 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method with analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Recoveries of dimethoate and omethoate from fortified control samples ranged from 67–110% (Brussels sprouts), 72–100% (broccoli), 76–91% (cabbage), and 68–102% (cauliflower). Samples were analysed within 9 months of collection.

Table 111 Residues of dimethoate and omethoate in broccoli in northern Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009h and Raufer – 2010f)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Broccoli (Monaco), 56920, Kerfourn, Brittany, France, 2008	2 (7)	40, 41	0.22, 0.23	371, 384	0	Whole plant	1.5	0.02	1.5
					7	Whole plant	0.17	0.03	0.20
					10	Inflorescence	0.07	0.02	0.09
					14	Inflorescence	< 0.01	< 0.01	< 0.02
					21	Inflorescence	< 0.01	< 0.01	< 0.02
Broccoli (Parthenon), 25348, Blomecsche Wildnis, Schleswig-Holstein, Germany, 2008	2 (7)	41, 41	0.24, 0.25	407, 415	0	Whole plant	1.9	0.03	1.9
					7	Whole plant	0.15	0.04	0.19
					10	Inflorescence	0.03	< 0.01	0.04
					14	Inflorescence	0.01	< 0.01	0.02

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					21	Inflorescence	< 0.01	< 0.01	< 0.02
Broccoli (Ironman), 67880, Innenheim, Alsace, France, 2009	2 (7)	41, 42	0.26, 0.25	433, 409	0	Whole plant	6.6	0.15	6.8
					21	Inflorescence	< 0.01	< 0.01	< 0.02
Broccoli (Steel), PR4, Hesketh Bank, Lancashire, UK, 2009	2 (7)	28-29, 29-41	0.24, 0.24	402, 400	0	Whole plant	7.9	0.06	8.0
					21	Inflorescence	< 0.01	< 0.01	< 0.02

No residues were detected in any of the untreated control samples.

Table 112 Residues of dimethoate and omethoate in cauliflower in northern Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009i, Raufer – 2010g, and Raufer – 2010h)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Cauliflower (Cortés), 56250, Elven, Bretagne, France, 2008	2 (7)	41, 42	0.23, 0.23	385, 386	0	Whole plant	1.2	0.04	1.2
					3	Whole plant	< 0.01	< 0.01	< 0.02
					7	Whole plant	0.26	0.05	0.31
					10	Inflorescence	< 0.01	< 0.01	< 0.02
					14	Inflorescence	< 0.01	< 0.01	< 0.02
					21	Inflorescence	< 0.01	< 0.01	< 0.02
					28	Inflorescence	< 0.01	< 0.01	< 0.02
Cauliflower (Clapton SG), 25348, Glückstadt, Schleswig-Holstein, Germany, 2008	2 (7)	39, 41	0.24, 0.24	401, 395	0	Whole plant	1.5	0.06	1.6
					3	Whole plant	0.82	0.12	0.95

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					7	Whole plant	0.38	0.13	0.52
					10	Inflorescence	0.02	< 0.01	0.03
					14	Inflorescence	0.01	< 0.01	0.02
					21	Inflorescence	< 0.01	< 0.01	< 0.02
					28	Inflorescence	< 0.01	< 0.01	< 0.02
Cauliflower (Dexter), 71277, Perouse-Rutesheim, Baden-Württemberg, Germany, 2008	2 (7)	41, 42	0.24, 0.23	402, 385	0	Whole plant	4.7	0.15	4.8
					3	Whole plant	1.3	0.14	1.4
					7	Whole plant	1.2	0.24	1.4
					10	Inflorescence	0.05	< 0.01	0.06
					14	Inflorescence	0.02	< 0.01	0.03
					21	Inflorescence	0.01	< 0.01	0.02
					28	Inflorescence	< 0.01	< 0.01	< 0.02
Cauliflower (Glacier), CH1, Sealand, Cheshire, UK, 2008	2 (6)	45, 45	0.25, 0.24	410, 403	0	Whole plant	3.5	0.20	3.7
					3	Whole plant	2.1	0.30	2.4
					7	Whole plant	1.1	0.27	1.4
					10	Inflorescence	0.07	< 0.01	0.08
					14	Inflorescence	0.10	< 0.01	0.11
					21	Inflorescence	0.13	< 0.01	0.14
					28	Inflorescence	0.08	< 0.01	0.09
Cauliflower (Korlanu), 67880, Innenheim, Alsace, France, 2009	2 (7)	43, 45	0.25, 0.25	413, 420	0	Whole plant	3.5	0.06	3.6

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					21	Inflorescence	0.01	< 0.01	0.02
Cauliflower (Optimist), PR4, Hesketh Bank, Lancashire, UK, 2009	2 (7)	41, 41-43	0.24, 0.24	407, 400	0	Whole plant	3.0	0.05	3.0
					21	Inflorescence	< 0.01	< 0.01	< 0.02
Cauliflower (Dexter), 71277, Rutesheim-Perouse, Baden-Württemberg, Germany, 2009	2 (7)	42, 43	0.23, 0.25	390, 412	0	Whole plant	3.4	0.22 c0.01	3.6
					21	Inflorescence	< 0.01	< 0.01	< 0.02
Cauliflower (Barcelona), 64-600, Uscikowo, Wielkopolska, Poland, 2009	2 (8)	39, 39	0.24, 0.25	398, 407	0	Whole plant	4.1	0.14	4.2
					21	Inflorescence	0.01	< 0.01	0.02
Cauliflower (Lecanu), D-25348, Blomesche Wildnis, Schleswig-Holstein, Germany, 2010	2 (7)	39, 41-43	0.24, 0.24	400, 406	0	Whole plant	2.2	0.05	2.2
					3	Whole plant	0.66	0.11	0.77
					7	Whole plant	0.36	0.10	0.46
					10	Inflorescence	0.02	< 0.01	0.03
					14	Inflorescence	< 0.01	< 0.01	< 0.02
					21	Inflorescence	< 0.01	< 0.01	< 0.02
					28	Inflorescence	< 0.01	< 0.01	< 0.02

No residues were detected in any of the untreated control samples, except where otherwise indicated.

Table 113 Residues of dimethoate and omethoate in Brussels sprouts in northern Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009g and Raufer – 2010e)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Brussels sprouts (Millennium), B76 0DF, Curdworth, Warwickshire, UK, 2008	2 (7)	45, 45	0.25, 0.24	412, 399	0	0.12	< 0.01	0.13
					3	0.08	< 0.01	0.09
					7	0.03	< 0.01	0.04
					10	0.02	< 0.01	0.03
					14	0.02	< 0.01	0.03
					21	0.02	< 0.01	0.03
					28	0.01	< 0.01	0.02
Brussels sprouts (Genius), 71277, Rutesheim-Perouse, Baden-Württemberg, Germany, 2008	2 (7)	45, 46	0.25, 0.25	413, 410	0	0.27	< 0.01	0.28
					3	0.16	< 0.01	0.17
					7	0.11	< 0.01	0.12
					10	0.06	< 0.01	0.07
					14	0.02	< 0.01	0.03
					21	0.01	< 0.01	0.02
					28	< 0.01	< 0.01	0.02
Brussels sprouts (Genius), 27478, Altenbruch, Niedersachsen, Germany, 2008	2 (7)	45, 46	0.25, 0.25	410, 408	0	0.34	< 0.01	0.35
					3	0.24	< 0.01	0.25
					7	0.14	< 0.01	0.15
					10	0.08	< 0.01	0.09
					14	0.06	< 0.01	0.07
					21	0.02	0.01	0.03
					28	< 0.01	< 0.01	< 0.02
Brussels sprouts (Cyrus), 5463, Fjelstad, Fyn, Denmark, 2008	2 (7)	45, 46	0.24, 0.24	405, 402	0	0.17	< 0.01 (ND)	0.17
					3	0.09	< 0.01	0.10
					7	0.05	< 0.01	0.06
					10	0.03	< 0.01	0.04
					14	0.02	< 0.01	0.03
					21	0.02	< 0.01	0.03

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					28	0.01	< 0.01	0.02
Brussels sprouts (Hellimus), PR9 8DX, Banks, Lancashire, UK, 2009	2 (7)	43, 45	0.25, 0.25	413, 413	0	0.06	< 0.01	0.07
					14	< 0.01	< 0.01	< 0.02
					21	< 0.01	< 0.01	< 0.02
					28	< 0.01	< 0.01	< 0.02
Brussels sprouts (Clodius), 5463, Harndrup, Funen, Denmark, 2009	2 (7)	46, 46-47	0.24, 0.24	398, 387	0	0.13	< 0.01	0.14
					14	0.01	< 0.01	0.02
					21	< 0.01	< 0.01	< 0.02
					28	< 0.01	< 0.01	< 0.02
Brussels sprouts (Genius), 27478, Altenbruch, Niedersachsen, Germany, 2009	2 (7)	43, 46-47	0.25, 0.24	412, 396	0	0.23	< 0.01	0.24
					14	0.03	< 0.01	0.04
					21	< 0.01	< 0.01	< 0.02
					28	< 0.01	< 0.01 (ND)	< 0.01
Brussels sprouts (Crispus), 03096, Burg/Spreewald, Brandenburg, Germany, 2009	2 (10)	45, 46	0.26, 0.25	432, 420	0	0.22	< 0.01	0.23
					14	0.05	< 0.01	0.06
					21	0.03	< 0.01	0.04
					28	< 0.01	< 0.01	< 0.02
Brussels sprouts (Genius), D-27478, Cuxhaven-Altenbruch, Niedersachsen, Germany, 2010	2 (6)	48, 49	0.24, 0.25	393, 413	0	0.29	< 0.01	0.29
					14	0.04	< 0.01	0.05
					21	0.03	< 0.01	0.04
					28	0.01	< 0.01	< 0.02

No residues were detected in any of the untreated control samples.

Table 144 Residues of dimethoate and omethoate in head cabbage in northern Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009i)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Head cabbage (Lennox), 45570, Dampierre en Burly, Centre, France, 2008	2 (10)	44, 46	0.25, 0.25	393, 398	0	Whole plant	0.73	0.03	0.76
					3	Whole plant	0.49	0.05	0.54
					7	Whole plant	0.44	0.12	0.56
					10	Whole plant	0.10	0.04	0.14
					14	Heads with wrapper leaves	< 0.01	< 0.01	< 0.02
Head cabbage (Attraction), PE22, Langrick, Lincolnshire, UK, 2008	2 (10)	47, 47	0.25, 0.24	414, 404	0	Whole plant	0.06	< 0.01	0.07
					3	Whole plant	0.03	< 0.01	0.04
					7	Whole plant	0.03	< 0.01	0.04
					10	Whole plant	0.05	0.02	0.07
					14	Heads with wrapper leaves	< 0.01	< 0.01	< 0.02
Head cabbage (Kraut-Kaiser), 71277, Rutesheim-Perouse, Baden-Württemberg, Germany, 2008	2 (10)	47, 48	0.25, 0.25	416, 418	0	Whole plant	0.36	0.02	0.38
					3	Whole plant	0.12	0.02	0.14
					7	Whole plant	0.07	0.03	0.10
					10	Whole plant	0.02	< 0.01	0.03
					14	Heads with wrapper	< 0.01	< 0.01	< 0.02

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
						leaves			
Head cabbage (Impala), 5400, Bogense, Fyn, Denmark, 2008	2 (10)	46, 47	0.24, 0.24	398, 400	0	Whole plant	1.1	0.02	1.1
					3	Whole plant	0.25	0.02	0.27
					7	Whole plant	0.12	0.03	0.15
					10	Whole plant	0.14	0.04	0.18
					14	Heads with wrapper leaves	< 0.01	< 0.01	< 0.02

No residues were detected in any of the untreated control samples.

A second series of trials was conducted in head cabbage (Raufer – 2009j) at sites in southern Europe during the 2008 growing season. A single 0.24 kg ai/ha foliar application of a 400 g/L dimethoate EC formulation was made using a small plot boom sprayer late in the season (with the final application timed for 2 weeks before harvest maturity), with sprouts (Brussels sprouts), whole plants or inflorescences depending on maturity (broccoli), whole plants or heads (cabbage) being collected at intervals from 0 to 14 days after application. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent and magnesium sulfate, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Recoveries of dimethoate and omethoate from fortified control samples 66–93% (cabbage). Samples were analysed within 6 months of collection.

Table 155 Residues of dimethoate and omethoate in head cabbage in southern Europe after a single 0.24 kg ai/ha foliar application of a 400 g/L EC formulation (Raufer – 2009j)

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Head cabbage (Milan), 66700, Angeles sur Mer, Languedoc-Roussillon, France, 2008	47	0.26	417	0	Whole plant	0.09	< 0.01 (ND)
				3	Whole plant	0.05	< 0.01 (ND)
				7	Whole plant	0.16	0.02
				10	Whole plant	0.09	< 0.01
				14	Heads with wrapper leaves	< 0.01	< 0.01 (ND)
Head cabbage (Grand slam),	48-49	0.24	397	0	Whole plant	0.72	< 0.01

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
57008, Nea-Magnisia, Thessaloniki, Greece, 2008							
				3	Whole plant	0.11	0.03
				7	Whole plant	0.08	0.03
				10	Whole plant	< 0.01	< 0.01
				14	Heads with wrapper leaves	< 0.01 (ND)	< 0.01 (ND)
Head cabbage (Sentinel), 46822, Bolbaite, Valencia, Spain, 2008	47	0.22	360	0	Whole plant	2.6	0.01
				3	Whole plant	1.2	0.04
				7	Whole plant	0.30	0.05
				10	Whole plant	0.33	0.08
				14	Heads with wrapper leaves	< 0.01	< 0.01 (ND)
Head cabbage (OS Cross), 40057, Granarolo, Emilia-Romagna, Italy, 2008	45-47	0.22	374	0	Whole plant	0.94	< 0.01
				3	Whole plant	0.10	< 0.01
				7	Whole plant	0.03	< 0.01
				10	Whole plant	0.03	< 0.01
				14	Heads with wrapper leaves	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the untreated control samples.

Melons (except watermelons)

A series of trials was conducted in melons (Wilson – 2001a and Wilson – 2002a) at sites in southern Europe during the 2000 and 2001 growing seasons. 2 × 0.06 kg ai/100 L (in a nominal spray volume of 1000 L/ha) foliar applications of a 400 g/L dimethoate EC formulation were made using a knapsack sprayer at a re-treatment interval of 10–15 days late in the season (with the final application timed for 3 weeks before harvest maturity), with samples being collected at 0 and 7 days after application (opposite quarters from 12 melons) and 14, 21 and 28 days after application (opposite quarters from 12 melons, separated into peel and pulp). Samples were frozen within 8 hours of collection. Melon (whole fruit, peel and pulp) samples were extracted twice by maceration with dichloromethane. The samples were centrifuged and the solvent extracts combined, diluted to volume and an aliquot evaporated to dryness. The sample was reconstituted in hexane, and partitioned into water. The water extract was partitioned a second time with hexane and the aqueous phase diluted to volume before analysis by LC-MS (LOQ = 0.01 mg/kg for both analytes). Recoveries of dimethoate and omethoate

from fortified control samples ranged from 71–110%. Samples were analysed within 8 months of collection.

Table 116 Residues of dimethoate and omethoate in melons in southern Europe after 2 × 0.062 kg ai/100 L foliar applications of a 400 g/L EC formulation (Wilson – 2001a and Wilson – 2002a)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
Melon (Jaguar), Mantova, Italy, 2000	2 (14)	71, 79	0.062, 0.062	0.62, 0.63	999, 1009	0	Whole fruit	0.22	0.01
						7	Whole fruit	0.10	0.02
						14	Peel	< 0.01	< 0.01
						14	Pulp	< 0.01	< 0.01
						14	Whole fruit	< 0.01	< 0.01
						21	Peel	< 0.01 (ND)	< 0.01
						21	Pulp	< 0.01 (ND)	< 0.01 (ND)
						21	Whole fruit	< 0.01 (ND)	< 0.01
						28	Peel	< 0.01 (ND)	< 0.01 (ND)
						28	Pulp	< 0.01 (ND)	< 0.01 (ND)
						28	Whole fruit	< 0.01 (ND)	< 0.01 (ND)
Melon (Supermarket), Cantu, Italy, 2000	2 (11)	79, 81	0.062, 0.062	0.64, 0.62	1022, 999	0	Whole fruit	0.41	< 0.01
						7	Whole fruit	0.34	0.20
						14	Peel	0.15	0.02
						14	Pulp	0.05	< 0.01
						14	Whole fruit	0.11	0.01
						21	Peel	0.03	< 0.01
						21	Pulp	< 0.01	< 0.01
						21	Whole fruit	0.02	< 0.01
						28	Peel	0.03	< 0.01
						28	Pulp	< 0.01	< 0.01
						28	Whole fruit	0.02	< 0.01
Melon (Amarillo), La Aljorra, Spain, 2000	2 (10)	69-71, 75	0.062, 0.062	0.62, 0.62	987, 1000	0	Whole fruit	0.04	< 0.01

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
						7	Whole fruit	< 0.01	< 0.01
						13	Peel	< 0.01 (ND)	0.01
						13	Pulp	< 0.01	< 0.01
						13	Whole fruit	< 0.01	< 0.01
						20	Peel	< 0.01 (ND)	< 0.01 (ND)
						20	Pulp	< 0.01 (ND)	< 0.01 (ND)
						20	Whole fruit	< 0.01 (ND)	< 0.01 (ND)
						27	Peel	< 0.01 (ND)	< 0.01 (ND)
						27	Pulp	< 0.01 (ND)	< 0.01 (ND)
						27	Whole fruit	< 0.01 (ND)	< 0.01 (ND)
Melon (Thrakiotiko), Loutoufi, Greece, 2000	2 (15)	77, 82	0.062, 0.062	0.63, 0.63	1007, 1010	0	Whole fruit	0.11	< 0.01 (ND)
						7	Whole fruit	< 0.01	< 0.01 (ND)
						14	Peel	< 0.01	< 0.01
						14	Pulp	< 0.01	< 0.01
						14	Whole fruit	< 0.01	< 0.01
						21	Peel	< 0.01	< 0.01
						21	Pulp	< 0.01 (ND)	< 0.01 (ND)
						21	Whole fruit	< 0.01	< 0.01
						28	Peel	< 0.01	< 0.01
						28	Pulp	< 0.01	< 0.01 (ND)
						28	Whole fruit	< 0.01	< 0.01
Melon (Yakura), Rodigo, Italy, 2001	2 (12)	69, 71	0.062, 0.062	0.63, 0.62	1012, 987	0	Whole fruit	0.26	< 0.01
						23	Peel	< 0.01	< 0.01
						23	Pulp	< 0.01	< 0.01 (ND)
						23	Whole fruit	< 0.01	< 0.01

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Portion analysed	Dimethoate (mg/kg)	Omethoate (mg/kg)
						30	Peel	< 0.01	< 0.01
						30	Pulp	< 0.01	< 0.01
						30	Whole fruit	< 0.01	< 0.01
Melon (Dolmen), Rodigo, Italy, 2001*	2 (14)	69, 72	0.062, 0.062	0.63, 0.63	1005, 1005	0	Whole fruit	0.09	< 0.01
						21	Peel	0.04	0.02
						21	Pulp	0.02	< 0.01
						21	Whole fruit	0.03	0.01
						28	Peel	0.01	0.01
						28	Pulp	0.01	< 0.01
						28	Whole fruit	0.01	< 0.01
Melon (Galia), La Aljorra, Spain, 2001	2 (15)	NA, 81	0.062, 0.062	0.66, 0.61	1062, 970	0	Whole fruit	0.17	0.01
						20	Peel	< 0.01	< 0.01
						20	Pulp	< 0.01 (ND)	< 0.01
						20	Whole fruit	< 0.01	< 0.01
						27	Peel	< 0.01 (ND)	< 0.01 (ND)
						27	Pulp	< 0.01 (ND)	< 0.01 (ND)
						27	Whole fruit	< 0.01 (ND)	< 0.01 (ND)
Melon (Galia), Albujon, Spain, 2001	2 (14)	NA, 81	0.062, 0.062	0.61, 0.64	985, 1027	0	Whole fruit	0.15	< 0.01 (ND)
						20	Peel	< 0.01	< 0.01
						20	Pulp	< 0.01 (ND)	< 0.01
						20	Whole fruit	< 0.01	< 0.01
						27	Peel	< 0.01 (ND)	< 0.01 (ND)
						27	Pulp	< 0.01 (ND)	< 0.01 (ND)
						27	Whole fruit	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the untreated control samples.

*Applications and samplings at second Rodigo trial were more than 1 month after those at the first Rodigo trial.

Fruiting vegetables, other than Cucurbits

Eggplant

Trials were conducted in field grown eggplant in Australia (Lean – 2010). 3 × 0.03 kg ai/100 L dilute foliar applications of a 400 g/L EC formulation were made to the point of run-off at 7-day intervals. Fruit was collected at intervals from 1 to 7 days after the last application. Analyses were completed within 6 months of collection.

Table 117 Residues of dimethoate and omethoate in Australia – 2008 trials (Lean – 2010)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Eggplant (Black Prince) – field grown, Fredericksfield, QLD, Australia, 2008	3 (7, 7)		0.03, 0.03, 0.03	0.19, 0.17, 0.18	626, 553, 603	1	0.62	0.08
						4	0.18	0.07
						6	0.28	0.05
						7	0.35	0.10
Eggplant (Chiha) – field grown, Meadowvale, QLD, Australia, 2008	3 (6, 8)		0.03, 0.03, 0.03	0.32, 0.31, 0.30	1053, 1033, 983	1	0.41	0.11
						3	0.22	0.07
						5	0.09	0.04
						7	0.13	0.09

No residues were found above the LOQ in untreated control samples.

Peppers, sweet (capsicum) – foliar and post-harvest treatment

Trials were conducted in capsicum in Australia in 2002 (dal Santo – 2002a and dal Santo – 2002 b), with fruit treated with both foliar and post-harvest dipping applications of a 400 g/L EC formulation. Crops were treated with 3 foliar applications of the product at 0.3 kg ai/ha at 7-day intervals. A post-harvest spray application was made at 0.04 kg ai/100 L to the treated capsicums 7 days after the last foliar application and to untreated capsicums. Samples were collected at 0, 1, 3 and 7 days after the 3rd foliar spray and also after the post harvest spray. Samples were stored frozen until analysis which was within 3 months.

Samples were homogenised with acetone. The extract was filtered and hexane/dichloromethane (1:1) added. After mixing in a separatory funnel the organic layer was collected and concentrated on a rotary evaporator. The aqueous layer was re-extracted with dichloromethane. The combined organic extracts were concentrated and resuspended in acetone prior to analysis by gas chromatography with a phosphorus specific flame photometric detector. The LOQ was 0.02 mg/kg for dimethoate and 0.04 mg/kg for omethoate. Recoveries of dimethoate and omethoate were acceptable (89–115%).

Table 118 Residues of dimethoate and omethoate in capsicums after foliar and post-harvest spray application (dal Santo – 2002a and dal Santo – 2002b)

Crop, variety, location	No. (RTI, days)	Method	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Capsicum, Aries, Bowen, QLD, Australia, 2002	3 (7, 7)	Foliar spray	0.3	250	0	0.16	< 0.04	0.20
					1	0.10	< 0.04	0.14
					3	0.12	< 0.04	0.16
					7	0.14	< 0.04	0.18
	3 (7, 7) + 1 (7)	3 × foliar spray + 1 × post-harvest spray	3 × 0.3 (foliar) + 1 × 0.04 kg ai/100 L (post harvest)	250 (foliar)	0	1.75	< 0.04	1.8
	1	1 × post-harvest spray	0.04 kg ai/100 L (post-harvest spray)		0	1.26	< 0.04	1.3
Capsicum, Merlin, Emerald Creek, QLD, Australia, 2002	3 (7, 7)	Foliar spray	0.03	518, 485, 510	0	0.05	< 0.02	0.07
					3	0.04	0.02	0.06
					5	0.03	0.02	0.05
	3 (7, 7) + 1 (0)	3 × foliar spray + 1 × post-harvest spray	3 × 0.3 (foliar) + 1 × 0.04 kg ai/100 L (post harvest)	518, 485, 510 (foliar)	7 (pre- and post-harvest)	2.2, 1.7	< 0.02, < 0.02	2.2, 1.7
	1	1 × post-harvest spray	0.04 kg ai/100 L (post-harvest spray)		7 (post-harvest)	1.5	< 0.02	1.5
Capsicum, Aries, Bowen, QLD, Australia, 2002*	3 (7, 7)	Foliar spray	0.3	496, 509, 498	0	0.15	0.02	0.17
					3	0.08	< 0.02	0.10
					5	0.04	0.02	0.06
					7	0.03	< 0.02	0.05
	3 (7, 7) + 1 (7)	3 × foliar spray + 1 × post-harvest spray	3 × 0.3 (foliar) + 1 × 0.04 kg ai/100 L (post harvest)	496, 509, 498 (foliar)	7 (pre-harvest) 0 (post harvest)	0.23	< 0.02	0.25
	1	1 × post-harvest spray	0.04 kg ai/100 L (post-harvest spray)		0	0.27, 0.17	< 0.02, < 0.02	0.29, 0.19

*Trial more than one month after the other Bowen trial.

No residues were found above the LOQ in any of the untreated control samples.

Table 119 Residues of dimethoate and omethoate in capsicum in Australia – 2008 trials (Lean – 2010)

Crop (variety), location, year	No. (RTI, days)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Capsicum (Inspiration) – greenhouse hydroponic grown, Kindred, TAS, Australia, 2008	3 (7, 6))	0.03, 0.03, 0.03	0.16, 0.15, 0.17	524, 505, 570	0	0.66	0.13	0.79
					3	0.32	0.11	0.43
					7	0.15	0.11	0.26
					14	0.05	0.05	0.10
Capsicum (Warlock) – field grown, Gatton, QLD, Australia, 2008	3 (6, 7)	0.03, 0.03, 0.03	0.32, 0.29, 0.31	1066, 966, 1033	0	0.30	0.04	0.34
					3	0.15	0.04	0.19
					7	0.04	0.02	0.06
					14	0.02	< 0.01	0.03

No residues were found above the LOQ in untreated control samples.

Tomatoes

A series of trials was conducted in field grown tomatoes (Wilson – 2001b and Wilson – 2002b) at sites in southern Europe during the 2000 and 2001 growing seasons. 2 × 0.1 kg ai/100 L (in a nominal spray volume of 600 L/ha) foliar applications of a 400 g/L dimethoate EC formulation were made using a knapsack sprayer at a re-treatment interval of 10–15 days late in the season (with the final application timed for 3 weeks before harvest maturity), with samples being collected at 0–28 days after application. Samples were frozen within 8 hours of collection. Samples were extracted twice by maceration with dichloromethane. The samples were centrifuged and the solvent extracts combined, diluted to volume and an aliquot evaporated to dryness. The sample was reconstituted in hexane, and partitioned into water. The water extract was partitioned a second time with hexane and the aqueous phase diluted to volume before analysis by LC-MS (LOQ = 0.01 mg/kg for both analytes). Recoveries of dimethoate and omethoate from fortified control samples ranged from 71–104%. Samples were analysed within 6 months of collection.

Table 120 Residues of dimethoate and omethoate in field grown tomatoes in southern Europe after 2 × 0.10 kg ai/100 L foliar applications of a 400 g/L EC formulation (Wilson – 2001b and Wilson – 2002b)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Tomatoes	2	69, 72	0.10,	0.64, 0.63	615,	0	0.30 (0.38,	0.02 (0.02,	0.32 (0.40,

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
(690), Stagno Lombardo, Italy, 2000	(14)		0.10		607		0.22)	0.02)	0.24)
						7	< 0.01	< 0.01 (0.01, < 0.01)	< 0.02(0.02, < 0.02)
						14	< 0.01	< 0.01	< 0.02
						21	≤ 0.01	≤ 0.01	≤ 0.02
						28	< 0.01	< 0.01	< 0.02
Tomatoes (PS 1296), Mulazzano Lodi, Italy, 2000	2 (14)	69, 72	0.10, 0.10	0.63, 0.63	607, 609	0	0.65 (0.43, 0.88)	0.02	0.68 (0.45, 0.90)
						7	0.02	0.02	0.04
						14	< 0.01	0.02	0.03
						21	≤ 0.01	0.01	0.02
						28	< 0.01	< 0.01	< 0.02
Tomatoes (Bodar), Miranda, Spain, 2000	2 (15)	71, 73	0.10, 0.10	0.62, 0.62	598, 596	0	0.33	0.02	0.35
						7	0.01	0.02	0.03
						14	< 0.01	0.02	0.03
						21	≤ 0.01	≤ 0.01	≤ 0.02
						28	< 0.01	< 0.01	< 0.02
Tomatoes (Brillante), Albujon, Spain, 2000	2 (13)	73, 81	0.10, 0.10	0.61, 0.64	591, 618	0	0.38 (0.38, 0.37)	0.04 (0.04, 0.03)	0.41, (0.42, 0.40)
						8	0.02	0.04 (0.05, 0.04)	0.06 (0.07, 0.06)
						13	< 0.01	0.03	0.04
						20	≤ 0.01	0.01	0.02
						27	< 0.01	< 0.01	< 0.02
Tomatoes (Rufus), Fiorenzuola, Italy, 2001	2 (14)	72, 82	0.10, 0.10	0.63, 0.62	604, 601	0	0.30	< 0.01	0.31
						23	≤ 0.01	≤ 0.01	≤ 0.02
Tomatoes (Isola), Mulazzano, Italy, 2001	2 (14)	72, 73-74	0.10, 0.10	0.62, 0.62	597, 600	0	0.37	0.02	0.39
						21	≤ 0.01	≤ 0.01	≤ 0.02
Tomatoes	2	63, 72	0.10,	0.62, 0.64	600,	0	0.34	0.02	0.36

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
(Rollesta), El Pinoso, Spain, 2001	(14)		0.10		619				
						21	< 0.01	< 0.01	< 0.02
Tomatoes (Lustro), Mazarron, Spain, 2001	2 (14)	63, 72	0.10, 0.10	0.62, 0.65	597, 628	0	0.29	0.02	0.31
						21	< 0.01	< 0.01	< 0.02

No residues were detected in any of the untreated control samples.

Trials were conducted in field grown tomatoes in Australia (Lean – 2010). 3 × 0.03 kg ai/100 L dilute foliar applications of a 400 g/L EC formulation were made to the point of run-off at 7-day intervals. Fruit was collected at various intervals, mainly from 0 to 14 days after the last application. Analyses were completed within 6 months of collection.

Table 121 Residues of dimethoate and omethoate in tomatoes in Australia – 2008 trials (Lean – 2010)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Tomatoes (processing) – field grown, Echuca, VIC, Australia 2008	3 (6, 7)		0.03, 0.03, 0.03	0.31, 0.30, 0.28	1030, 1010, 920	0	1.9	0.62
						3	0.63	0.79
						7	0.33	0.27
						15	0.06	0.16
Tomatoes (Swanson) – field grown, Virginia, SA, Australia, 2008	3 (7, 6)		0.03, 0.03, 0.03	0.33, 0.32, 0.30	1100, 1055, 1007	0	1.5	0.30
						3	0.83	0.27
						7	0.49	0.34
						14	0.12	0.22
Tomatoes (Guardian) – field grown, Ayr, QLD, Australia, 2008	3 (7, 6)		0.03, 0.03, 0.03	0.19, 0.18, 0.18	623, 600, 605	7	0.52	0.40
Tomatoes (Roma) – field grown, Doonan, QLD, Australia,	3 (8, 7)		0.03, 0.03, 0.03	0.24, 0.23, 0.22	785, 780, 725	29	< 0.01	< 0.01

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Spray conc. (kg ai/100 L)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
2009								

No residues were found above the LOQ in untreated control samples.

Lettuce (leaf)

A series of trials was conducted in leafy lettuce varieties (Raufer – 2009l, Raufer – 2011c and Raufer – 2012b) at sites in northern Europe during the 2008, 2010 and 2011 growing seasons. 2 × 0.24 kg ai/ha foliar applications of a 400 g/L dimethoate EC formulation were made using a knapsack sprayer at a re-treatment interval of 7-10 days, with the final application timed for 2 weeks before harvest maturity, with whole plants or heads depending on maturity being collected at intervals from 0 up to 21 days after application. Samples were frozen within 4 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method involving extraction with acetonitrile followed by addition of magnesium sulfate, sodium chloride and sodium citrate to create a partition with an aliquot of the organic phase further cleaned up with PSA sorbent and magnesium sulfate, followed by analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes). Recoveries of dimethoate and omethoate from fortified control samples ranged from 70-112%. Samples were analysed within 10 months of collection.

Table 122 Residues of dimethoate and omethoate in leafy lettuce in northern Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009l, Raufer – 2011c and Raufer – 2012b)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	DM (mg/kg)	OM (mg/kg)	DM + OM (mg/kg)
Lettuce (Red Lollo 'Nation'), 71440, Savigny sur Seille, Bourgogne, France, 2008	2 (7)	15, 16	0.25, 0.24	395, 386	Whole plants w/o roots	0	18	0.13	18
					Whole plants w/o roots	3	2.7	0.38	3.1
					Whole plants w/o roots	7	0.48	0.16	0.65
					Whole plants w/o roots	10	0.10	0.02	0.12
					Heads	14	0.05	0.02	0.07
					Heads	21	< 0.01	< 0.01	< 0.02
Lettuce (Red Lollo 'Bastille'), WR8, Defford, Worcestershire, UK, 2008	2 (7)	39, 39	0.24, 0.24	403, 402	Whole plants w/o roots	0	8.9	0.06	9.0
					Whole plants w/o roots	4	1.1	0.14	1.2
					Whole plants w/o	7	0.29	0.07	0.37

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	DM (mg/kg)	OM (mg/kg)	DM + OM (mg/kg)
					roots				
					Whole plants w/o roots	10	0.15	0.05	0.20
					Heads	14	< 0.01	< 0.01	< 0.02
					Heads	21	< 0.01	< 0.01	< 0.02
Lettuce (White Lollo 'Locarno'), 64514, Pamiatkowo, Wielkopolska, Poland, 2008	2 (7)	42, 44	0.25, 0.25	413, 412	Whole plants w/o roots	0	5.3	0.06	5.4
					Whole plants w/o roots	3	1.6 c< 0.01	0.22	1.8
					Whole plants w/o roots	7	0.54	0.17	0.72
					Whole plants w/o roots	10	0.16	0.10	0.27
					Heads	14	0.03	0.03	0.06
					Heads	21	< 0.01	< 0.01	< 0.02
Lettuce (White Lollo 'Lugarno'), 5500, Middelfart, Fyn, Denmark, 2008	2 (7)	44, 47	0.24, 0.25	406, 417	Whole plants w/o roots	0	3.6	0.09	3.7
					Whole plants w/o roots	3	0.05	0.05	0.10
					Whole plants w/o roots	7	0.13, 0.13 c0.07	0.05, 0.04	0.18, 0.17
					Whole plants w/o roots	10	0.07	0.05	0.12
					Heads	14	0.02	0.03	0.05
					Heads	21	< 0.01	< 0.01	< 0.02)
Lettuce (Lollo 'Bastille'), PR46, Holmeswood, Lancashire, UK, 2010	2 (7)	12, 14	0.24, 0.24	403, 397	Whole plants w/o roots	0	19	0.11	19
					Heads	21	0.01	< 0.01	0.02
Lettuce (Lollo 'Carmesi'), 77930, Chailly-en-Biere, Seine et Marne, France, 2011	2 (7)	18-19, 33	0.24, 0.24	402, 408	Whole plants w/o roots	0	17	0.17	17
					Heads	21	0.09	0.04	0.12

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	DM (mg/kg)	OM (mg/kg)	DM + OM (mg/kg)
							(0.10, 0.08)	(0.04, 0.03)	(0.14, 0.11)
Lettuce (Batavia 'Marittima'), 64514, Pamiatkowo, Wielkopolska, Poland, 2011	2 (7)	19, 41	0.24, 0.24	403, 394	Whole plants w/o roots	0	13	0.15	13
					Heads	21	< 0.01	< 0.01	< 0.02
Lettuce (Lollo 'Anthony'), 64514, Pamiatkowo, Wielkopolska, Poland, 2011	2 (7)	18, 41	0.23, 0.23	387, 391	Whole plants w/o roots	0	9.2	0.12	9.3
					Heads	21	< 0.01	< 0.01	< 0.02

Except where otherwise noted, no residues were detected in any of the untreated control samples.

Legume vegetables

Peas (succulent with pods)

Four trials were conducted in peas in the USA during the 1993 growing season (Rice – 1994). A single application of a 400 g/L EC formulation was made close to harvest at a target rate of 0.20 kg ai/ha. Samples of peas with pods, vines and hay (sun-dried vines) were collected 0, 3 and 7 days after application. Samples were frozen within 6 hours of collection. Samples were homogenised and extracted with acetone or acetone/water for samples with water content <45%, and an aliquot was cleaned up by partitioning (addition of dichloromethane, further acetone and sodium chloride). The aqueous phase was partitioned twice more with dichloromethane, and the combined organic phases evaporated to dryness and reconstituted in hexane/acetone for further cleanup by solid phase extraction (Celite/charcoal column, elution with 1:1 hexane/acetone). Eluted samples were concentrated and made up to volume in acetone and analysed by GC-FPD in phosphorus mode (LOQ = 0.01 mg/kg for both analytes). Concurrent recoveries for peas were within an acceptable range (80-120% for both analytes), as were dimethoate recoveries for vines and hay (70-100%). Recoveries for omethoate from vines and hay were 50-120% and 60-140%, however only one vine sample and two hay samples were outside the range 70-120%, with all of these being at the 0.01 mg/kg fortification. Fortified recoveries at levels similar to the samples were within the acceptable range, therefore the results are considered valid. Samples were analysed within 3 months of collection.

Table 123 Residues of dimethoate and omethoate in pea seed (succulent with pods) after a single application of dimethoate – USA trials 1993 (Rice-1994)

Crop (variety), location, year	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Peas, (Knight), Hillsboro, OR, USA, 1993	0.19	161	0	0.44 (0.39, 0.50)	0.02 (0.02, 0.02)
			3	0.20 (0.22, 0.19)	0.06 (0.07, 0.06)
			7	0.08 (0.09, 0.08)	0.10 (0.10, 0.09)
Peas (FR652), Moses Lake, WA, USA, 1993	0.19	163	0	0.50 (0.52, 0.48)	< 0.01 (< 0.01, < 0.01)

Crop (variety), location, year	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
			3	0.26 (0.26, 0.25)	0.07 (0.07, 0.07)
			7	0.20 (0.19, 0.20)	0.07 (0.07, 0.04)
Peas (Sunburst), Lake Mills, WI, USA, 1993	0.19	161	0	0.36 (0.39, 0.34)	0.02 (0.02, 0.02)
			3	0.21 (0.22, 0.20)	0.08 (0.08, 0.08)
			7	0.10 (0.10, 0.09)	0.06 (0.06, 0.07)
Peas (77 EP/Nunhems), Verona, WI, USA, 1993	0.19	151	0	0.27 (0.29, 0.25)	0.02 (0.02, 0.02)
			3	0.15 (0.15, 0.15)	0.07 (0.07, 0.07)
			7	0.02 (0.02, 0.03)	0.04 (0.04, 0.04)

No residues were detected in any of the untreated control samples.

Two trials were conducted in peas with pods in California in the 1997 growing season (Samoil – 2000). At each site, two treated plots were set up, one receiving 4 × 0.20 kg ai/ha foliar applications of a 400 g/L EC formulation at approximately 14-day intervals, while the second received 2 × 0.20 kg ai/ha approximately 14 days apart., using a tractor-mounted boom sprayer or a backpack sprayer. Samples were frozen with 2 hours of collection.

Samples were extracted by homogenisation with 5:95 ethanol/ethyl acetate in the presence of sodium sulphate. The extracted was reduced in volume and cleaned up by solid phase extraction (Celite/activated charcoal column, elution with 1:1 acetone/hexane). The extracts were analysed by GC-FPD operating in the phosphorus mode. The method LOQ was 0.06 mg/kg for both dimethoate and omethoate. Concurrent method recoveries ranged from 75–122%, with the exception of the 0.02 mg/kg omethoate fortification (138–140%). Samples were analysed within 24 months of collection. Acceptable recoveries (mean 85% for dimethoate, omethoate was not tested) were achieved for concurrently stored fortified samples.

Table 124 Residues of dimethoate and omethoate in peas with pods (succulent) – USA trials 1997 (Samoil – 2000)

Crop (variety), location, year	No. (RTI, days)	Growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Peas (Oregon Sugar Pod II), Salinas, CA, USA, 1997	4 (14, 14, 14)	8-14 inch, 10% bloom, 100% bloom/50% pods, mature	0.19, 0.19, 0.19, 0.19	430, 421, 448, 448	2	0.43 (0.46, 0.40)	0.031 (0.031, 0.031)
	2 (14)	100% bloom/50% pods, mature	0.19, 0.19	448, 448	2	0.40 (0.39, 0.40)	0.030 (0.030, 0.029)
Peas (Oregon Sugar Pod II), Salinas, CA, USA, 1997	4 (14, 13, 14)	8-14 inch, 10-14 inch (50% bloom), 12-16 inch (50% bloom/50% pods), 13-16 inch (100% pods and flowers)	0.19, 0.19, 0.19, 0.19	448, 448, 440, 440	2	0.37 (0.36, 0.37)	0.025 (0.022, 0.028)
	2 (14)	12-16 inch	0.19, 0.19	440, 440	2	0.41 (0.42,	0.032 (0.031,

Crop (variety), location, year	No. (RTI, days)	Growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
		(50% bloom/50% pods), 13-16 inch (100% pods and flowers)				0.40)	0.033)

No residues were detected in untreated control samples. Trials were 1 month apart.

Peas (succulent, without pods)

Three trials were conducted in peas without pods in the USA during 2000 (Corley – 2004). At each site, two treated plots were set up, one receiving 4 × 0.20 kg ai/ha foliar applications of a 400 g/L EC formulation at approximately 14-day intervals, while the second received 2 × 0.39 kg ai/ha approximately 14 days apart., using a backpack hand boom sprayer. Samples were frozen within 3 hours of collection. Samples were extracted by homogenising three times with acetone. The filtered acetone extract was cleaned up by liquid/liquid partition (addition of water, further acetone, dichloromethane and sodium chloride). The aqueous phase was partitioned twice more with dichloromethane. The organic extracts were combined, evaporated to dryness and cleaned up by solid phase extraction (silica column). Cleaned sample extracts were analysed by GC-FPD operating in phosphorus mode. The validated method LOQ was 0.01 mg/kg for both dimethoate and omethoate. Concurrent method recoveries for dimethoate and omethoate ranged from 81–103%.

Table 125 Residues of dimethoate and omethoate in pea seed (succulent without pods) – USA trials 2000 (Corley – 2004)

Crop (variety), location, year	No. (RTI, days)	Growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Peas (Cascadia), Kimberley, ID, USA, 2000	4 (15, 13, 14)	4-5 inch, 6-8 inch (early bloom), 12 inch (flowering), mature	0.20, 0.19, 0.20, 0.19	287, 282, 292, 281	4	0.033 (0.032, 0.034)	< 0.01 (< 0.01, < 0.01)
					7	0.015 (0.02, 0.01)	< 0.01 (< 0.01, < 0.01)
					14	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
	2 (14)	12 inch (flowering), mature	0.40, 0.37	297, 275	14	0.020 (0.020, 0.019)	0.012 (0.013, 0.011)
Peas (Oregon Trial), Moxee, WA, USA, 2000	4 (13, 15, 13)	3-4 inch (seedling), 9 inch, 16 inch (fruiting), 16 inch (fruiting)	0.19, 0.19, 0.20, 0.19	381, 381, 389, 383	5	0.028 (0.025, 0.031)	< 0.01 (< 0.01, < 0.01)
					14	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

Crop (variety), location, year	No. (RTI, days)	Growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
	2 (14)	12 inch (budding), 16 inch (fruiting)	0.40, 0.39	391, 386	14	0.018 (0.016, 0.020)	< 0.01 (< 0.01, < 0.01)
Peas (Progress 9), Prosser, WA, 2000	4 (14, 14, 15)	2-5 inch, 4-8 inch, 5-10 inch, 7-14 inch (fruiting)	0.19, 0.19, 0.19, 0.19	356, 359, 374, 372	5	0.018 (0.019, 0.017)	< 0.01 (< 0.01, < 0.01)
					7	0.01 (0.011, < 0.01)	< 0.01 (< 0.01, < 0.01)
					13	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
	2 (14)	4-8 inch, 5-10 inch	0.37, 0.38	350, 369	15	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

Except where otherwise noted, no residues were detected in any of the untreated control samples.

Yard long bean

A series of residue trials for dimethoate in yard long bean was supplied by the government of Thailand.

Plots were treated with four applications of a 400 g/L EC formulation of dimethoate at a target rate of 0.6 kg ai/ha (0.08 kg ai/100 L). Samples of beans with pod were collected at intervals from 0 to 14 days after the last application and frozen.

Samples were analysed using a QuEChERS-based method. Homogenised samples were extract with 0.1% acetic acid in acetonitrile, then magnesium sulfate and sodium chloride were added to create a partition. After centrifuging, the acetonitrile layer was cleaned up by dispersive solid phase extraction (PSA sorbent and magnesium sulfate), then centrifuged and the supernatant transferred to a GC vial for analysis by GC-FPD. The validated method LOQ was 0.05 mg/kg for each analyte, while the detection limits were 0.02 mg/kg. Method linearity was good ($r^2 > 0.99$). Concurrent recoveries at fortification levels of 0.05, 0.1, and 0.2 mg/kg ranged from 75–117% for dimethoate and omethoate. Samples were analysed on the day of collection.

Table 126 Residues of dimethoate and omethoate in yard long bean for trials conducted in Thailand in 2010-12

Crop, variety, location, site no.	No. of applications, (interval, days)	Application rate (kg ai/ha)	Spray conc. (kg ai/100 L)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Yard long bean (Lum Num Shee), Nong-suea, Pathumthanee, Thailand, 2010	4 (7, 7, 8)	0.6, 0.6, 0.6, 0.6	0.08, 0.08, 0.08, 0.08	750, 750, 750, 750	0	3.9	0.12	4.0
					1	2.1	0.17	2.3
					3	0.17	0.10	0.28

Crop, variety, location, site no.	No. of applications, (interval, days)	Application rate (kg ai/ha)	Spray conc. (kg ai/100 L)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					5	< 0.05	< 0.05	< 0.10
					7	< 0.05	< 0.05	< 0.10
					10	< 0.05	< 0.05	< 0.10
					14	< 0.05	< 0.05	< 0.10
Yard long bean (Lum Num Shee), Dumnernsaduak, Rachaburee, Thailand, 2010	4 (7, 7, 7)	0.6, 0.6, 0.6, 0.6	0.08, 0.08, 0.08, 0.08	750, 750, 750, 750	0	4.8	0.16	5.0
					1	1.9	0.63	2.6
					3	0.47	0.46	0.96
					5	0.06	0.32	0.40
					7	< 0.05	< 0.05	< 0.10
					10	< 0.05	< 0.05	< 0.10
					14	< 0.05	< 0.05	< 0.10
Yard long bean (Lum Num Shee), Amphoe Mueang, Nakhon Pathom, Thailand, 2010	3 (7, 8)	0.6, 0.6, 0.6	0.08, 0.08, 0.08	750, 750, 750	0	2.2	0.24	2.5
					1	1.1	0.49	1.6
					3	0.21	0.41	0.62
					5	< 0.05	0.09	0.14
					7	< 0.05	< 0.05	< 0.10
					10	< 0.05	< 0.05	< 0.10
					14	< 0.05	< 0.05	< 0.10
Yard long bean (Lum Num Shee), Amphoe Mueang, Nakhon Pathom, Thailand, 2011	3 (7, 8)	0.6, 0.6, 0.6	0.08, 0.08, 0.08	750, 750, 750	0	2.6	0.45	3.0
					1	2.1	0.51	2.6
					3	0.45	0.31	0.76
					5	0.08	< 0.05	0.13
					7	0.05	< 0.05	< 0.10
					10	< 0.05	< 0.05	< 0.10
					14	< 0.05	< 0.05	< 0.10
Yard long bean (Saifa No. 5), Amphoe Sansai, Chiang Mai, Thailand, 2012	3 (7, 7)	0.6, 0.6, 0.6	0.08, 0.08, 0.08	750, 750, 750	0	3.5	0.42	3.9
					1	2.3	0.63	3.0

Crop, variety, location, site no.	No. of applications, (interval, days)	Application rate (kg ai/ha)	Spray conc. (kg ai/100 L)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					3	0.42	0.38	0.80
					5	0.06	0.19	0.25
					7	< 0.05	< 0.05	< 0.10
					10	< 0.05	< 0.05	< 0.10
					14	< 0.05	< 0.05	< 0.10
Yard long bean (Dok Klong 10), Amphoe Samgo, Suphanburi, Thailand, 2012	3 (7, 8)	0.6, 0.6, 0.6	0.08, 0.08, 0.08	750, 750, 750	0	2.5	0.31	2.8
					1	1.3	0.42	1.7
					3	0.21	0.26	0.47
					5	< 0.05	< 0.05	< 0.10
					7	< 0.05	< 0.05	< 0.10
					10	< 0.05	< 0.05	< 0.10
					14	< 0.05	< 0.05	< 0.10

No residues were detected in any of the untreated control samples.

Pulses

Peas (dry)

Five trials in peas (dry) were conducted in the USA during the 1997 growing season (Samoil – 2002). Plots were treated with 3 × 0.19 kg ai/ha foliar applications of a 400 g/L EC formulation of dimethoate at approximately 7 day intervals. Samples of dry peas were separated from plant material in the field and at two sites were dried in the field for 6 or 13 days after cutting before collection. Samples were frozen within 3 hours of collection. Dry pea sample were analysed for dimethoate alone by homogenisation with 5:95 ethanol/ethyl acetate in the presence of sodium sulphate. The extracted was reduced in volume and cleaned up by solid phase extraction (Celite/activated charcoal column, elution with 1:1 acetone/hexane). The extracts were analysed by GC-FPD operating in the phosphorus mode (LOQ = 0.02 mg/kg). Means concurrent recoveries of dimethoate ranged from 81–110%. Analyses were completed within 9 months of sample collection. Stability samples showed recoveries of 82–87% after 15 months storage, indicating that samples were unlikely to have been affected by degradation on storage.

Table 127 Residues of dimethoate and omethoate in peas (dry) – USA trials 1997 (Samoil – 2002)

Crop (variety), location, year	No. (RTI, days)	Growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)
Peas (Alaska), Kimberley, ID, USA, 1997	3 (7, 7)	Pod filling, pod filling, mature	0.18, 0.18, 0.19	187, 187, 196	8 + 6 days field drying	< 0.02
					8 + 13 days field drying	< 0.02
Peas (Alaska),	3 (7, 7)	Pod filling, pod	0.19, 0.19, 0.18	187, 196,	8 + 6 days	< 0.02

Crop (variety), location, year	No. (RTI, days)	Growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)
Kimberley, ID, USA, 1997		filling, mature		178	field drying	
					8 + 13 days field drying	< 0.02
Peas (B-160), Pullman, WA, USA, 1997	3 (7, 7)	Late bloom, post bloom, commencing drying	0.19, 0.18, 0.19	168, 121, 131	14	< 0.02
					21	< 0.02
Peas (B-160) Pullman, WA, USA, 1997	3 (7, 7)	Late bloom, post bloom, commencing drying	0.19, 0.19, 0.19	159, 131, 131	14	< 0.02
					21	< 0.02
Peas (B-160), Pullman, WA, USA, 1997	3 (7, 7)	Late bloom, post bloom, commencing drying	0.19, 0.19, 0.20	168, 121, 131	14	< 0.02
					21	< 0.02

No residues were detected in untreated control samples.

Beans (dry)

Three residue trials each in mung beans, navy beans and soya beans were conducted in Australia during the 2012 growing season (Litzow – 2013). Three applications of a 400 g/L EC formulation were made at 0.32 kg ai/ha at approximately 14 day intervals. Three of the trials also included applications at 0.64 kg ai/ha. Treatment were applied as a low volume broadcast spray in sufficient water (82–110 L/ha) to ensure even and thorough coverage of the crop. Representative forage samples were taken at 0 and 6–7 days after the last application. Seed and harvest trash samples were taken at 13–14 days after the last application. The moisture content of forage and trash was determined. Samples were stored frozen until analysis which was completed within 6 months of sample collection.

Dimethoate and omethoate residues were extracted from blended and homogenised sample with acetonitrile/water (9:1). An aliquot of the extract was diluted with water, filtered and analysed for dimethoate and omethoate by LC-MS/MS. Quantitation of dimethoate and omethoate was achieved by comparison with mixed external standards. The LOQ was 0.05 mg/kg, the LOD was 0.02 mg/kg. Recoveries from fortified control samples of mung bean, navy bean and soya bean forage, seed and trash were within acceptable limits (97–114%).

Table 128 Residues of dimethoate and omethoate in mung beans (Litzow – 2013)

Crop, variety, location, site no.	Application rate (kg ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (kg)
Mung beans, Crystal, Narrabri, NSW, 120277	0.32	3 (14)	91, 94, 92	13	0.066	< 0.05	0.17
	0.64	3 (14)	91, 93, 93	13	0.33	0.069	0.40

Crop, variety, location, site no.	Application rate (kg ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (kg)
Mung beans, Crystal, Edgeroi, NSW, 120278	0.32	3 (14)	91, 94, 92	13	0.40	0.064	0.46
Mung beans, Crystal, Wellcamp, Qld, 120279	0.32	3 (14)	96, 107, 104	14	< 0.05	< 0.05	< 0.10

No residues were found above the LOQ in any of the untreated control samples.

Table 129 Residues of dimethoate and omethoate in navy beans (Litzow – 2013)

Crop, variety, location, site no.	Application rate (kg ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (kg)
Navybeans, Arwon, Kumbia, Qld, 120280	0.32	3 (14)	99, 110, 108	14	< 0.05	< 0.05	< 0.10
	0.64	3 (14)	99, 110, 108	14	< 0.05	< 0.05	< 0.10
Navybeans, Arwon, Kumbia, Qld, 120281	0.32	3 (14)	100, 110, 107	14	< 0.05	< 0.05	< 0.10
Navybeans, Spearfelt, Euberta, NSW, 120282	0.32	3 (13, 15)	127, 133, 108	14	< 0.05	< 0.05	< 0.10

No residues were found above the LOQ in any of the untreated control samples.

Table 130 Residues of dimethoate and omethoate in soya beans (Litzow – 2013)

Crop, variety, location, site no.	Application rate (kg ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)
Soya beans, Rose, Collegeview, Qld, 120283	0.32	3 (13, 15)	83, 95, 82	14	< 0.05	< 0.05
	0.64	3 (13, 15)	82, 95, 82	14	< 0.05	< 0.05
Soya beans, Ascott, Condamine Plains, Qld, 120284	0.32	3 (14)	83, 102, 82	14	< 0.05	< 0.05
Soya beans, Bunya, Edgeroi, NSW, 120285	0.32	3 (14)	92	14	< 0.05	< 0.05

No residues were found above the LOQ in the untreated control samples.

Root and tuber vegetables

Carrots

A series of field decline trials was conducted in carrots in northern Europe in the 2004, 2005 and 2007 growing seasons (Jones – 2005, Jones – 2006, and Jones-2008). 4 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation were made at intervals of 7 days. Samples were collected at intervals from immediately before the final application up to 42 days after, and frozen within 8 hours of collection. Samples were analysed for dimethoate and omethoate by homogenisation with acetonitrile and water, followed by addition of sodium chloride to create a partition. An aliquot of the acetonitrile phase was dried with sodium sulfate, evaporated to dryness and reconstituted in 3:1 acetonitrile/toluene before solid phase extraction cleanup (EnviCarb and primary amine columns, with 3:1 acetonitrile/toluene as elution solvent. Eluted samples were evaporated to dryness and reconstituted in 1:1 methanol/water before analysis by LC-MS/MS. The method LOQ was 0.001 mg/kg for both analytes. Concurrent recoveries ranged from 76-106%. Analyses were completed within 6 months of sample collection.

Table 131 Residues of dimethoate and omethoate in carrots in northern Europe after 4 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Jones – 2005, Jones – 2006 and Jones-2008)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (kg)
Carrots (Trevour), Wholsea, East Yorkshire, UK, 2004	4 (7, 7, 7)	45, 45, 45-47, 45-47	0.25, 0.25, 0.24, 0.24	415, 413, 401, 405	0-	0.009	0.009	0.018
					0+	0.030	0.009	0.039
					7	0.012	0.007	0.019
					10	0.010	0.009	0.019
					14	0.004	0.009	0.013
					21	0.002	0.005	0.007
					28	0.001	0.005	0.006
Carrots (Nairobi), Ravenshead, Nottinghamshire, UK, 2004	4 (7, 7, 7)	45-47, 45-47, 45-47, 47	0.25, 0.25, 0.24, 0.24	421, 410, 392, 401	0-	0.036 (0.034, 0.038)	0.007	0.043 (0.041, 0.045)
					0+	0.039 (0.037, 0.041)	0.008 (0.008, 0.007)	0.047 (0.045, 0.048)
					7	0.046 (0.043, 0.048)	0.008	0.054 (0.051, 0.056)
					10	0.051 (0.050, 0.052)	0.010 (0.011, 0.009)	0.061 (0.06, 0.061)
					14	0.033	0.011	0.044
					21	0.018	0.009	0.027
					28	0.008	0.008	0.016
Carrots (Chambord), Lacrost, 71290, Saone-et-Loire, France, 2004	4 (7, 7, 7)	44, 47, 48, 48	0.25, 0.24, 0.25, 0.25	412, 402, 416, 411	0-	0.007	0.004	0.011

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (kg)
					0+	0.060	0.005	0.065
					7	0.021	0.005	0.026
					10	0.016	0.007	0.023
					14	0.005	0.003	0.008
					21	0.001	0.002	0.003
					28	<u>< 0.001</u>	<u>0.001</u>	<u>< 0.002</u>
Carrots (Chambord), St Lambert du Leeves, 49400, Maine-et-Loire, France, 2004	4 (7, 8, 6)	42, 45, 47, 48	0.24, 0.24, 0.24, 0.24	395, 394, 401, 392	0-	0.062	0.011	0.073
					0+	0.085	0.009	0.094
					7	0.053	0.015	0.068
					10	0.029	0.009	0.038
					14	0.020	0.009	0.029
					21	0.009	0.007	0.016
					28	<u>0.006</u>	<u>0.006</u>	<u>0.012</u>
Carrots (Narobi), Market Weighton, Yorkshire, UK, 2005	4 (7, 7, 7)	45, 45-47, 47, 48	0.23, 0.24, 0.24, 0.24	388, 406, 402, 402	0-	0.009	0.006	0.015
					0+	0.005	0.008	0.013
					21	0.002	0.014	0.016
					28	< 0.001	<u>0.013</u>	<u>0.014</u>
					35	<u>0.001</u>	0.006	0.007
					42	< 0.001	0.007	0.008
Carrots (Nairobi), Hilton, Staffordshire, UK, 2005	4 (7, 7, 7)	45, 46, 46-48, 46-48	0.24, 0.24, 0.24, 0.24	402, 400, 395, 401	0-	0.012	0.004	0.016
					0+	0.036	0.004	0.040
					21	0.004	0.002	0.006
					28	<u>0.004</u>	<u>0.003</u>	<u>0.007</u>
					35	0.002	0.002	0.004
					42	0.001	0.002	0.003
Carrots (Bolero), St Lambert des Levees, 49400, Maine-et-Loire, France, 2005	4 (7, 6, 7)	42, 43, 47, 48	0.25, 0.25, 0.25, 0.25	411, 413, 412, 418	0-	0.015	0.005	0.020
					0+	0.027	0.005	0.032
					21	0.005	0.005	0.010
					28	<u>0.002</u>	<u>0.004</u>	<u>0.006</u>
					35	< 0.001	0.003	0.004

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (kg)
					42	< 0.001	0.003	0.004
Carrots (Napoli), Brain sur Allonnes, 49650, Maine-et-Loire, France, 2005	4 (7, 7, 7)	13, 15-16, 41, 42	0.24, 0.25, 0.24, 0.24	405, 414, 398, 401	0-	0.027	0.012	0.039
					0+	0.088	0.011	0.099
					21	< 0.001	0.003	0.004
					28	< 0.001	0.002	0.003
					35	< 0.001	0.001	0.002
					42	< 0.001	< 0.001	< 0.002
Carrots (Nairobi), Bothamstall, Nottinghamshire, UK, 2007	4 (7, 8, 6)	14, 15, 41-43, 43	0.24, 0.24, 0.24, 0.24	399, 395, 405, 404	28	< 0.001	< 0.001	< 0.002
					35	< 0.001	< 0.001	< 0.002
					42	< 0.001	< 0.001	< 0.002
Carrots (Nepal), Isleham, Cambridgeshire, UK, 2007	4 (6, 8, 7)	11-12, 12, 13, 15-16	0.24, 0.24, 0.24, 0.24	406, 407, 394, 404	28	< 0.001	< 0.001	< 0.002
					35	< 0.001	< 0.001	< 0.002
					42	< 0.001	< 0.001	< 0.002
Carrots (Montdibel), Longue, Maine-et-Loire, 49160, France, 2007	4 (7, 7, 7)	19, 41, 41-42, 43	0.25, 0.25, 0.25, 0.24	410, 408, 419, 398	28	< 0.001	0.0013	0.002
					35	< 0.001	< 0.001	< 0.002
					42	< 0.001	< 0.001	< 0.002
Carrots (Nerac), Logumkloster, 6240, Denmark, 2007	4 (7, 7, 7)	44, 45, 46, 47	0.25, 0.25, 0.25, 0.25	420, 424, 424, 411	27	< 0.001	0.0013	0.002
					35	< 0.001	0.001	0.002
					42	< 0.001	< 0.001	< 0.002

No residues were detected in any of the untreated control samples.

A further series of trials was conducted in carrots (Raufer – 2009o) at sites in southern Europe during the 2008 growing seasons. 3 × 0.24 kg ai/ha foliar applications of a 400 g/L dimethoate EC formulation were made at a re-treatment interval of 7 days, with the final application timed for 4 weeks before harvest maturity, with whole plants or roots and tops, depending on the size and growth stage of the crop being collected at intervals from 0 to 35 days after the last application. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method with analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes in whole plants and leaves, and 0.001 mg/kg in roots). Concurrent recoveries of dimethoate and omethoate from fortified control samples ranged from 70–112%. Samples were analysed within 7 months of collection.

Table 132 Residues of dimethoate and omethoate in carrots in southern Europe after 3 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009o)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (kg)
Carrots (Chambord), 66700, Argeles sur Mer, Languedoc-Roussillon, France, 2008	3 (7, 7)	43, 44, 45	0.26, 0.26, 0.25	317, 310, 303	Whole plants	0	1.9	0.25	2.17
					Whole plants	7	0.15	0.23	0.40
					Whole plants	14	0.01	0.10	0.12
					Roots	21	< 0.001	< 0.001	< 0.002
					Tops	21	< 0.01	0.18	0.20
					Roots	28	< 0.001	< 0.001	< 0.002
					Roots	35	< 0.001	< 0.001	< 0.002
Carrots (Navis F1), 11140, Conil de la Frontera, Andalusia, Spain, 2008	3 (7, 7)	41, 41, 43	0.24, 0.24, 0.24	300, 301, 299	Whole plants	0	2.8	0.23	3.05
					Whole plants	7	< 0.01	< 0.01	< 0.02
					Whole plants	14	< 0.01	0.02	0.03
					Roots	21	< 0.001	< 0.001	< 0.002
					Tops	21	< 0.01	0.03	0.04
					Roots	28	< 0.001	< 0.001	< 0.002
					Roots	35	< 0.001	< 0.001	< 0.002
Carrots (Bolero), 57008, Nea-Magnisia, Thessaloniki, Greece, 2008	3 (7, 7)	43, 44, 45	0.23, 0.24, 0.23	293, 302, 291	Whole plants	0	1.8	0.27	2.09
					Whole plants	7	0.42	0.40	0.85
					Whole plants	14	0.05	0.16	0.22
					Roots	21	0.01	0.004	0.01
					Tops	21	0.02	0.39	0.44
					Roots	28	0.002	0.003	0.005
					Roots	35	< 0.001	0.001	< 0.002
Carrots (Dondonia), 45020, Lusia,	3 (7, 7)	42, 43, 44	0.24, 0.24, 0.24	297, 303,	Whole plants	0	8.6	0.12	8.73

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (kg)
Veneto, Italy, 2008				297					
					Whole plants	7	0.06	0.05	0.11
					Whole plants	14	0.01	0.03	0.04
					Roots	21	0.003	0.003	0.01
					Tops	21	< 0.01	0.02	0.03
					Roots	28	0.001	0.003	0.004
					Roots	35	< 0.001	< 0.001	< 0.002

Sugar beet

A series of trials was conducted in sugar beet (Raufer – 2009m, Raufer – 2009n, Raufer – 2010i, Raufer – 2010j, and Raufer – 2010k) at sites in northern and southern Europe during the 2008, 2009 and 2010 growing seasons. 2 × 0.24 kg ai/ha foliar applications of a 400 g/L dimethoate EC formulation were made using a knapsack sprayer at a re-treatment interval of 21 days, with the final application timed for 4 weeks before harvest maturity, with whole plants being sampled at on the day of the last application, and mature harvest samples (separated into leaves and roots) being collected 28 days after the last application. At decline trial sites, additional samples were collected at 7, 14, and 21 days. Where reduction in sample size was required, mature roots were quartered and opposite quarters retained. Samples were frozen within 7 hours of collection and analysed for dimethoate and omethoate using a QuEChERS with analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes in whole plants and leaves, and 0.001 mg/kg in roots). Concurrent recoveries of dimethoate and omethoate from fortified control samples ranged from 70–119%. Samples were analysed within 9 months of collection.

Table 133 Residues of dimethoate and omethoate in sugar beet in Europe after 2 × 0.24 kg ai/ha foliar applications of a 400 g/L EC formulation (Raufer – 2009m, Raufer – 2009n, Raufer – 2010i, Raufer – 2010j, and Raufer – 2010k)

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Sugar beet (Pondus), LN3, Bardney, Lincolnshire, UK, 2008	2 (21)	39, 43-45	0.24, 0.25	200, 208	Whole plant	0	0.53	0.03	0.56
					Leaves	28	≤ 0.01	≤ 0.01	≤ 0.02
					Roots	28	≤ 0.001	≤ 0.001	≤ 0.002
Sugar beet (Jambus), 64560, Szczepankowo, Wielkopolska, Poland, 2008	2 (14)	39, 39	0.24, 0.24	197, 198	Whole plant	0	2.2	0.09	2.30
					Leaves	21	< 0.01	0.09	0.10
					Roots	21	< 0.001	< 0.001	< 0.002
Sugar beet (Lucata), 86947, Weil, Bayern,	2 (21)	42, 44-45	0.25, 0.23	208, 192	Whole plant	0	0.31	0.04	0.35

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Germany, 2008									
					Leaves	28	< 0.01	0.05	0.06
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Stine), 5500, Middelfart, Fyn, Denmark, 2008	2 (21)	39, 39	0.24, 0.24	198, 200	Whole plant	0	1.24 (0.94, 1.55)	0.03 (0.03, 0.03)	1.27 (0.97, 1.58)
					Leaves	28	< 0.01	0.03 (0.03, 0.03)	0.04 (0.04, 0.04)
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Gazelle), 11500, El Puerto de Santa Maria, Andalusia, Spain, 2008	2 (21)	38, 39	0.24, 0.24	203, 201	Whole plant	0	1.3	0.05	1.35
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Panther), 11406, Jerez de la Frontera, Andalusia, Spain, 2008	2 (21)	37, 37-38	0.24, 0.24	199, 200	Whole plant	0	1.1	0.03	1.13
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Austin), 58100, Giannitsa, Thessaloniki, Greece, 2008	2 (21)	39, 39	0.25, 0.23	208, 194	Whole plant	0	0.66	0.05	0.71
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Genio), 40054, Budrio, Emilia Romagna, Italy, 2008	2 (21)	39, 45-46	0.24, 0.24	202, 202	Whole plant	0	0.80	0.34	1.17
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Artur), 64606, Popowko, Wielkopolska, Poland, 2009	2 (20)	39, 39	0.24, 0.23	208, 191	Whole plant	0	1.3	0.03	1.33
					Whole plant	7	< 0.01	0.03	0.04
					Whole plant	14	< 0.01	0.03	0.04
					Whole	21	< 0.01	0.01	0.02

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					plant				
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Bobcat), YO86, North Duffield, North Yorkshire, UK, 2009	2 (28)	45, 47	0.25, 0.25	210, 207	Whole plant	0	0.95	0.04	0.99
					Whole plant	7	0.02	0.02	0.04
					Whole plant	14	< 0.01	< 0.01	< 0.02
					Whole plant	21	< 0.01	0.01	0.02
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Hugo), 15345, Altlandsberg, Brandenburg, Germany, 2009	2 (21)	45, 49	0.27, 0.27	223, 222	Whole plant	0	1.6	0.03	1.63
					Whole plant	7	0.09	< 0.01	0.1
					Whole plant	14	< 0.01	< 0.01	< 0.02
					Whole plant	21	< 0.01	< 0.01	< 0.02
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Topper), 5464, Brenderup, Fyn, Denmark, 2009	2 (21)	39, 39	0.24, 0.25	198, 203	Whole plant	0	1.6	0.07	1.67
					Whole plant	7	0.10	0.10	0.21
					Whole plant	14	< 0.01	< 0.01	< 0.02
					Whole plant	21	< 0.01	0.04	0.05
					Leaves	28	< 0.01	0.03	0.04
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Henrike), 64560, Karolin, Wielkopolksa, Poland, 2009	2 (20)	39, 39	0.25, 0.23	208, 191	Whole plant	0	1.5	0.02	1.52
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet	2	39, 43	0.24, 0.26	200,	Whole	0	2.5	0.10	2.61

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
(Mandela), NG22, Kirklington, Nottinghamshire, UK, 2010	(21)			217	plant				
					Whole plant	7	0.05	0.09	0.15
					Whole plant	14	< 0.01	0.06	0.07
					Whole plant	21	< 0.01	0.03	0.04
					Leaves	28	< 0.01	0.02	0.03
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Noelia), 42212, Covarrubius, Soria, Spain, 2010	2 (21)	40-42, 47-49	0.24, 0.25	197, 207	Whole plant	0	1.1	0.07	1.18
					Whole plant	7	0.10	0.08	0.19
					Whole plant	14	< 0.01	0.03	0.04
					Whole plant	21	< 0.01	0.02	0.03
					Leaves	28	< 0.01	0.05	0.06
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Rizor), 40054, Budrio, Emilia Romagna, Italy, 2010	2 (21)	39, 39	0.25, 0.27	209, 229	Whole plant	0	1.8	0.07	1.88
					Whole plant	7	< 0.01	0.05	0.06
					Whole plant	14	< 0.01	< 0.01	< 0.02
					Whole plant	21	< 0.01	< 0.01	< 0.02
					Leaves	28	< 0.01	< 0.01	< 0.02
					Roots	28	< 0.001	< 0.001	< 0.002
Sugar beet (Corsica), 58100, Giannitsa Valtos, Pella, Greece, 2010	2 (21)	38-39, 39-43	0.24, 0.25	203, 027	Whole plant	0	0.80	0.03	0.83
					Whole plant	7	0.01	0.04	0.05
					Whole plant	14	< 0.01	< 0.01	< 0.02
					Whole plant	21	< 0.01	< 0.01	< 0.02

Crop (variety), location, year	No. (RTI, days)	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
					Leaves	28	≤ 0.01	≤ 0.01	≤ 0.02
					Roots	28	≤ 0.001	≤ 0.001	≤ 0.002
Sugar beet (Austine), 58500, Dafni, Pella, Greece, 2010	2 (21)	37-38, 44	0.25, 0.25	207, 207	Whole plant	0	1.4	0.12	1.53
					Whole plant	7	< 0.01	0.03	0.03
					Whole plant	14	< 0.01	< 0.01	< 0.02
					Whole plant	21	< 0.01	< 0.01	< 0.02
					Leaves	28	≤ 0.01	≤ 0.01	≤ 0.02
					Roots	28	≤ 0.001	≤ 0.001	≤ 0.002

No residues were detected in any of the untreated control samples.

A series of trials for sugar beet grown in Europe during the 2014 growing season included analysis for six additional dimethoate metabolites (White – 2015a and White – 2015b). A single 0.2 kg ai/ foliar application of a 400 g/L dimethoate EC formulation was made, with treated and control leaf and roots samples being collected at 28 day (harvest trials) or at intervals from 0 to 28 days (decline trials) after the last application. Samples were frozen within 8 hours of collection and analysed for dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate.

Dimethoate, omethoate, and dimethoate carboxylic acid were analysed using a QuEChERS-based method with analysis by LC-MS/MS. For determination of O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, O-desmethyl omethoate, desmethyl dimethoate and O-desmethyl-N-desmethyl omethoate, samples were extracted with methanol/water (1:1 v/v), purified by solid phase extraction (Strata AX-W column, elution with 99:1 v/v methanol/35% ammonia), evaporated to dryness and reconstituted in dilute formic acid prior to LC-MS/MS analysis. The LOQs were 0.01 mg/kg for each analyte. Concurrent method recoveries were within the ranges 76–117% (dimethoate), 66–116% (omethoate), 66–108% (dimethoate carboxylic acid), 67–102% (O-desmethyl omethoate carboxylic acid), 71–113% (O-desmethyl isodimethoate), 69–101% (desmethyl dimethoate), 66–107% (O-desmethyl omethoate), and 72–98% (O-desmethyl N-desmethyl omethoate), respectively. Extractions and analysed were completed within 9 months of sample collection.

Table 134 Residues of dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in sugar beet in Europe during the 2014 growing season after a single 0.2 kg ai/ha foliar application of a 400 g/L EC formulation (White – 2015a and White – 2015b)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)				
						DM	OM	DCA	ODI	OCA
Sugar beet (Barents), 45300, Sermaises, Loiret, France,	35	0.21	210	Tops	0	1.80	0.07	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)				
						DM	OM	DCA	ODI	OCA
2014										
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.04	0.06	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	< 0.01	0.05	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	< 0.01 (ND)	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Copernicus), 45300, Yevre, la Ville, Loiret, France, 2014	49	0.20	195	Tops	0	3.58	0.14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.06	0.13	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	0.02	0.04	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	< 0.01 (ND)	0.04	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Artus), 71665, Vauhingen and der Enz, Pulverdingen, Baden-Württemberg, Germany, 2014	48	0.21	205	Tops	0	1.56	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)				
						DM	OM	DCA	ODI	OCA
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	8	0.19	0.23	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	8	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	13	0.05	0.08	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	13	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	< 0.01	0.07	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Annika), 21739, Dollern, Niedersachsen, Germany, 2014	45	0.20	203	Tops	0	1.58	0.04	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	6	0.16	0.08	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	6	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	13	0.08	0.17	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	13	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	20	< 0.01 (ND)	0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	20	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Springbok), DN22 0PT, Retford, Nottinghamshire, UK, 2014	39	0.19	193	Tops	28	< 0.01 (ND)	< 0.01	0.01	< 0.01 (ND)	< 0.01
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)				
						DM	OM	DCA	ODI	OCA
Sugar beet (Cayman), PE12 6LW, Fosdyke, Lincolnshire, UK, 2014	39	0.24	237	Tops	28	< 0.01 (ND)	0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Okapi), 67600, Ebersheim, Alsace, France, 2014	49	0.21	212	Tops	28	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Julius), 15345, Altlandsberg, Brandenburg, Germany, 2014	49	0.21	210	Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Charlie), 41740, Lebrija, Andalusia, Spain, 2014	37	0.21	213	Tops	0	0.14	0.14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	< 0.01 (ND)	0.09	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	< 0.01 (ND)	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	20	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	20	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	27	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	27	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Columbus), 41720, Los Palacios y Villafranca, Andalusia, Spain, 2014	36	0.21	208	Tops	0	0.16	0.08	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)				
						DM	OM	DCA	ODI	OCA
				Roots	0	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.03	0.13	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	< 0.01	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	20	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	20	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	27	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	27	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Marinella), 40024, Catel San Pietro Terme, Emilia Romagna, Italy, 2014	39/49	0.22	216	Tops	0	1.19	0.18	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	8	0.01	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	8	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	15	< 0.01	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	15	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Omella), 40054, Budrio, Emilia Romagna, Italy 2014	39/49	0.20	202	Tops	0	0.81	0.33	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)				
						DM	OM	DCA	ODI	OCA
				Tops	8	0.01	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	8	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	15	< 0.01 (ND)	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Roots	15	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Roots	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Cillon), 41740, Lebrisa, Andalusia, Spain, 2014	37	0.21	212	Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Portal), 11540, Sanlucar de Barrameda, Andalusia, Spain, 2014	36	0.22	217	Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Chipiona), 11520, Rota, Andalusia, Spain, 2014	37	0.21	213	Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Chipiona), 11406, Serez, Andalusia, Spain, 2014	37	0.21	213	Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the untreated control samples.

DM = dimethoate; OM = omethoate; DCA = dimethoate carboxylic acid; ODI = O-desmethyl isodimethoate; OCA = O-desmethyl omethoate carboxylic acid.

Table 135 Residues of desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in sugar beet in Europe during the 2014 growing season after a single 0.2 kg ai/ha foliar application of a 400 g/L EC formulation (White – 2015a and White – 2015b)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
Sugar beet (Barents), 45300, Sermaises, Loiret, France, 2014	35	0.21	210	Tops	0	0.03	0.01	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.10	0.02	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	0.15	0.04	< 0.01 (ND)
				Roots	14	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	0.60	0.18	< 0.01 (ND)
				Roots	21	0.02	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	0.26	0.02	< 0.01 (ND)
				Roots	28	0.02	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Copernicus), 45300, Yevre, la Ville, Loiret, France, 2014	49	0.20	195	Tops	0	0.09	0.02	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.18	0.02	< 0.01 (ND)
				Roots	7	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	0.10	0.02	< 0.01 (ND)
				Roots	14	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	0.26	0.11	< 0.01 (ND)
				Roots	21	0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	0.21	0.04	< 0.01 (ND)
				Roots	28	0.03	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Artus), 71665, Vauhingen and der Enz, Pulverdingen,	48	0.21	205	Tops	0	0.01	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
Baden-Württemberg, Germany, 2014								
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	8	0.27	0.17	< 0.01 (ND)
				Roots	8	0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	13	0.13	0.04	< 0.01 (ND)
				Roots	13	0.02	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	0.14	0.01	< 0.01 (ND)
				Roots	21	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	0.12	< 0.01	< 0.01 (ND)
				Roots	28	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Annika), 21739, Dollern, Niedersachsen, Germany, 2014	45	0.20	203	Tops	0	0.03	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	6	0.17	0.01	< 0.01 (ND)
				Roots	6	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	13	0.34	0.03	< 0.01 (ND)
				Roots	13	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	20	0.14	< 0.01	< 0.01 (ND)
				Roots	20	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	0.10	< 0.01	< 0.01 (ND)
				Roots	28	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Springbok), DN22 0PT, Retford, Nottinghamshire, UK, 2014	39	0.19	193	Tops	28	0.16	0.03	< 0.01 (ND)
				Roots	28	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Sugar beet	39	0.24	237	Tops	28	0.05	0.02	< 0.01

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
(Cayman), PE12 6LW, Fosdyke, Lincolnshire, UK, 2014								(ND)
				Roots	28	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Okapi), 67600, Ebersheim, Alsace, France, 2014	49	0.21	212	Tops	28	0.03	< 0.01	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Julius), 15345, Altlandsberg, Brandenburg, Germany, 2014	49	0.21	210	Tops	28	0.16	0.02	< 0.01 (ND)
				Roots	28	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Charlie), 41740, Lebrija, Andalusia, Spain, 2014	37	0.21	213	Tops	0	0.14	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.06	< 0.01 (ND)	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	14	0.02	< 0.01 (ND)	< 0.01 (ND)
				Roots	14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	20	0.05	< 0.01 (ND)	< 0.01 (ND)
				Roots	20	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	27	0.02	< 0.01 (ND)	< 0.01 (ND)
				Roots	27	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Columbus), 41720, Los Palacios y Villafranca, Andalusia, Spain, 2014	36	0.21	208	Tops	0	0.07	< 0.01 (ND)	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	7	0.08	< 0.01	< 0.01 (ND)
				Roots	7	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
				Tops	14	0.10	< 0.01 (ND)	< 0.01 (ND)
				Roots	14	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	20	0.04	< 0.01 (ND)	< 0.01 (ND)
				Roots	20	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	27	0.03	< 0.01 (ND)	< 0.01 (ND)
				Roots	27	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Marinella), 40024, Catel San Pietro Terme, Emilia Romagna, Italy, 2014	39/49	0.22	216	Tops	0	0.17	< 0.01	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	8	0.23	< 0.01	< 0.01 (ND)
				Roots	8	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	15	0.55	0.02	< 0.01 (ND)
				Roots	15	0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	0.19	< 0.01	< 0.01 (ND)
				Roots	21	0.01	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	0.14 c0.03, 0.02	< 0.01	< 0.01 (ND)
				Roots	28	< 0.01 c0.01, 0.02	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Omella), 40054, Budrio, Emilia Romagna, Italy 2014	39/49	0.20	202	Tops	0	0.13 c0.02	< 0.01	< 0.01 (ND)
				Roots	0	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	8	0.07	< 0.01	< 0.01 (ND)
				Roots	8	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	15	< 0.01	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
				Roots	15	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	21	0.02	< 0.01 (ND)	< 0.01 (ND)
				Roots	21	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Tops	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Cillon), 41740, Lebrisa, Andalusia, Spain, 2014	37	0.21	212	Tops	28	0.02	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Portal), 11540, Sanlucar de Barrameda, Andalusia, Spain, 2014	36	0.22	217	Tops	28	0.04	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Chipiona), 11520, Rota, Andalusia, Spain, 2014	37	0.21	213	Tops	28	0.02	< 0.01 (ND)	< 0.01 (ND)
				Roots	28	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Sugar beet (Chipiona), 11406, Serez, Andalusia, Spain, 2014	37	0.21	213	Tops	28	0.07	< 0.01	< 0.01 (ND)
				Roots	28	< 0.01	< 0.01 (ND)	< 0.01 (ND)

Except where otherwise noted, no residues were detected in any of the untreated control samples.

DMD = desmethyl dimethoate, ODO = O-desmethyl omethoate, ONO = O-desmethyl N-desmethyl omethoate.

Turnip

A series of 7 single point residue trials was conducted in turnips in the 1994 growing season (Samoil-1998). 3 × 0.28 kg ai/ha foliar applications of a 480 g/L EC formulation were made and samples of leaves and roots were collected at a target interval of 14 day after the last application and frozen within 3 hours of collection. Samples were extracted with acetone/water and partitioned into dichloromethane via addition of water and sodium chloride, an aliquot of the dichloromethane was evaporated to dryness and reconstituted in 1:1 hexane/acetone then cleaned up by solid phase extraction (Celite/charcoal column, elution with 1:1 hexane/acetone). Cleaned extracts were analysed by GC-FPD (phosphorus mode). Concurrent recoveries ranged from 69–124% (dimethoate, tops), 78–123% (omethoate, tops), 79–106% (dimethoate, roots), and 75–108% (omethoate, roots). Samples were analysed within 19 months of collection.

Table 136 Residues of dimethoate and omethoate in turnip tops in the USA during the 1994 growing season after 3 × 0.48 kg ai/ha foliar applications of a 400 g/L EC formulation (Samoil-1998)

Crop (variety), location, year	No. (RTI, days)	Rate (kg ai/ha)	DALA	Residues (mg/kg)	
				Dimethoate	Omethoate
Turnips (Purple Top), Fayetteville, AR, USA, 1994	3 (7, 7)	0.28, 0.28, 0.28	14	< 0.1	< 0.1
Turnips (Purple Top), Salinas, CA, USA, 1994	3 (7, 7)	0.28, 0.28, 0.28	15	0.15 (0.18, 0.13)	0.31 (0.34, 0.28)
Turnips (All Top), Gainesville, FL, USA, 1994	3 (8, 7)	0.28, 0.28, 0.28	13	0.55 (0.53, 0.56)	0.21 (0.21, 0.21)
Turnips (Purple Top), Tifton, GA, USA, 1994	3 (6, 7)	0.28, 0.28, 0.28	14	0.15 (0.11, 0.18)	< 0.1
Turnips (Purple Top), Willard, OH, USA, 1994	3 (8, 10)	0.28, 0.28, 0.28	14	< 0.1	< 0.1
Turnips (Purple Top), Willard, OH, USA, 1994	3 (7, 7)	0.28, 0.28, 0.28	14	< 0.1	< 0.1
Turnips (Purple Top), Weslaco, TX, USA, 1994	3 (8, 9)	0.28, 0.28, 0.28	15	< 0.1	< 0.1

Table 137 Residues of dimethoate and omethoate in turnip roots in the USA during the 1994 growing season after 3 × 0.48 kg ai/ha foliar applications of a 400 g/L EC formulation (Samoil-1998)

Crop (variety), location, year	No. (RTI, days)	Rate (kg ai/ha)	DALA	Residues (mg/kg)	
				Dimethoate	Omethoate
Turnips (Purple Top), Fayetteville, AR, USA, 1994	3 (7, 7)	0.28, 0.28, 0.28	14	< 0.1	< 0.1
Turnips (Purple Top), Salinas, CA, USA, 1994	3 (7, 7)	0.28, 0.28, 0.28	15	< 0.1	< 0.1
Turnips (All Top), Gainesville, FL, USA, 1994	3 (8, 7)	0.28, 0.28, 0.28	13	< 0.1	< 0.1
Turnips (Purple Top), Tifton, GA, USA, 1994	3 (6, 7)	0.28, 0.28, 0.28	14	< 0.1	< 0.1
Turnips (Purple Top), Willard, OH, USA, 1994	3 (8, 10)	0.28, 0.28, 0.28	14	< 0.1	< 0.1
Turnips (Purple Top), Willard, OH, USA, 1994	3 (7, 8)	0.28, 0.28, 0.28	13	< 0.1	< 0.1
Turnips (Purple Top), Weslaco, TX, USA, 1994	3 (7, 8)	0.28, 0.28, 0.28	13	< 0.1	< 0.1

Asparagus

Two residue trials in asparagus conducted in Italy during the 2000 season were provided to the Meeting (Wilson – 2002c). The first application (of a 400 g/L EC formulation) was made during the early harvest period while spears were vigorously growing, around 3 weeks before scheduled harvest. Samples of asparagus spears were collected at intervals from 0 to 28 days after this application. A second application was made post-harvest (at the fern stage), with further spears being collected the following season. Samples were frozen within 7 hours of collection. The samples were extracted by maceration with dichloromethane, and an aliquot was evaporated to dryness. The extract was reconstituted in hexane, and cleaned up by liquid-liquid partition (addition of water). The hexane was

discarded and the aqueous phase made up to volume as necessary for analysis by LC-MS (LOQ = 0.01 mg/kg for both analytes). Concurrent recoveries ranged from 90–104% for dimethoate and 74–79% for omethoate. Samples were extracted and analysed within 5 months of collection.

Table 138 Residues of dimethoate and omethoate in asparagus spears (Wilson – 2002c)

Crop (variety), location, year	No.	Date, growth stage	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Asparagus (Hybrid SF144), Montanaso Lombardo, Lodi, Italy	1	20 April 2000 (8-10 cm spears)	0.42	1008	0	1.40 (1.27, 1.53)	0.02
					7	< 0.01	< 0.01 (ND)
					14	< 0.01 (ND)	< 0.01 (ND)
					21	< 0.01 (ND)	< 0.01
					28	< 0.01 (ND)	< 0.01 (ND)
	2	20 April 2000 (8-10 cm spears), 18 July 2000 (fern stage)	0.42, 0.42	1008, 1012	294	< 0.01 (ND)	< 0.01 (ND)
Asparagus (Eros), San Damiano Macra, Dronero, Italy	1	2 May 2000 (15-20 cm spears)	0.41	997	0	1.00 (0.88, 1.11)	< 0.01
					7	0.01	< 0.01 (ND)
					14	< 0.01 (ND)	< 0.01 (ND)
					21	< 0.01	0.01
					28	< 0.01 (ND)	< 0.01 (ND)
	2	2 May 2000 (15-20 cm spears), 9 August 2000 (fern stage)	0.41, 0.42	997, 1003	310	< 0.01 (ND)	< 0.01 (ND)

Cereal grains

Barley

Trials were conducted in barley in Europe in the 2008 and 2009 growing seasons (Raufer – 2009p and Raufer – 2010l). A single foliar application of a 400 g/L EC dimethoate formulation was made at BBCH 59 (end of heading) at 0.2 kg ai/ha. In magnitude of residue studies, whole plant samples were collected on the day of application, while grain and straw samples were collected at harvest maturity, while in decline trials, whole plant samples were collected between 0 and 14 days after application, ears and hay (whole plants cut and dried in the field or laboratory depending on weather for 7–12 days to achieve 80–90% dry matter before sampling) were collected at 21 days, and grain and straw at 42 days. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method with analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes in whole plants and straw, and 0.001 mg/kg in grain). Concurrent recoveries of dimethoate and

omethoate from fortified control samples ranged from 77–110%. Samples were analysed within 6 months of collection.

Table 139 Residues of dimethoate and omethoate in barley in Europe after a single 0.20 kg ai/ha foliar application of a 400 g/L EC formulation (Raufer – 2009p and Raufer – 2010l)

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Barley (Waggon), NG15, Papplewick, Nottinghamshire, UK, 2008	59	0.20	196	Whole plant	0	4.1	0.05	4.2
				Straw	56	< 0.01	< 0.01	< 0.02
				Grain	56	< 0.001	< 0.001	< 0.002
Barley (Carola), 64606, Popowko, Wielkopolska, Poland, 2008	59	0.21	208	Whole plant	0	2.5	0.02	2.5
				Straw	38	0.05	< 0.01	0.06
				Grain	38	0.016	0.003	0.019
Barley (Power), 5500, Middelfart, Fyn, Denmark, 2008*	53-55	0.21	208	Whole plant	0	7.5	0.08	7.6
				Straw	60	< 0.01	< 0.01	< 0.02
				Grain	60	< 0.001	< 0.001	< 0.002
Barley (Chess), 5500, Middelfart, Fyn, Denmark, 2008*	59	0.20	202	Whole plant	0	3.8	0.04	3.8
				Straw	63	< 0.01	< 0.01	< 0.02
				Grain	63	< 0.001	< 0.001	< 0.002
Barley (Widawa), 64600, Uscikowo, Wielkopolska, Poland, 2009	59	0.21	210	Whole plant	0	5.6	0.05	5.6
				Whole plant	7	0.08	0.07	0.15
				Whole plant	13	< 0.01	< 0.01	< 0.02
				Hay (DM = 85%)	21 (+12)	< 0.01 < 0.012 (DW)	< 0.01 < 0.012 (DW)	< 0.02 < 0.024 (DW)
				Ears	21	< 0.01	< 0.01	< 0.02
				Straw	42	< 0.01	< 0.01	< 0.02
				Grain	42	< 0.001	< 0.001	< 0.002
Barley (Waggon), PR4, Tarleton, Lancashire, UK, 2009	59	0.19	192	Whole plant	0	2.8	0.03	2.8
				Whole plant	7	0.10	0.08	0.18

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
				Whole plant	14	< 0.01	< 0.01	< 0.02
				Hay (DM = 69%)	21 (+8)	< 0.01 < 0.014 (DW)	< 0.01 < 0.014 (DW)	< 0.02 < 0.028 (DW)
				Ears	21	< 0.01	< 0.01	< 0.02
				Straw	48	< 0.01	< 0.01	< 0.02
				Grain	48	< 0.001	< 0.001	< 0.002
Barley (Quench), 5400, Bogense, Fyn, Denmark, 2009	59	0.20	198	Whole plant	0	5.3	0.04	5.3
				Whole plant	7	0.71	0.17	0.88
				Whole plant	14	0.32	0.10	0.42
				Hay (DM = 80%)	21 (+8)	0.11 0.14 (DW)	0.04 0.05 (DW)	0.15 0.19 (DW)
				Ears	21	0.04	0.03	0.07
				Straw	42	< 0.01	< 0.01	< 0.02
				Grain	42	< 0.001	< 0.001	< 0.002
Barley (Safron), 5500, Middelfart, Fyn, Denmark, 2009	59	0.20	195	Whole plant	0	5.1	0.03	5.1
				Whole plant	7	0.37	0.09	0.46
				Whole plant	14	0.03	0.02	0.05
				Hay (DM = 71%)	21 (+7)	< 0.01 < 0.014 (DW)	< 0.01 < 0.014 (DW)	< 0.02 < 0.028 (DW)
				Ears	21	< 0.01	< 0.01	< 0.02
				Straw	42	< 0.01	< 0.01	< 0.02
				Grain	42	< 0.001	< 0.001	< 0.002

No residues were detected in any of the untreated control samples, with the exception of a single whole plant sample where residues below the LOQ were detected for dimethoate and omethoate.

*The two Danish trials in 2008 were conducted with applications less than 1 month apart and in the same location and are not regarded as independent trials.

Wheat

Trials were conducted in wheat in Europe in the 2008 and 2009 growing seasons (Raufer – 2009q, Raufer – 2009r, Raufer – 2010m, and Raufer – 2010n). A single foliar application of a 400 g/L EC dimethoate formulation was made at BBCH 69 (end of flowering) at 0.2 kg ai/ha. In magnitude of residue studies, whole plant samples were collected on the day of application, while grain and straw samples were collected at harvest maturity, while in decline trials, whole plant samples were collected between 0 and 14 days after application, ears and hay (whole plants cut and dried in the field or

laboratory depending on weather for 7–12 days to achieve 80–90% dry matter before sampling) were collected at 21 days, and grain and straw at 42 days. Samples were frozen within 6 hours of collection and analysed for dimethoate and omethoate using a QuEChERS based method with analysis by LC-MS/MS (LOQ = 0.01 mg/kg for both analytes in whole plants and straw, and 0.001 mg/kg in grain). Concurrent recoveries of dimethoate and omethoate from fortified control samples ranged from 67–110%. Samples were analysed within 7 months of collection.

Table 140 Residues of dimethoate and omethoate in wheat in Europe after a single 0.20 kg ai/ha foliar application of a 400 g/L EC formulation (Raufer – 2009q, Raufer – 2009r, Raufer – 2010m, and Raufer – 2010n)

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Wheat (Paragon), S80, Thorpe Salvin, Nottinghamshire, UK, 2008	69	0.20	197	Whole plant	0	3.7	0.10	3.8
				Straw	35	< 0.01	< 0.01	< 0.02
				Grain	35	< 0.001	< 0.001	< 0.002
Wheat (Naridana), 64606, Osowo Nowe, Wielkopolska, Poland, 2008	69	0.20	197	Whole plant	0	3.3	0.03	3.3
				Straw	51	< 0.01	< 0.01	< 0.02
				Grain	51	< 0.001	< 0.001	< 0.002
Wheat (Certo), 86899, Ellighofen, Germany, 2008	69	0.21	207	Whole plant	0	4.6	0.05	4.6
				Straw	43	< 0.01	< 0.01	< 0.02
				Grain	43	< 0.001	< 0.001	< 0.002
Wheat (Smuggler), 5500, Middelfart, Fyn, Denmark, 2008	69	0.20	203	Whole plant	0	3.3	0.04	3.3
				Straw	49	< 0.01	< 0.01	< 0.02
				Grain	49	< 0.001	< 0.001	< 0.002
Wheat (Mesapia), 57018, Melissohori, Thessaloniki, Greece, 2008	69	0.20	203	Whole plant	0	2.4	0.02	2.4
				Straw	32	0.08	< 0.01	0.09
				Grain	32	< 0.001	< 0.001	< 0.002
Wheat (Anza), 50490, Villareal de Huerva, Zaragoza, Spain, 2008	69	0.21	206	Whole plant	0	3.2	0.10	3.3
				Straw	50	< 0.01	< 0.01	< 0.02

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
				Grain	50	< 0.001	< 0.001	< 0.002
Durum wheat (Ahilcar), 50368, Mainar, Zaragoza, Spain, 2008	69	0.21	207	Whole plant	0	3.6	0.06	3.7
				Straw	48	0.02	< 0.01	0.03
				Grain	48	< 0.001	< 0.001	< 0.002
Wheat (Mieti), 40054, Budrio, Emilia Romagna, Italy, 2008	69-71	0.21	211	Whole plant	0	3.9	0.17	4.1
				Straw	34	< 0.01	< 0.01	< 0.02
				Grain	34	< 0.001	< 0.001	< 0.002
Wheat (Bombona), 64606, Niemieczkowo, Wielkopolska, Poland, 2008	69	0.21	205	Whole plant	0	4.5	0.03	4.5
				Whole plant	7	0.15	0.08	0.23
				Whole plant	14	0.05	0.03	0.08
				Hay DM = 85%	21 (+12)	0.02 0.023 (DW)	< 0.01 < 0.011 (DW)	0.03 0.034 (DW)
				Ears	21	0.04	0.01	0.05
				Straw	41	0.01	< 0.01	0.02
				Grain	41	0.002	< 0.001	0.003
Wheat (Granary), L40 0RJ, Burscough Bridge, Lancashire, UK, 2009	69	0.21	208	Whole plant	0	4.0	0.12	4.1
				Whole plant	7	< 0.01	0.02	0.03
				Whole plant	14	< 0.01	< 0.01	< 0.02
				Hay DM = 77%	21 (+10)	< 0.01 < 0.013 (DW)	< 0.01 < 0.013 (DW)	< 0.02 < 0.026 (DW)
				Ears	21	< 0.01	< 0.01	< 0.02
				Straw	41	< 0.01	< 0.01	< 0.02
				Grain	41	< 0.001	< 0.001	< 0.002
Wheat (Brilliant), 16356, Blumberg,	69	0.21	213	Whole plant	0	3.0	0.03	3.0

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
Brandenburg, Germany, 2009								
				Whole plant	7	0.05	0.08	0.13
				Whole plant	14	< 0.01	0.02	0.03
				Hay DM = 83%	21 (+9)	< 0.01 < 0.012 (DW)	< 0.01 < 0.012 (DW)	< 0.02 < 0.024 (DW)
				Ears	21	< 0.01	< 0.01	< 0.02
				Straw	42	< 0.01	< 0.01	< 0.02
				Grain	42	< 0.001	< 0.001	< 0.002
Wheat (Taifun), 5500, Middelfart, Fyn, Denmark, 2009	69	0.20	196	Whole plant	0	4.5	0.05	4.6
				Whole plant	7	0.25	0.10	0.35
				Whole plant	14	0.01	0.02	0.03
				Hay DM = 82%	21 (+7)	< 0.01 < 0.012 (DW)	< 0.01 < 0.012 (DW)	< 0.02 < 0.024 (DW)
				Ears	21	< 0.01	< 0.01	< 0.02
				Straw	41	< 0.01	< 0.01	< 0.02
				Grain	41	< 0.001	< 0.001	< 0.002
Wheat (Messapia), 57018, Melissohori, Thessaloniki, Greece, 2009	69	0.20	201	Whole plant	0	4.5	0.02	4.5
				Whole plant	7	0.70	0.06	0.76
				Whole plant	14	0.56	0.05	0.61
				Hay DM = 89%	21 (+3)	0.33 0.37 (DW)	0.03 0.033 (DW)	0.36 0.40 (DW)
				Ears	21	0.18	0.04	0.22
				Straw	42	0.05	< 0.01	0.06
				Grain	42	0.002	< 0.001	0.003
Wheat (Boticelli), 50360, Daroca, Aragon, Spain, 2009	69	0.21	208	Whole plant	0	2.7	0.13	2.8
				Whole plant	7	0.07	0.10	0.17

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)	DM + OM (mg/kg)
				Whole plant	14	0.02	0.03	0.05
				Hay DM = 90%	21 (+7)	0.03 0.033 (DW)	0.02 0.022 (DW)	0.05 0.055 (DW)
				Ears	21	0.03	0.02	0.05
				Straw	42	< 0.01	< 0.01	< 0.02
				Grain	42	< 0.001	< 0.001	< 0.002
Wheat (Gallareta), 50490, Villareal de Huerva, Zaragoza, Spain, 2009	69	0.21	207	Whole plant	0	3.5	0.03	3.5
				Whole plant	7	0.83	0.07	0.90
				Whole plant	14	1.4	0.10	1.5
				Hay DM = 91%	21 (+4)	1.3 1.4 (DW)	0.07 0.077 (DW)	1.4 1.5 (DW)
				Ears	21	1.4	0.12	1.5
				Straw	42	0.83	0.074	0.90
				Grain	42	0.005	< 0.001	0.006
Wheat (Mieti), 40050, Funo, Bologna, Italy, 2009	69	0.21	213	Whole plant	0	4.6	0.06	4.7
				Whole plant	5	0.89	0.14	1.0
				Whole plant	14	0.21	0.07	0.28
				Hay DM = 82%	21 (+0)	0.17 0.21 (DW)	< 0.01 < 0.012 (DW)	0.18 0.22 (DW)
				Ears	21	0.02	0.07	0.09
				Straw	42	0.19	0.05	0.24
				Grain	42	< 0.001	0.001	0.002

No residues were detected in any of the untreated control samples, with the exception of one straw sample where a detectable dimethoate residue < LOQ was found.

A series of trials for wheat grown in Europe during the 2013 and 2014 growing seasons included analysis for six additional dimethoate metabolites (Eversfield – 2014a and Oxspring-2014). A single 0.2 kg ai/ foliar application of a 400 g/L dimethoate EC formulation was made at BBCH 69 (end of flowering). For the decline trials, treated and control whole plant, green ears/remaining plant, or straw/grain samples (depending on crop maturity) were collected at intervals from the day of application to harvest (38–50 days after application). For the magnitude of residues studies, samples of grain and straw were collected at harvest maturity. Samples were frozen within 8 hours of

collection and analysed for dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate.

Dimethoate, omethoate, and dimethoate carboxylic acid were analysed using a QuEChERS-based method with analysis by LC-MS/MS. For determination of O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, O-desmethyl omethoate, desmethyl dimethoate and O-desmethyl-N-desmethyl omethoate, samples were extracted with methanol/water (1:1 v/v), purified by solid phase extraction (Strata AX-W column, elution with 99:1 v/v methanol/35% ammonia), evaporated to dryness and reconstituted in dilute formic acid prior to LC-MS/MS analysis. Matrix-matching was employed. The LOQs were 0.01 mg/kg for each analyte. Concurrent method recoveries were within the ranges 71–106% (dimethoate), 77–111% (omethoate), 71–99% (dimethoate carboxylic acid), 68–103% (O-desmethyl omethoate carboxylic acid), 71–106% (O-desmethyl isodimethoate), 70–110% (desmethyl dimethoate), 74–103% (O-desmethyl omethoate), and 72–107% (O-desmethyl N-desmethyl omethoate), respectively. Extractions and analyses were completed within 8 months of sample collection.

Table 141 Residues of dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, and O-desmethyl isodimethoate in sugar beet in Europe during the 2014 growing season after a single 0.2 kg ai/ha foliar application of a 400 g/L EC formulation (Eversfield – 2014a and Oxspring-2014)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)					
						DM	OM	DM + OM (mg/kg)	DCA	ODI	OCA
Wheat (Gladiator), DE73 8BL, Wilson, Derbyshire, UK, 2013	69	0.22	223	Whole plant	0	4.45	0.04	4.5	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Green ear	7	0.32	0.18	0.50	< 0.01	< 0.01 (ND)	< 0.01
				Remaining plant	7	0.63	0.32	0.95	< 0.01	< 0.01 (ND)	< 0.01
				Green ear	14	0.07	0.06	0.13	< 0.01 (ND)	< 0.01 (ND)	< 0.01
				Remaining plant	14	0.31	0.04	0.35	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Grain	45	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	45	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Wheat (Courtot), 45300, Rouvres St Jean, Loiret, France, 2013	69	0.20	201	Whole plant	0	3.32	0.02	3.3	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Green ear	7	0.23	0.09	0.32	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	7	0.08	0.07	0.15	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Green ear	14	0.06	0.04	0.10	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Remaining	14	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)					
						DM	OM	DM + OM (mg/kg)	DCA	ODI	OCA
				plant					(ND)	(ND)	(ND)
				Grain	50	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	50	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (Tabasco), 21726, Kaken, Niedersachsen, Germany, 2013	69	0.20	203	Whole plant	0	3.20	0.05	3.3	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Green ear	6	0.25	0.14	0.39	< 0.01 (ND)	< 0.01 (ND)	< 0.01
				Remaining plant	6	0.05	0.07	0.12	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Green ear	13	< 0.01	0.04	0.05	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	13	< 0.01	0.01	0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Grain	50	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	50	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Wheat (Frisco), 71679, Asperg, Baden-Württemberg, Germany, 2013	69	0.20	202	Whole plant	0	4.03	0.03	4.1	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Green ear	7	0.12	0.08	0.20	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	7	0.01	0.02	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Green ear	14	0.10	0.10	0.20	0.08	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	14	< 0.01	0.02	0.03	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Grain	38	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	38	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (Areso), 71260, Montbellet, Bourgogne, France, 2014	69	0.19	193	24	Grain	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				24	Straw	0.02	< 0.01	0.03	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (Aprillio), 71290, Simandre, Saone-et-Loire,	69	0.21	210	35	Grain	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)					
						DM	OM	DM + OM (mg/kg)	DCA	ODI	OCA
France, 2014											
				35	Straw	0.01	< 0.01	0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (KWS Santiago), DE73 7FW, Twyford, Derbyshire, UK, 2014	69	0.20	207	45	Grain	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				45	Straw	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01 (ND)	< 0.01
Wheat (Kerobino), 71706, Markgröningen, Baden-Württemberg, Germany, 2014	69	0.21	217	47	Grain	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				47	Straw	< 0.01	< 0.01	< 0.02	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the untreated control samples.

DM = dimethoate; OM = omethoate; DCA = dimethoate carboxylic acid; ODI = O-desmethyl isodimethoate; OCA = O-desmethyl omethoate carboxylic acid.

Table 142 Residues of desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in wheat in Europe during the 2014 growing season after a single 0.2 kg ai/ha foliar application of a 400 g/L EC formulation (Eversfield – 2014a and Oxspring-2014)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
Wheat (Gladiator), DE73 8BL, Wilson, Derbyshire, UK, 2013	69	0.22	223	Whole plant	0	0.02	< 0.01 (ND)	< 0.01 (ND)
				Green ear	7	0.12	< 0.01	< 0.01 (ND)
				Remaining plant	7	0.16	< 0.01	< 0.01 (ND)
				Green ear	14	0.06	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	14	0.07	< 0.01	< 0.01 (ND)
				Grain	45	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Straw	45	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Wheat (Courtot), 45300, Rouvres St Jean, Loiret, France,	69	0.20	201	Whole plant	0	0.01	< 0.01	< 0.01 (ND)

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
2013								
				Green ear	7	0.08	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	7	0.04	< 0.01 (ND)	< 0.01 (ND)
				Green ear	14	0.03	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	14	0.02	< 0.01 (ND)	< 0.01 (ND)
				Grain	50	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	50	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (Tabasco), 21726, Kaken, Niedersachsen, Germany, 2013	69	0.20	203	Whole plant	0	0.02	< 0.01	< 0.01 (ND)
				Green ear	6	0.06	< 0.01	< 0.01 (ND)
				Remaining plant	6	0.03	< 0.01 (ND)	< 0.01 (ND)
				Green ear	13	0.05	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	13	0.02	< 0.01 (ND)	< 0.01 (ND)
				Grain	50	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	50	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (Frisco), 71679, Asperg, Baden-Württemberg, Germany, 2013	69	0.20	202	Whole plant	0	0.02	< 0.01 (ND)	< 0.01 (ND)
				Green ear	7	0.06	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	7	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				Green ear	14	0.03	< 0.01 (ND)	< 0.01 (ND)
				Remaining plant	14	0.01	< 0.01 (ND)	< 0.01 (ND)
				Grain	38	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				Straw	38	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (Areso), 71260, Montbellet, Bourgogne, France, 2014	69	0.19	193	24	Grain	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				24	Straw	< 0.01	< 0.01	< 0.01

Crop (variety), location, year	Growth stage (BBCH)	Rate (kg ai/ha)	Volume (L/ha)	Fraction analysed	DALA	Residues (mg/kg)		
						DMD	ODO	ONO
						(ND)	(ND)	(ND)
Wheat (Aprillio), 71290, Simandre, Saone-et-Loire, France, 2014	69	0.21	210	35	Grain	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				35	Straw	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
Wheat (KWS Santiago), DE73 7FW, Twyford, Derbyshire, UK, 2014	69	0.20	207	45	Grain	< 0.01	< 0.01 (ND)	< 0.01 (ND)
				45	Straw	< 0.01	< 0.01 (ND)	< 0.01 (ND)
Wheat (Kerobino), 71706, Markgröningen, Baden-Württemberg, Germany, 2014	69	0.21	217	47	Grain	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)
				47	Straw	< 0.01 (ND)	< 0.01 (ND)	< 0.01 (ND)

No residues were detected in any of the untreated control samples.

DMD = desmethyl dimethoate, ODO = O-desmethyl omethoate, ONO = O-desmethyl N-desmethyl omethoate.

Four trials were conducted in Germany and the UK in 2001 as part of a processing study (Wilson – 2003c). Plots were treated twice with a foliar application of a 400 g/L EC formulation of dimethoate. The first application was made at growth stage BBCH 73/75 and the second 7-11 days later. Each trial had two treated plots. Plot 2 was treated at 1×, i.e. 0.35 kg ai/ha (first application) and 5×, i.e. 1.75 kg ai/ha (second application) the recommended application rate. Plot 3 was treated at 5 fold for both applications.

Wheat whole plant RAC samples were taken 0 days after the last application (DALA), and then straw and grain RAC samples and grain processing samples were taken at earliest commercial harvest at 16-32 DALA.

Residues of dimethoate and omethoate in wheat RAC and processed commodities were quantitated by LC/MS/MS using a method described in study number SCI/050. Acceptable concurrent recovery data for wheat commodities were obtained for each analyte. Samples were analysed within 6 months of collection.

Table 143 Residue trials in wheat in Germany and the UK-2001 season (Wilson – 2003c)

Trial No., Location, Year (Variety)	No. (RTI, days)	Growth stage (BBCH)	Application rate (g a.i./ha)	Spray volume (L/ha)	Spray concentration (g a.i./100L)	Portion analysed	DALA	Residues (mg/kg)	
								Dimethoate	Omethoate
SCI/079-01, Manningtree, United Kingdom, 2001 (Claire)	2 (11)	73-75 77-83	379 1889	214 214	177 883	Green plant	0	29.26	0.05
						Straw (RAC)	16	2.55	0.10
						Grain (RAC)	16	0.017	0.002

Trial No., Location, Year (Variety)	No. (RTI, days)	Growth stage (BBCH)	Application rate (g a.i./ha)	Spray volume (L/ha)	Spray concentration (g a.i./100L)	Portion analysed	DALA	Residues (mg/kg)	
								Dimethoate	Omethoate
SCI/079-02,	2	73-75	375	213	176	Green plant	0	29.70	0.09
Tendring,	(7)	77-83	1883	214	880	Straw (RAC)	32	0.74	0.02
United Kingdom, 2001						Grain (RAC)	32	0.026	0.003
(Hereward)									
SCI/079-03,	2	75	346	196	177	Green plant	0	30.64	0.10
Brunne,	(7)	77	1790	203	882	Straw (RAC)	21	0.87	0.04
Germany, 2001						Grain (RAC)	21	0.038	0.005
(Aristos)									
SCI/079-04,	2	75	355	201	177	Green plant	0	35.93	0.09
Blumberg,	(8)	77	1801	204	883	Straw (RAC)	18	2.98	0.09
Germany, 2001						Grain (RAC)	18	0.110	0.007
(Pegassos)									

DALA = Days After Last Application

LOQ of dimethoate and omethoate = 0.01 mg/kg for wheat green plant and straw

LOQ of dimethoate and omethoate = 0.001 mg/kg for wheat grain

In another study, eight residues trials were conducted on wheat at four sites in Southern France and four sites in Spain in 2014 (Tandy-2015).

Table 144 Residues of dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in wheat after a single application of a 400 g/L EC formulation – 2014 season in France (Tandy – 2015)

Trial No., Location, Year, Variety	No.	Growth stage (BBCH)	Appl'n rate (g ai/ha)	Spray volume (L/ha)	Spray conc (g ai/100L)	DAA	Portion analysed	Residues (mg/kg)						
								DM	OM	DM + OM (mg/kg)	DCA	ODOCA	DD	ODO
S14-01362-01,	1	69-71	158	158	100	39	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
						39	Straw	0.02	< 0.01	0.03	< 0.01	< 0.003	< 0.01	< 0.003
Treatment 2														
Meauzac, Tarn et Garonne,														
France, 2014														
(Winter wheat-Soleilho)														
S14-01362-02,	1	69	232	232	100	50	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
						50	Straw	0.01	< 0.01	0.02	< 0.003	< 0.003	< 0.003	< 0.003
Montech, Tarn et Garonne,														
France, 2014														

Trial No., Location, Year, Variety (Winter wheat-Ingenio)	No.	Growth stage (BBCH)	Appl'n rate (g ai/ha)	Spray volume (L/ha)	Spray conc (g ai/100L)	DAA	Portion analysed	Residues (mg/kg)						
								DM	OM	DM + OM (mg/kg)	DCA	ODOCA	DD	ODO
S14-01362-03, Bages, Pyrenees Orientales, France, 2014 (Winter wheat-Sirtaki)	1	69	202	202	100	0	Whole plant	4.61	0.07	4.7	< 0.01	< 0.003	0.04	< 0.01
						7	Whole plant	1.69	0.29	2.0	< 0.01	< 0.01	0.22	0.01
						12	Whole plant	0.59	0.16	0.76	< 0.003	< 0.003	0.11	< 0.01
						38	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.01	< 0.003
						38	Straw	0.76	0.05	0.81	< 0.01	< 0.003	0.04	0.06
							Straw	0.57	0.07	0.64	< 0.01	< 0.01	0.03	0.06
							Straw	0.63	0.06	0.69	< 0.01	< 0.003	0.04	0.08
								Mean = 0.65	Mean = 0.06	Mean = 0.71				
S14-01362-04, Villareal de Huerva, Aragon, Spain, 2014 (Winter wheat-Botticelli)	1	69	208	208	100	0	Whole plant	8.27	0.09	8.4	0.01	< 0.003	0.05	0.01
						7	Whole plant	0.96	0.10	1.1	< 0.003	< 0.01	0.08	< 0.01
						14	Whole plant	0.25	0.04	0.29	< 0.003	< 0.003	0.05	< 0.003
						56	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
						56	Straw	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
S14-01362-05, Lagueruela, Teruel, Spain, 2014 (Winter wheat-Marius)	1	69	194	194	100	0	Whole plant	6.06	0.12	6.2	< 0.01	< 0.003	0.05	< 0.01
						7	Whole plant	0.59	0.14	0.74	< 0.003	< 0.003	0.11	< 0.01
						14	Whole plant	0.32	0.12	0.45	< 0.01	< 0.003	0.04	< 0.003
						41	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
						41	Straw	0.05	0.01	0.06	< 0.01	< 0.003	< 0.01	< 0.01
S14-01362-06, Fonfria, Aragon, Spain, 2014 (Winter wheat-Marius)	1	69	208	207	100	50	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
						50	Straw	0.04	< 0.01	0.05	< 0.01	< 0.003	< 0.003	< 0.003
S14-01362-07, Torralba de los Frailes, Spain, 2014 (Winter wheat-Marius)	1	69	215	213	101	35	Grain	< 0.01	< 0.01	< 0.02	< 0.003	< 0.003	< 0.003	< 0.003
						35	Straw	0.05	< 0.01	0.06	< 0.01	< 0.003	< 0.003	< 0.003
S14-01362-08,	1	69	207	207	100	0	Whole plant	6.52	0.03	6.6	0.01	< 0.003	0.04	0.01

Trial No., Location, Year, Variety	No.	Growth stage (BBCH)	Appl'n rate (g ai/ha)	Spray volume (L/ha)	Spray conc (g ai/100L)	DAA	Portion analysed	Residues (mg/kg)						
								DM	OM	DM + OM (mg/kg)	DCA	ODOCA	DD	ODO
Saint Cyprien, Pyrenees Orientales, France, 2014 (Winter wheat-Babylone)						7	Whole plant	2.49	0.17	2.7	< 0.01	< 0.003	0.16	0.02
						12	Whole plant	1.52	0.13	1.7	< 0.01	< 0.003	0.05	< 0.01
						33	Grain	0.01	< 0.01	0.02	< 0.003	< 0.003	< 0.01	< 0.003
						33	Straw	0.88	0.02	0.90	0.12	0.02	0.21	0.02
						33	Straw	0.58	0.02	0.60	0.11	0.05	0.14	0.02
						33	Straw	0.58	0.01	0.59	0.11	0.03	0.12	0.01
								Mean = 0.68	Mean = 0.02	Mean = 0.70				

LOQ of dimethoate and metabolites = 0.01 mg/kg for all matrices

LOD of dimethoate and metabolites = 0.003 mg/kg for all matrices

DCA = Dimethoate carboxylic acid

ODOCA = O-desmethyl omethoate carboxylic acid

DD = Desmethyl dimethoate

ODO = O-desmethyl omethoate

Note: Residues of O-desmethyl iso-dimethoate were < 0.003 mg/kg in all samples except 7 day whole plant in trial S14-01362-08 (< 0.01 mg/kg)

Residues of O-desmethyl N-desmethyl omethoate were < 0.003 mg/kg in all samples

Rape seed

A series of 8 field trials in oilseed rape (canola) was conducted in Australia during the 2011/12 growing season (Haller – 2012). A single foliar application of a 400 g/L EC formulation at a target rate of 0.14 kg ai/ha was made to the treated plots at each site, with the trials being conducted using a reverse decline design, with the application made at intervals of 0 to 21 days before harvest, and seed and fodder collected from all plots simultaneously at harvest and frozen within 6 hours of collection. Samples were analysed for dimethoate and omethoate using a QuEChERS-based method (AATM-S-60.1, revision 2), with analysis by LC-MS/MS. A method LOQ of 0.02 mg/kg in seed and 0.05 mg/kg in fodder was demonstrated for both analytes. Individual concurrent recoveries were in the range 79–125%. Analyses were completed within 5 months of sample collection.

Table 145 Residues of dimethoate and omethoate in canola in Australia after a single 0.14 kg ai/ha foliar application of a 400 g/L EC formulation (Haller-2012)

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Residues (mg/kg)		
						DM	OM	DM + OM
Canola (Clearfield), Freeling, SA 5372, Australia, 2011/12	81	0.14	100	Seed	21	0.051	< 0.02	0.07
				Fodder (as received)	21	0.084	< 0.05	0.13
	83	0.14	100	Seed	14	0.047	< 0.02	0.067
				Fodder (as received)	14	0.48	< 0.05	0.53

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Residues (mg/kg)		
						DM	OM	DM + OM
	NR	0.14	100	Seed	7	0.02	< 0.02	0.04
1				Fodder (as received)	7	1.2	< 0.05	1.2
	89	0.14	100	Seed	0	0.17	< 0.02 (ND)	0.19
				Fodder (as received)	0	1.0 (FW)	< 0.05	1.0
Canola (46Y83), Freeling, SA 5371, Australia, 2011/12	NR	0.14	100	Seed	21	0.084	< 0.02	0.086
				Fodder (as received)	21	0.12	< 0.05	0.17
	85	0.14	101	Seed	14	0.045	< 0.02	0.065
				Fodder (as received)	14	0.54	< 0.05	0.59
	NR	0.14	100	Seed	7	< 0.02	< 0.02	< 0.04
				Fodder (as received)	7	1.1	< 0.05	1.2
	89	0.14	100	Seed	0	0.091	< 0.02	0.11
				Fodder (as received)	0	3.1	< 0.05	3.2
Canola (Scaddan), Gerogery, NSW 2642, Australia, 2011/12	82	0.14	68	Seed	21	< 0.02	< 0.02	< 0.04
				Fodder (as received)	21	< 0.05	< 0.05	< 0.10
	84	0.14	68	Seed	13	0.02	< 0.02	< 0.04
				Fodder (as received)	13	< 0.05 0.1 (FW)	< 0.05	< 0.10
	86	0.14	68	Seed	6	0.027	< 0.02	0.047
				Fodder (as received)	6	0.21	< 0.05	0.26
	89	0.15	71	Seed	0	0.16	< 0.02	0.18
				Fodder (as received)	0	2.4	< 0.05	2.4
Canola (Roundup Ready – GT61), Buckland, WA 6401, Australia, 2011/12	87-88	0.14	100	Seed	21	< 0.02	< 0.02	< 0.04

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Residues (mg/kg)		
						DM	OM	DM + OM
				Fodder (as received)	21	1.1	< 0.05	1.2
	88	0.14	100	Seed	14	< 0.02	< 0.02	< 0.04
				Fodder (as received)	14	5.5 (6.1, 4.9)	0.12 (0.11, 0.14)	5.6 (6.2, 5.0)
	89	0.14	100	Seed	7	< 0.02	< 0.02	< 0.04
				Fodder (as received)	7	4.4 (4.4, 4.5)	0.15 (0.15, 0.15)	4.6 (4.6, 4.6)
	89	0.14	100	Seed	0	0.12	< 0.02	0.14
				Fodder (as received)	0	6.0 (5.3, 6.8)	0.04 (0.04, 0.05)	6.0 (5.3, 6.8)
Canola (TT – Cobbler), Jennapullin, WA 6401, Australia, 2011/12	87-88	0.14	100	Seed	21	< 0.02	< 0.02	< 0.04
				Fodder (as received)	21		< 0.05 ()	1.2
	88	0.14	100	Seed	14	0.02	< 0.02	0.04
				Fodder (as received)	14	6.8 (7.2, 6.4)	0.14 (0.12, 0.15)	6.9 (7.3, 6.6)
	89	0.14	100	Seed	7	< 0.02	< 0.02	< 0.04
				Fodder (as received)	7	2.8 (2.3, 3.4)	0.095 (0.080, 0.11)	2.9 (2.4, 3.5)
	89	0.14	100	Seed	0	0.085	< 0.02	0.10
				Fodder (as received)	0	8.2 (8.6, 7.7)	0.05 (0.05, 0.05)	8.2 (8.6, 7.8)
Canola (555TT), Deloraine, TAS 7304, Australia, 2011/12	88	0.14	100	Seed	21	< 0.02	< 0.02	< 0.04
				Fodder (as received)	21	0.05	< 0.05	< 0.10
	88	0.14	100	Seed	14	0.04	< 0.02	0.06
				Fodder (as received)	14	0.64	< 0.05	0.69
	89	0.15	103	Seed	7	0.066	< 0.02	0.086
				Fodder (as received)	7	1.2	< 0.05	1.2
	89	0.14	100	Seed	0	0.17	< 0.02	0.19
				Fodder (as received)	0	2.5	< 0.05	2.6
Canola (Hyola 575), Balliang,	89	0.14	94	Seed	21	< 0.02	< 0.02	< 0.04

Crop (variety), location, year	Growth stage (BBCH)	Application rate (kg ai/ha)	Spray volume (L/ha)	Portion analysed	DALA	Residues (mg/kg)		
						DM	OM	DM + OM
VIC 3340, Australia, 2011/12								
				Fodder (as received)	21	0.37	0.067	0.44
	89	0.13	91	Seed	14	0.026	< 0.02	0.046
				Fodder (as received)	14	2.1	0.16	2.3
	89	0.14	96	Seed	7	< 0.02	< 0.02	< 0.04
				Fodder (as received)	7	2.8	0.17	3.0
	89	0.14	99	Seed	0	0.083	< 0.02	0.10
				Fodder (as received)	0	2.9	< 0.05	3.0
Canola (46Y83), Avalon, VIC 3212, Australia, 2011/12	79-80	0.14	99	Seed	20	< 0.02	< 0.02	< 0.04
				Fodder (as received)	20	0.069	< 0.05	0.074
	81	0.14	96	Seed	14	< 0.02	< 0.02	< 0.04
				Fodder (as received)	14	0.067	< 0.05	0.12
	82	0.14	98	Seed	7	0.028	0.02	0.048
				Fodder (as received)	7	1.0	0.10	1.1
	89	0.14	100	Seed	0	0.073	< 0.02	0.093
				Fodder (as received)	0	3.4	< 0.05	3.4

Residues in all untreated control samples were <LOQ.

Sugar beet tops

Residue data in sugar beet leaves (tops) was collected as part of the residue trials in sugar beet (refer to Table 131).

Cereal forage and fodder

Residue data in barley hay (Table 137), barley straw (Table 137), wheat hay (Table 138) and wheat straw (Table 138, Table 139, andTable 142) was collected as part of the cereal residue trials.

Legume forage and fodder

Table 146 Residues of dimethoate and omethoate in pea vines after a single application of dimethoate – USA trials 1993 (Rice-1994)

Crop (variety), location,	Application rate	Spray volume	DALA	Dimethoate	Omethoate

year	(kg ai/ha)	(L/ha)		(mg/kg)	(mg/kg)
Peas, (Knight), Hillsboro, OR, USA, 1993 DM = 21.1%	0.19	161	0	5.36 (5.26, 5.45)	0.17 (0.16, 0.18)
			3	3.38 (2.97, 3.78)	0.42 (0.39, 0.45)
			7	1.62 (2.06, 1.18) c0.01	0.42 (0.47, 0.36)
Peas (FR652), Moses Lake, WA, USA, 1993 DM = 22.6%	0.19	163	0	4.58 (4.34, 4.83)	0.04 (0.05, 0.03)
			3	2.18 (1.83, 2.53)	0.23 (0.25, 0.21)
			7	3.48 (3.58, 3.38)	0.32 (0.43, 0.21)
Peas (Sunburst), Lake Mills, WI, USA, 1993 DM = 17.9%	0.19	161	0	5.66 (4.44, 6.87)	0.19 (0.19, 0.19)
			3	1.15 (1.43, 0.87)	0.84 (1.13, 0.54)
			7	0.22 (0.27, 0.18)	0.38 (0.40, 0.36)
Peas (77 EP/Nunhems), Verona, MI, USA, 1993 DM 21.4%	0.19	151	0	6.18 (5.70, 5.99, 5.71, 7.32)	0.32 (0.24, 0.34, 0.33, 0.37)
			3	2.19 (1.86, 2.63, 1.89, 2.38)	0.81 (0.84, 0.87, 0.75, 0.77)
			7	0.09 (0.09, 0.10, 0.08, 0.10)	0.08 (0.07, 0.05, 0.13, 0.10)

Except where otherwise noted, no residues were detected in any of the untreated control samples.

Table 147 Residues of dimethoate and omethoate in pea hay after a single application of dimethoate – USA trials 1993 (Rice-1994)

Crop (variety), location, year	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Peas, (Knight), Hillsboro, OR, USA, 1993 DM = 67.6%	0.19	161	0	10.82 (11.48, 10.16)	1.38 (1.56, 1.20)
			3	6.76 (5.86, 7.67)	1.38 (1.01, 1.76)
			7	3.98 (5.92, 2.03)	0.96 (1.06, 0.86)
Peas (FR652), Moses Lake, WA, USA, 1993 DM = 76.1%	0.19	163	0	9.86 (10.49, 9.22)	0.38 (0.49, 0.26)
			3	6.10 (5.25, 6.95)	0.38 (0.39, 0.37)
			7	4.64 (5.15, 4.46, 4.80, 4.15)	0.34 (0.52, 0.33, 0.28, 0.24)
Peas (Sunburst), Lake Mills, WI, USA, 1993 DM = 42%	0.19	161	0	1.66 (1.68, 1.64)	0.76 (0.75, 0.76)
			3	1.10 (0.95, 1.25)	0.70 (0.59, 0.80)
			7	0.36 (0.40, 0.33)	0.75 (0.78, 0.72)
Peas (77 EP/Nunhems),	0.19	151	0	9.24 (8.68, 7.49,	1.66 (1.57, 1.45,

Crop (variety), location, year	Application rate (kg ai/ha)	Spray volume (L/ha)	DALA	Dimethoate (mg/kg)	Omethoate (mg/kg)
Verona, MI, USA, 1993 DM = 64.8%				10.29, 10.50)	1.90, 1.74)
			3	0.85 (0.86, 0.95, 0.75)	0.29 (0.28, 0.31, 0.28)
			7	0.14 (0.09, 0.17, 0.17)	0.11 (0.07, 0.13, 0.13)

No residues were detected in any of the untreated control samples.

Pulse forage and fodder

Table 148 Residues of dimethoate and omethoate in mung bean forage (Litzow – 2013)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	Moisture (%)
Mung beans, Crystal, Narrabri, NSW, 120277	UTC	-	-	-	0.05*	0.04*	< 0.09	41.2
	320	3 (14)	91, 94, 92	0	34.4	0.29	34.7	58.8
				7	1.57	0.11	1.69	32.1
	640	3 (14)	91, 93, 93	0	53.6	0.33	54.0	57.0
				7	4.40	0.38	4.80	31.3
Mung beans, Crystal, Edgeroi, NSW, 120278	UTC	-	-	-	< 0.02	< 0.02	< 0.04	61.0
	320	3 (14)	91, 94, 92	0	23.2	0.22	23.5	47.9
				7	3.96	0.30	4.28	27.0
Mung beans, Crystal, Wellcamp, Qld, 120279	UTC	-	-	-	< 0.02	< 0.02	< 0.04	58.1
	320	3 (14)	96, 107, 104	0	35.4	1.14	36.6	57.9
				7	3.81	1.17	5.06	67.9

Table 149 Residues of dimethoate and omethoate in navy bean forage (Litzow – 2013)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	Moisture (%)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	Moisture (%)
Navybeans, Arwon, Kumbia, Qld, 120280	UTC	-	-	-	< 0.02	< 0.02	< 0.04	80.3
	320	3 (14)	99, 110, 108	0	67.0	1.66	68.8	82.5
				7	3.19	1.43	4.73	78.2
	640	3 (14)	99, 110, 108	0	98.5	2.20	101	82.5
				7	6.92	2.51	9.62	75.8
Navybeans, Arwon, Kumbia, Qld, 120281	UTC	-	-	-	< 0.02	< 0.02	< 0.04	79.2
	320	3 (14)	100, 110, 107	0	42.4	1.33	43.8	79.2
				7	1.45	1.54	3.11	74.2
Navybeans, Spearfelt, Euberta, NSW, 120282	UTC	-	-	-	< 0.02	< 0.02	< 0.04	61.8
	320	3 (13, 15)	127, 133, 108	0	23.4	0.87	24.3	56.3
				7	2.02	0.24	2.28	36.7

* between LOD and LOQ

Table 150 Residues of dimethoate and omethoate in soya bean forage (Litzow – 2013)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Sample	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	Moisture (%)
Soya beans, Rose, Collegeview, Qld, 120283	UTC	-	-	Forage	-	< 0.02	< 0.02	< 0.04	32.1
	320	3 (13, 15)	83, 95, 82	Forage	0	14.0	0.085	14.1	36.0
					7	2.31	0.086	2.40	14.5
	640	3 (13, 15)	82, 95, 82	Forage	0	15.3	0.086	15.4	39.0
					7	2.77	0.18	2.96	34.4
	UTC	-	-	Trash	-	< 0.02	< 0.02	< 0.04	20.8
Soya beans, Ascott, Condamine Plains, Qld, 120284	UTC	-	-	Forage	-	< 0.02	< 0.02	< 0.04	70.3

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Sample	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	Moisture (%)
	320	3 (14)	83, 102, 82	Forage	0	49.0	1.80	50.9	72.1
					7	0.89	0.60	1.54	69.3
Soya beans, Bunya, Edgeroi, NSW, 120285	UTC	-	-	Forage	-	0.04*	< 0.02	0.06	48.9
	320	3 (14)	92	Forage	0	22.5	0.71	23.3	39.3
					6	9.93	0.51	10.5	28.7

* between LOD and LOQ

Table 151 Residues of dimethoate and omethoate in mung bean trash (Litzow – 2013)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	DM + 7 × OM (mg/kg)
Mung beans, Crystal, Narrabri, NSW, 120277	UTC	-	-	-	0.095	< 0.02	0.097	0.24
	320	3 (14)	91, 94, 92	13	2.07	0.079	2.16	2.62
	640	3 (14)	91, 93, 93	13	4.43	0.22	4.66	5.97
Mung beans, Crystal, Edgeroi, NSW, 120278	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3(14)	91, 94, 92	13	1.77	0.19	1.97	3.10
Mung beans, Crystal, Wellcamp, Qld, 120279	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3 (14)	96, 107, 104	14	1.63	0.09*	1.73	2.26

Table 152 Residues of dimethoate and omethoate in navy bean trash (Litzow – 2013)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	DM + 7 × OM (mg/kg)
Navybeans, Arwon, Kumbia, Qld, 120280	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3 (14)	99, 110, 108	14	0.45	0.75	1.25	5.70
	640	3 (14)	99, 110, 108	14	1.38	1.91	3.44	14.8
Navybeans, Arwon, Kumbia, Qld, 120281	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3(14)	100, 110, 107	14	0.15*	0.25	0.42	1.90
Navybeans, Spearfelt, Euberta, NSW, 120282	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3 (13, 15)	127, 133, 108	14	5.04	0.37	5.43	7.63

Table 153 Residues of dimethoate and omethoate in soya bean trash (Litzow – 2013)

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	DM + 7 × OM (mg/kg)
Soya beans, Rose, Collegeview, Qld, 120283	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3 (13, 15)	83, 95, 82	14	2.89	0.10	2.99	3.59
	640	3 (13, 15)	82, 95, 82	14	3.09	0.17	3.27	4.28
Soya beans, Ascott, Condamine Plains, Qld, 120284	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16
	320	3 (14)	83, 102, 82	14	0.12	0.053*	0.18	0.49
Soya beans,	UTC	-	-	-	< 0.02	< 0.02	< 0.04	< 0.16

Crop, variety, location, site no.	Application rate (g ai/ha)	No. of applications, (interval, days)	Spray volume (L/ha)	Days after last application	Dimethoate (mg/kg) (dry weight)	Omethoate (mg/kg) (dry weight)	DM + OM (mg/kg) (dry weight)	DM + 7 × OM (mg/kg)
Bunya, Edgeroi, NSW, 120285								
	320	3 (14)	92	14	6.52	0.59	7.15	10.6

* between LOD and LOQ

Residues in processed commodities

Hydrolysis study

The effect of hydrolytic conditions on the nature of dimethoate and omethoate residues was tested separately for the two compounds using radiolabels (Aikens – 1999). Solutions of the two compounds (labelled with carbon-14 at the methoxy groups) were prepared in buffer solutions at a concentration of 1 µg/mL, and incubated in the dark under three different regimes simulating different food processes:

- pH 4, 90 °C, 20 minutes (simulating pasteurisation);
- pH 5, 100 °C, 60 minutes (simulating baking, brewing or boiling); and
- pH 6, 120 °C, 20 minutes (simulating sterilisation)

Samples were analysed after incubation by HPLC and TLC with reference substances and where necessary, with LC-MS to assist with identifying degradation products.

Table 154 Hydrolysis of ^{14}C-(methoxy)-dimethoate under simulated processing conditions

Residue component	% applied radioactivity		
	pH 4, 90 °C, 20 minutes	pH 5, 100 °C, 60 minutes	pH 6, 120 °C, 20 minutes
Unidentified component 1	0.5	1.2	2.4
Unidentified component 2	0.4	0.8	1.2
Unidentified component 3	0.9	2.5	2.4
Des-O-methyl isodimethoate (tentative identification)	0.3	0.9	5.3
Desmethyl dimethoate	4.7	28.1	59.5
Dimethoate	93.3	66.4	29.5

Table 155 Hydrolysis of ^{14}C-(methoxy)-omethoate under simulated processing conditions

Residue component	% applied radioactivity		
	pH 4, 90 °C, 20 minutes	pH 5, 100 °C, 60 minutes	pH 6, 120 °C, 20 minutes
Unidentified component 1	ND	0.1	3.0
Dimethyl phosphoric acid	0.2	1.5	19.2
O-desmethyl omethoate	6.3	36.2	62.6
Omethoate	92.9	61.3	5.5

Neither dimethoate nor omethoate hydrolysed significantly under conditions simulating pasteurisation.

A greater degree of hydrolysis observed under conditions simulating baking, boiling or brewing, with approximately 34% of the applied dimethoate and 39% of omethoate hydrolysed after 1 hour at pH 5/100 °C. The main products of hydrolysis were the O-desmethylated metabolites in both cases (28% and 36% for dimethoate and omethoate respectively).

A further increase in hydrolysis was observed under conditions simulating sterilisation, with over 70% of dimethoate and > 90% of omethoate hydrolysed after 20 minutes at pH 6/120 °C. Again, O-desmethyl dimethoate and O-desmethyl omethoate were the major hydrolysates (60% and 63% respesctively). For omethoate, dimethyl phosphoric acid was also observed at 19% of the AR.

No conversion of dimethoate to omethoate was observed during the experiment.

Residues after processing

The fate of dimethoate residues during processing of raw agricultural commodities was investigated in citrus fruit (orange), assorted tropical and sub-tropical fruits-edible peel (olives), Brassica vegetables (cabbage) and cereals (wheat).

As a measure for the transfer of residues into processed products, a transfer factor (TF) was used, which is defined as:

$$TF = \frac{\text{Residue in processed products (mg/kg)}}{\text{Residue in raw agricultural commodity (mg/kg)}}$$

Oranges

The effect of processing (laboratory scale) on residues of dimethoate in oranges (Table 144) was investigated in one field trial conducted in the USA during the 1993 growing season (Rice *et al* 1994, 40898, 177 DMT).

Two applications of a 480 g/L EC dimethoate formulation were made to oranges at 8513 and 8.626g ai/ha with a retreatment interval of 14 days. Oranges were harvested 14 days after the last application and processed to juice, dry pulp, molasses and oil and analysed for residues of dimethoate and omethoate.

Fruits were washed in a processing line. Fruits were washed for about 30 seconds and rinsed with potable water, brushed to remove excess water and dried at ambient temperature or at about 40 °C to give washed whole oranges. Fruit juice was obtained using commercial equipment equipped with continuous water-spray nozzles for maximum recovery of peel oil. The extracted juice was collected and pumped to a juice finisher. The finisher screens excess pulp from the juice. Fresh juice and finisher pulp were collected and stored frozen.

The oil/water/peel-frit emulsion from the extractor was passed through a 0.05 cm screen to separate the peels. The oil/water emulsion was further screened (0.02 cm) to remove insoluble fibres and stored for 5 hours at ambient temperatures. The lower unemulsified water phase was removed and the oil emulsion further stored at 4 °C for 16 hours. The oil was separated using a continuous centrifuge and stored at -18 °C for 16 hours to freeze out remaining water. The cold-pressed oil was filtered to remove solids and remaining water.

The peel from the extractor was collected and transferred to a plant feed mill. The sample was chopped and passed through a 1.75 cm screen to collect the wet pulp. Liquid lime slurry (0.12 kg/L water) was added at 0.3% lime per weight of wet pulp and pressed to separate the press liquor from the press cake. The press cake was dried at 143°C to a moisture content of 8–10%.

The press liquor was passed through a shaker screen and heated to boil under vacuum. The liquor was concentrated to approximately 50° Brix to obtain molasses.

Residues of dimethoate and omethoate in orange RAC and processed commodities were quantitated by the method entitled "Determination of Dimethoate and Omethoate Residues in Oranges and Its Processed Commodities by Gas Chromatography". Acceptable concurrent recovery data for orange commodities were obtained for both analytes. Samples were analysed within 2 months of collection.

Table 156 Residues in orange processed fractions from the foliar application of dimethoate to oranges in the USA (Rice *et al* 1994, 40898, 177 DMT)

Trial No., Location, Year, (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate*	Omethoate*	Dimethoate	Omethoate
98.FL.OR Loxahatchee, Southeastern Florida, USA, 1993 (Valencia) 2 applications 14 day RTI Growth stage of last application-86 8626, 8513 g ai/ha 1928, 1904 L/ha	Whole oranges-unwashed	14	1.45	0.15	-	-
	Whole oranges-washed	14	1.51	0.16	1.04	1.07
	Juice	14	0.21	0.03	0.14	0.20
	Dry pulp	14	3.05	0.24	2.1	1.6
	Molasses	14	8.44	0.89	5.8	5.9
	Oil	14	0.29	< 0.01	0.20	< 0.07

DALA = Days After Last Application

* Mean value

LOQ of dimethoate and omethoate = 0.01 mg/kg for oranges and all processed commodities

Cabbage

The effect of processing (laboratory scale) on residues of dimethoate in white cabbage (Table 145) was investigated in one field trial conducted in Germany during the 1999 growing season (Schulz 2000, IF-99/09379-00, 409 DMT).

Cabbages with incurred residues were obtained after plants were treated with six foliar sprays of a 400 g/L EC dimethoate formulation at seven day intervals, with a pre-harvest interval of 21 days. Five applications were made at a nominal rate of 1 L/ha (400g a.i./ha) and the sixth application with a nominal rate of 3 L/ha (1.2 kg a.i./ha). The actual application rates were 400–409 and 1233 g a.i./ha). The wetting agent Agral was added (0.03%).

Cabbage bulk RAC samples were harvested 21 days after the last application (BBCH growth stage 49). Cabbage head samples were processed into salad and cooked cabbage.

The outer leaves from 6 heads were removed. The outer stalks were cut up. The heads were then quartered and the inner stalks removed. The outer and inner stalks were combined and considered as the stalks sample. The rest of the quartered heads were sliced to obtain cabbage salad samples.

The outer leaves and stalks of 6 heads were removed and discarded. The remaining cores were boiled covered with tap water until done (fork test). The heads were then transferred into a plastic dish and quartered with a knife. During heating up, the boiling process and cooling, the saucepan was covered with a lid. The quartered cores and the boiled water were allowed to cool overnight (cooked white cabbage). The remaining water in the plastic dish and the boiled water were combined. An aliquot of boiled water was taken for analysis.

Residues of dimethoate and omethoate in cabbage RAC and processed commodities were quantitated by the method described in Agrisearch study number AK/3377/CN validated under study

number IF-99/13623-00. Acceptable concurrent recovery data for cabbage commodities were obtained for both analytes. Samples were analysed within 5 months of collection.

Table 157 Residues in cabbage processed fractions from the foliar application of dimethoate to cabbages in Germany (Schulz 2000, IF-99/09379-00, 409 DMT)

Trial No., Location, Year (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
AT-99/020-0, Gernsheim-Allmendfeld, Hesse, Germany, 1999 (Galaxy) 6 applications 7 day RTI Growth stage of last application - 47 403, 400, 403, 409, 409, 1,233 g ai/ha 302, 599, 603, 613, 613, 617 L/ha	White cabbage (RAC)	21	0.0514	0.0828	1	1
	Outer leaves		0.187	0.495	3.7	6.0
	Core, cut to salad		0.0108	< 0.01	0.2	< 0.12
	Stalks		0.041	< 0.01	0.8	< 0.12
	Cooked white cabbage		< 0.01	< 0.01	< 0.19	< 0.12
	Boiled water		< 0.01	< 0.01	< 0.19	< 0.12

DALA = Days After Last Application

LOQ of dimethoate and omethoate = 0.01 mg/kg for cabbage and all processed commodities

NC = Not Calculable

Olives

The effect of processing (laboratory scale) on residues of dimethoate and omethoate in olives (Table 146) was investigated in one field trial conducted in Greece during the 2000 growing season (Wilson 2002d, SCI 051/013403, 490 DMT).

Olives with incurred residues were obtained after plants were treated with four foliar sprays of a 400 g/L EC dimethoate formulation in May, July, August and November, with a pre-harvest interval of 21 days. Applications were made at nominal rates of 150 mL/ha (plot 2) and 750 mL/ha (plot 3). The amount of dimethoate applied to treated plots 2 and 3 was equivalent to 747–756 g a.i/.ha and 3735–3766 g a.i/.ha respectively. The nominal application volume for both plots was 1200 L/ha. Samples were taken at 0 and 21 days, and for plot 2 only, also at 14, 29, 35 and 42 days.

Samples of olive flesh were placed into freezers on the day of collection after the separation of flesh from the stone.

Samples for processing were taken at 21 DALA. The olives were processed into raw olive oil, refined olive oil and canned olives (0 days, 10 days and 6 months storage). Both non-sterilised and sterilised canned olives were sampled after each storage period. Samples of all the intermediate products were also taken for analysis, including olives (prior to processing), wash water (after washing), olives (after washing), cake (after centrifugation), margines/water (after centrifugation), soap (after filtration) and the brine from the canned olives.

Olives were washed with 1 L per kg of fruits and spin-dried. Washed olives and wash water specimens were taken.

Washed olives were milled using a hammer-type mill. The resulting olive pulp was placed inside a thermo-malaxer with warm water (50 °C), mixed for 20 minutes, boiling water was then added and mixed for further 10 minutes. The pulp was centrifuged and boiling water was added to rinse the jar. The liquid mix (margines, water and oil) was separated from the cake. The floating oil was collected from the remaining liquid (margines and water) and filtered to obtain raw oil. A

solution of soda (115 g/L, 2 mL per 100 mL oil) was added to the raw oil, heated to 60–70 °C for 30 minutes and cooled at ambient temperatures for 1 hour. The refined oil was separated from the waste (soap) and filtered.

A brine (110 g salt/1 L water) was prepared. The olives and brine (2 L brine per 1 kg olives) were mixed in a glass demijohn. The preparation was mixed each day during the first month, then once a week during the 5 other months. The preparation was stored at 15–25 °C for 3 months and at 5–10 °C until sampling at 6 months. During maturation, the density of the brine was checked with a densimeter and salt was added if needed to obtain a density of 1077.

For sampling at day 0, the canned olives were prepared directly in the glass jars. After 10 days of steeping, olives were removed and placed with brine in glass jars. The proportions of canned olives were two thirds of olives and one third of brine. After closing with a protective lid, half of the jars were sterilised at 115–120 °C for 10 minutes, labelled and frozen and the other half were not sterilised, just labelled and frozen. After 6 months of steeping, for each specimen, two canned olive samples were prepared with the following proportions: 400 g of 450 g of olives and 250 g of brine. One of these samples was sterilised and the other was not sterilised.

Residues of dimethoate and omethoate in olive RAC and processed commodities were quantitated by LC-MS/MS using a method developed and validated in study number SCI/026. Acceptable concurrent recovery data for olive commodities were obtained for both analytes. Samples were analysed within 8 months of collection.

Table 158 Residues in olive processed fractions from the foliar application of dimethoate to olives in Greece and Spain (Wilson 2002, SCI 051/013403, 490 DMT)

Trial No., Location, Year (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
SCI/051-01 – plot 2 Karies, Argolida, Hesse, Greece, 2000 (Ladolia) 4 applications 67, 24 and 104 day RTI Growth stage of last application-80 756, 749, 747, 749 g ai/ha 1212, 1201, 1198, 1201 L/ha	Olives	0	3.66, 5.08	0.10, 0.08 (c 0.03)	-	-
		14	1.13, 0.98	0.50,0.46	-	-
		21	0.49, 0.50	0.52, 0.56	-	-
		28	0.11, 0.11	0.37, 0.40	-	-
		35	0.15, 0.11	0.40, 0.40	-	-
		42	0.14, 0.11	0.48, 0.39	-	-
	RAC prior to processing	21	0.39	0.37	-	-
	RAC after washing		0.53	0.44	1.4	1.2
	Wash water		<LOQ	ND	< 0.03	0
	Cake		0.35	0.26	0.90	0.70
	Margines/water		0.33	0.24	0.85	0.65
	Soap		ND	ND	0	0
	Raw olive oil		0.17	ND	0.44	0
	Refined olive oil		<LOQ	ND	< 0.03	0
	0 day canned olives (sterilised)		0.12	0.07	0.31	0.19
	0 day canned brine (sterilised)		0.08	0.03	0.21	0.08
	0 day canned olives (non-sterilised)		0.36	0.33	0.92	0.89
	0 day canned brine (non-sterilised)		0.03	0.03	0.08	0.08
	10 day canned olives (sterilised)		0.03	0.01	0.08	0.03
	10 day canned brine (sterilised)		0.02	<LOQ	0.05	< 0.03

Trial No., Location, Year (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
	10 day canned olives (non-sterilised)		0.29	0.27	0.74	0.73
	10 day canned brine (non-sterilised)		0.08	0.05	0.21	0.14
	6 month canned olives (sterilised)		0.02	ND	0.05	0
	6 month canned brine (sterilised)		0.01	ND	0.03	0
	6 month canned olives (non-sterilised)		0.11	0.02	0.28	0.05
	6 month canned brine (non-sterilised)		0.08	0.03	0.21	0.08
SCI/051-01 – plot 3 Karies, Argolida, Hesse, Greece, 2000 (Ladolia) 4 applications 67, 24 and 104 day RTI Growth stage of last application-80 3766, 3757, 3735, 3744 g ai/ha 1208, 1205, 1198, 1201 L/ha	Olives	0	22.03, 24.34	0.16, 0.15 (c 0.03)	-	-
		21	4.56, 5.02	1.44, 1.51	-	-
	RAC prior to processing	21	3.01	0.92	-	-
	RAC after washing		4.74	1.23	1.6	1.3
	Wash water		0.06	<LOQ	0.02	< 0.01
	Cake		2.75	0.64	0.91	0.70
	Margines/water		2.32	0.59	0.77	0.64
	Soap		ND	ND	0	0
	Raw olive oil		1.26	<LOQ	0.42	< 0.01
	Refined olive oil		0.02	ND	0.007	0
	0 day canned olives (sterilised)		0.34	0.04	0.11	0.04
	0 day canned brine (sterilised)		0.23	0.02	0.076	0.02
	0 day canned olives (non-sterilised)		4.02	0.98	1.3	1.1
	0 day canned brine (non-sterilised)		0.19	0.07	0.063	0.08
	10 day canned olives (sterilised)		0.08	0.02	0.03	0.02
	10 day canned brine (sterilised)		0.05	0.01	0.02	0.01
	10 day canned olives (non-sterilised)		3.22	0.77	1.1	0.84
	10 day canned brine (non-sterilised)		0.46	0.11	0.15	0.12
	6 month canned olives (sterilised)		0.20	0.02	0.07	0.02
	6 month canned brine (sterilised)		0.10	ND	0.03	0
	6 month canned olives (non-sterilised)		0.92	0.06	0.31	0.07
	6 month canned brine (non-sterilised)		0.62	0.07	0.21	0.08

DALA = Days After Last Application

LOQ of dimethoate and omethoate = 0.01 mg/kg for all processed commodities

ND = Not Detected (< 0.002 mg/kg)

In a similar study, the effect of processing (laboratory scale) on residues of dimethoate in olives (Table 147) was investigated in two trials conducted in Greece and one in Spain during the 2001 growing season (Wilson 2003b, SCI 072/024286, 574 DMT).

Olives with incurred residues were obtained after plants were treated with four foliar sprays of a nominal 400 g/L EC dimethoate formulation in May, July, August and with the last application at a pre-harvest interval of 21 ± 1 days. Applications were made at a nominal concentration of 750 mL product/100L. The nominal application volume was 700 or 1200 L/ha depending on plant spacing. Actual application rates were 2181–2187 g a.i./ha for plant spacing above 9 m × 9 m and 3738–3828 g a.i./ha for plant spacing below 9 m × 9 m. RAC samples were taken at 0 and 20 or 21 days after the last application (DALA).

Samples of olive flesh were placed into freezers on the day of collection after the separation of flesh from the stone. Processing was carried out similarly to that described in the previous study (Wilson 2002d, SCI 051/013403, 490 DMT).

Residues of dimethoate and omethoate in olive RAC and processed commodities were quantitated by a method previously developed and validated by Huntingdon Life Sciences in study number SCI/026 (Report number SCI 026/004068). Acceptable concurrent recovery data for olive commodities were obtained for each analyte. Samples were analysed within 11 months of collection.

Table 159 Residues in olive processed fractions from the foliar application of dimethoate to olives in Greece and Spain (Wilson 2003, SCI 072/024286, 574 DMT)

Trial No., Location, Year, (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
SCI/072-01 Karia, Greece, 2001 (Megaritikes) 4 applications 54, 44 and 62 day RTI Growth stage of last application-86 2187, 2186, 2186, 2181 g ai/ha 702, 701, 701, 700 L/ha	RAC olives	0	19.34 (c 0.14)	0.24 (c 0.08)	-	-
	RAC olives	21	5.49 (c 0.08)	1.37 (c 0.07)	-	-
	RAC prior to processing	21	4.82 (c 0.03)	1.27 (c 0.02)	-	-
	Raw olive oil		1.55 (c 0.03)	ND	0.32	0
	Refined oil		1.27 (c 0.01)	0.03	0.26	0.02
	6 month canned olives (sterilised)		0.60	0.03	0.12	0.02
	6 month canned olives (non-sterilised)		1.33 (c 0.01)	0.10	0.28	0.08
	6 month canned brine (sterilised)		0.29	0.04	0.06	0.03
	6 month canned brine (non-sterilised)		0.58 (c 0.01)	0.09	0.14	0.07
SCI/072-02 Koutsopodio, Greece, 2001-02 (Koroneikes) 4 applications 55, 44 and 140 day RTI Growth stage of last application-88 3755, 3750, 3751, 3743 g ai/ha 1204, 1203, 1203, 1200	RAC olives	0	20.98 (c 0.01)	0.02	-	-
	RAC olives	20	13.50	0.46	-	-
	RAC prior to processing	20	9.80	0.47	-	-
	Raw olive oil		3.22	ND	0.33	0
	Refined oil		2.81	ND	0.29	0
	6 month canned olives (sterilised)		0.81	ND	0.08	0
	6 month canned olives (non-sterilised)		3.67	0.07	0.37	0.15
	6 month canned brine		0.39	ND	0.040	0

Trial No., Location, Year, (Variety) L/ha	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
	(sterilised)					
	6 month canned brine (non-sterilised)		2.26	0.04	0.23	0.09
SCI/072-03 Pedralba, Spain, 2001 (Villalonga) 4 applications 44, 35 and 77 day RTI Growth stage of last application-85 3760, 3738, 3828, 3808 g ai/ha 1206, 1199, 1228, 1221 L/ha	RAC olives	0	12.34	0.14	-	-
	RAC olives	20	8.95	1.63	-	-
	RAC prior to processing	20	10.72	1.45	-	-
	Raw olive oil		2.72	ND	0.25	0
	Refined oil		2.68	ND	0.25	0
	6 month canned olives (sterilised)		0.70	0.01	0.065	0.007
	6 month canned olives (non-sterilised)		2.61	0.21	0.24	0.14
	6 month canned brine (sterilised)		0.23	0.01	0.02	0.008
	6 month canned brine (non-sterilised)		1.39	0.12	0.13	0.08

DALA = Days After Last Application

LOQ of dimethoate and omethoate = 0.01 mg/kg for all processed commodities

ND = Not Detected (< 0.002 mg/kg)

The effect of processing (laboratory scale) on residues of dimethoate in olives (Table 148) was also investigated in two trials conducted in Italy and Spain during the 2013 growing season (Eversfield 2014, S13-04249, 1608 DMT).

At each trial two applications of a 400 g/L EC dimethoate formulation were made, using a foliar broadcast sprayer. The applications were made at a nominal rate of 1.44 kg a.i./ha and water volume of 1200 L/ha. Applications were made with a 10–11 day retreatment interval with the final application being made 27–28 days before commercial harvest. Mature olive fruit were transported at ambient temperature to the processing facility where they were processed into olive oil and table olives.

Oil

Healthy fruits were washed using tap water at ambient temperature (ratio of 1:1) and strained (washed olives and washing water). Olives were crushed with the help of a mill to give a homogeneous paste. The paste was then stirred slowly and warmed to 35 °C in a waterbath. Water was added and the paste was centrifuged for 2 minutes. The liquid phase was separated from the solid phase (pomace sample taken) and weights recorded. The liquid phase was transferred to a separating funnel where the two phases separated into vegetation water (aqueous phase) and crude oil. Crude oil was filtered with a vacuum pump and a qualitative filter paper (VWR – particle retention 12–15 µm). The pH was recorded and a sample of virgin oil taken. Hot demineralised water was added to the virgin oil (% was recorded). The mixture was continuously stirred and heated to 90 °C. After 30 minutes, a solution of about 0.5% of citric acid was added to the oil (at about 55% and weights were recorded). After another 20 minutes of stirring at 90 °C, the aqueous waste was removed by separation in a separating funnel. A soda solution (concentrations and weights were recorded) was added to the oil (still under continuous stirring, above 95 °C). After 15–20 minutes 10% hot demineralised water was added and stirred for another 5–15 minutes. The mixture was transferred into a separating funnel to separate the aqueous from the oil phase (was mixed to the total aqueous waste). The aqueous wastes from degumming and neutralisation were mixed. The oil was filtered with help of a vacuum pump and filter

12–15µm and a sample taken (oil before desodoration). The oil was heated to 190 °C for 20 minutes and a refined oil sample taken.

Table olives

Healthy olives were covered with a solution of NaOH at 2% in a pot, with a ratio of olives/solution = 1.0. The weights of olives and solution were recorded. During the de-bittering treatment, the olives were stored at ambient temperature for about 8 hours. At the end of the alkaline treatment, the lye solution was drained off and the fruits were covered with tap water at ambient temperature in the operation of washing (ratio olives/water = 1.0). The duration of the washing step was between 15 and 16 hours, at ambient temperature (laboratory room). After washing, the washed debittered olives were drained and covered with a sodium chloride solution at 10%, with a ratio olives/brine = 1.0. A washing water sample was taken. The weights of olives and brine were recorded. The olives were initially stored at ambient temperature, and after one week, they were stored in chilled conditions (target temperature +7°C) in order to prevent the development of moulds at the top of the brine. The table olives were ready after 19 days. A brine sample was taken. The pH was recorded each working day during the brining.

The analytical method AGR/MOA/DIMETHOATE-1 was used to analyse dimethoate and its metabolites omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid and O-desmethyl iso-dimethoate. The analytical method AGR/MOA/DIMETHOATE-2 was used to analyse the dimethoate metabolites O-desmethyl omethoate, desmethyl dimethoate and O-desmethyl N-desmethyl omethoate. The limit of quantification for dimethoate and its metabolites in all olive matrices was set at 0.01 mg/kg. Acceptable concurrent recovery data for olive commodities were obtained for each analyte. Samples were analysed within 5 months of collection.

Table 160 Residues in olive processed fractions from the foliar application of dimethoate to olives in Italy and Spain (Eversfield 2014, S13-04249, 1608 DMT)

Trial No., Location, Year, (Variety)	Sample	DALA	Residues (mg/kg)				Processing Factor			
			Dimethoate	Omethoate	DCA	ODOCA	DD	ODO	Dimethoate	Omethoate
S13-04249-01 El Coronil, Andalucia, Spain 2013 (Manzanilla) 2 applications 11 day RTI Growth stage of last application -87 1410, 1440g ai/ha 1172, 1198 L/ha	Olive, not washed	27	0.02	0.46	0.01	0.06	0.77	0.11	-	-
	Washing water	27	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.50	< 0.02
	Washed olive	27	0.01	0.47	< 0.01	0.06	0.70	0.13	0.50	1.0
	Pomace	27	0.08	0.36	< 0.01	0.04	0.33	0.08	4.0	0.78
	Paste	27	0.05	0.48	0.03	0.06	0.79	0.13	2.5	1.0
	Vegetation water	27	0.04	0.22	< 0.01	0.03	0.30	0.06	2.0	0.48
	Crude oil	27	0.03	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	1.5	< 0.02
	Virgin oil	27	0.03	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	1.5	< 0.02
	Oil before desodorati on	27	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.50	< 0.02
	Aqueous waste	27	0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.50	< 0.02
	Refined oil	27	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.50	< 0.02
	Olive, not	27	< 0.01	0.49	0.01	0.07	0.88	0.13	-	-

Trial No., Location, Year, (Variety)	Sample	DALA	Residues (mg/kg)				Processing Factor			
			Dimethoate	Omethoate	DCA	ODOCA	DD	ODO	Dimethoate	Omethoate
	washed									
	Washing water	27	< 0.01	< 0.01	< 0.01	0.01	0.24	0.08	-	< 0.02
	Lye	27	< 0.01	< 0.01	< 0.01	0.08	< 0.01	< 0.01	-	< 0.02
	Debittered olive	27	< 0.01	0.07	< 0.01	0.04	0.62	0.16	-	< 0.02
	Brine	27	< 0.01	< 0.01	< 0.01	0.03	0.44	0.13	-	< 0.02
	Table olives	27	< 0.01	0.02	< 0.01	0.02	0.28	0.09	-	< 0.02
S13-04249-02 Castel di Ludica, Sicily, Italy 2013 (Nocellare Dell' Etna) 2 applications 10 day RTI Growth stage of last application -81 1340, 1410g ai/ha 1114, 1173 L/ha	Olive, not washed (prior to oil preparation)	28	0.19	0.56	0.15	0.06	0.75	0.12	-	-
	Virgin oil	28	0.10	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.5	< 0.02
	Refined oil	28	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.05	< 0.01
	Olive, not washed (prior to table olive preparation)	28	0.02	0.45	0.02	0.08	1.51	0.13	-	-
	Table olives	28	0.02	0.01	< 0.01	0.01	0.41	0.07	1.0	0.02

DALA = Days After Last Application

LOQ of dimethoate and metabolites = 0.01 mg/kg for olives and all processed commodities

DCA = Dimethoate carboxylic acid

ODOCA = O-desmethyl omethoate carboxylic acid

DD = Desmethyl dimethoate

ODO = O-desmethyl omethoate

Note: No quantifiable residues of O-desmethyl iso-dimethoate or O-desmethyl N-desmethyl omethoate were observed in any samples

In another study the effect of processing (laboratory scale) on residues of dimethoate in olives (Table 149) was investigated in two of eight trials conducted in Southern Europe (Southern France, Greece, Italy, and Spain,) during the 2017 growing season (Gemrot 2018, 17-00467, 2017RES-DMT3277).

At each trial two applications of a 400 g/L EC dimethoate formulation were made at a nominal rate of 480 g a.i./ha and water volume of 1200 L/ha. Applications were made with a 10 day retreatment interval with the final application being made 28 days before commercial harvest.

In the decline curves, RAC specimens (whole fruit) were collected at 0, 7–8, 13–14, 21–22 and 28 DALA (commercial harvest). In the harvest trials, RAC specimens (whole fruit) were collected at the time of commercial harvest, 28 DALA.

In the processing trials, RAC specimens for processing (whole fruit) were collected at the time of commercial harvest, 27–28 DALA. Processing into crude and refined oil and canned olives was carried out in a similar way to that described in the previous study (Eversfield 2014, S13-04249, 1608 DMT).

Dimethoate and its metabolites omethoate and dimethoate carboxylic acid were analysed using methods described in the report and amendment of study S12-04027, except for crude and refined oil for which the method was modified and validated during this study.

Desmethyl dimethoate, O-desmethyl omethoate, O-desmethyl iso-dimethoate, O-desmethyl omethoate carboxylic acid and O-desmethyl N-desmethyl omethoate were analysed using methods described in the report and related amendment of studies S12-04027 and S13-04322. The limit of quantification for dimethoate and its metabolites in all olive matrices was set at 0.01 mg/kg. Samples were analysed within 5 months of collection.

Table 161 Residues in olive processed fractions from the foliar application of dimethoate to olives in France and Spain (Gemrot 2018, 17-00467, 2017RES-DMT3277)

Trial No., Location, Year, (Variety)	Sample	DALA	Residues (mg/kg)					Processing Factor	
			Dimethoate	Omethoate	ODOCA	DD	ODO	Dimethoate	Omethoate
17-00467-07 Saint Sauveur de Cruzieres, Auvergne Rhone Alpes, France 2017 (Picholine) 2 applications 10 day RTI Growth stage of last application-37 486, 484g ai/ha 1214, 1211 L/ha	Olives, prior to processing	27	< 0.01	0.21	0.02	0.11	0.03	-	-
	Crude oil	27	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	-	< 0.05
	Refined oil	27	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	-	< 0.05
	Canned olives	27	< 0.01	< 0.01	0.02	0.12	0.20	-	< 0.05
17-00467-08 Zafarraya, Granada/Andalucia, Spain 2017 (Picual) 2 applications 10 day RTI Growth stage of last application-38 495, 478 g ai/ha 1238, 1195 L/ha	Olives, prior to processing	28	0.08	0.24	0.04	0.07	0.03	-	-
	Crude oil	28	0.02	< 0.01	< 0.01	< 0.01	< 0.01	0.25	< 0.04
	Refined oil	28	0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.125	< 0.04
	Canned olives	28	0.01	< 0.01	0.03	0.09	0.15	0.125	< 0.04

DALA = Days After Last Application

LOQ of dimethoate and metabolites = 0.01 mg/kg for olives and all processed commodities

ND = Not Detectable (< 0.002 mg/kg)

NC = Not Calculable

ODOCA = O-desmethyl omethoate carboxylic acid

DD = Desmethyl dimethoate

ODO = O-desmethyl omethoate

Note: No quantifiable residues of dimethoate carboxylic acid, O-desmethyl iso-dimethoate or O-desmethyl N-desmethyl omethoate were observed in any samples

Wheat

The effect of processing (laboratory scale) on residues of dimethoate in wheat (Table 151) was investigated in four trials conducted in Germany and the United Kingdom in the 2001 growing season (Wilson 2003c, SCI 079/014476, 576 DMT). Wheat with incurred residues was obtained after plants were sprayed twice with a 400 g/L EC dimethoate formulation. The first application was made at growth stage BBCH 73/75 and the second 7-11 days later. Each trial had two treated plots. Plot 2 was treated at 1 fold (first application) and 5 fold (second application) the recommended application rate. Plot 3 was treated at 5 fold for both applications. Processed fractions from Plot 3 were analysed for the processing part of the study.

Wheat whole plant RAC samples were taken 0 days after the last application (DALA), and then straw and grain RAC samples and grain processing samples were taken at earliest commercial harvest at 16–32 DALA.

In the balance trial (SCI/079-03), wheat grain samples were processed according to simulated commercial procedures into cleaned grain, screenings, wholemeal flour, middlings, white flour, cleaned bran, germ rich fraction, toppings, type 550 flour and wholemeal bread. For the follow-up trials (SCI/079-01, SCI/079-02 and SCI/079-04), the grain was processed into wholemeal flour, white flour, cleaned bran, germ rich fraction, toppings, type 550 flour and wholemeal bread.

Wholemeal

Wheat was fed to a mill. The six flour fractions (three break and three reductions), bran and offal were collected, weighed and recorded. The bran and offal was passed through the bran finisher twice. The weights of the cleaned bran, cleaned offal, bran flour and offal flour were recorded. All 10 fractions were blended together for approximately 20 minutes.

Approximately 3 kg of wheat was fed into a mill to produce approximately 2 kg of middlings. The feed to the first reduction section of the mill was disconnected in order to collect the middlings fraction. The break roll was engaged and the reduction rolls left apart prior to starting the mill.

Approximately 15 kg of conditioned wheat was used to produce white flour, cleaned coarse bran, the germ rich fraction and toppings (Bran Finisher flours from bran and offal) and Type 550 flour.

The uncleaned bran was weighed, recorded and passed through the Bran Finisher twice. The cleaned offal was designated as the "germ rich" fraction.

The bran flour and offal flour were blended together to produce toppings.

The ash content (on a dry matter) of the white flour and the toppings were determined. Based on the results, suitable quantities of white flour and toppings were blended together to produce Type 550 flour with an ash content of between 0.51–0.63% (dry matter basis).

Residues of dimethoate and omethoate in wheat RAC and processed commodities were quantitated by LC/MS/MS using a method described in study number SCI/050. Acceptable concurrent recovery data for wheat commodities were obtained for each analyte. Samples were analysed within 6 months of collection.

Table 162 Residues in wheat processed fractions from the foliar application of dimethoate to wheat in Germany and the United Kingdom (Wilson 2003c, SCI 079/014476, 576 DMT)

Trial No., Location, Year (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
SCI/079-01, Manningtree, United Kingdom, 2001 (Claire) 2 applications (11 day RTI) Growth stage at last application - BBCH 77/83 1903 and 1841 g ai/ha 215 and 208 L/ha	Green plant	0	36.40	0.17	-	-
	Straw (RAC)	16	2.55	0.05	-	-
	Grain (RAC)	16	0.022	0.005	-	-
	Grain BP	16	0.013	0.002	-	-
	Wholemeal flour		0.008	< 0.001	0.62	< 0.5
	White flour		0.004	< 0.001	0.31	< 0.5
	Cleaned bran		0.052	0.007	4.00	3.50
	Germ rich		0.037 (*c 0.002*)	0.004	2.85	2.00
	Toppings		0.015	0.001	1.15	0.50
	Type 550 flour		0.003	< 0.001	0.23	< 0.5
	Wholemeal bread		0.023	0.003	1.77	1.50
SCI/079-02, Tendring, United Kingdom, 2001 (Hereward) 2 applications (7 day RTI) Growth stage at last application - BBCH 77/83 1783 and 1854 g ai/ha 202 and 211 L/ha	Green plant	0	31.15	0.28	-	-
	Straw (RAC)	32	1.31	0.02	-	-
	Grain (RAC)	32	0.034	0.007	-	-
	Grain BP	32	0.018	0.003	-	-
	Wholemeal flour		0.004	< 0.001	0.22	< 0.3
	White flour		0.001	< 0.001	0.06	< 0.3
	Cleaned bran		0.025	0.004	1.39	1.33
	Germ rich		0.015	0.002	0.83	0.67
	Toppings		0.007	< 0.001	0.39	< 0.3
	Type 550 flour		< 0.001	< 0.001	< 0.06	< 0.3
	Wholemeal bread		0.013	0.001	0.72	0.33
SCI/079-03, Brunne, Germany, 2001 (Aristos) 2 applications (7 day RTI) Growth stage at last application - BBCH 77 1775 and 1797 g ai/ha 201 and 203 L/ha	Green plant	0	49.48	0.28	-	-
	Straw (RAC)	22	2.09	0.04	-	-
	Grain (RAC)	22	0.157	0.017	-	-
	Grain BP	22	0.005	< 0.001	-	-
	Grain AC		0.004	ND	0.8	-
	Screenings		0.070	0.003	14	-
	Wholemeal flour		0.016	< 0.001	3.2	-
	Middlings		0.015	< 0.001	3	-
	White flour		0.004	< 0.001	0.8	-
	Cleaned bran		0.076	0.005	15	-
	Germ rich		0.048	0.003	9.6	-
	Toppings		0.024	< 0.001	4.8	-
	Type 550 flour		0.003	< 0.001	0.6	-
	Wholemeal bread		0.027	0.002	5.4	-

Trial No., Location, Year (Variety)	Sample	DALA	Residues (mg/kg)		Processing Factor	
			Dimethoate	Omethoate	Dimethoate	Omethoate
SCI/079-04,	Green plant	0	40.60	0.26	-	-
Blumberg,	Straw (RAC)	18	1.08	0.02	-	-
Germany, 2001	Grain (RAC)	18	0.080	0.003	-	-
(Pegassos)	Grain BP	18	0.075	0.002	-	-
2 applications (8 day RTI)	Wholemeal flour		0.051	0.002	0.7	1.0
Growth stage at last application	White flour		0.010	< 0.001	0.1	< 0.5
- BBCH 77	Cleaned bran		0.363	0.010	4.8	5.0
1783 and 1758 g ai/ha	Germ rich		0.225	0.008	3.0	4.0
202 and 199 L/ha	Toppings		0.094	0.002	1.3	1.0
	Type 550 flour		0.009	< 0.001	0.1	< 0.5
	Wholemeal bread		0.145	0.005	1.9	2.5

DALA = Days After Last Application

LOQ of dimethoate and omethoate = 0.01 mg/kg for wheat green plant and straw

LOQ of dimethoate and omethoate = 0.001 mg/kg for wheat grain, wholemeal flour, bran, wheat germ and bread

BP = Before Processing (Uncleaned grain)

AC = After Cleaning

In another study, eight residues trials were conducted on wheat at four sites in Southern France and four sites in Spain in 2014 (Tandy 2015, S14-01362, 1712 DMT). Two of these (S14-01362-01 and S14-01362-02) were also processing trials. For trials S14-01362-01 and S14-01362-02, one application of a 400 g/L EC formulation of dimethoate was made at 200 g a.i./ha to plot 2 and also at 1000 g a.i./ha to plot 3, the latter for the purpose of processing. Due to an error in sampling plot 3 of trial S14-01362-02, data regarding untreated and processed specimens was not reported. In the other six trials one application was made at 200 g a.i./ha.

Processing specimens from trial S14-01362-01 were processed into refined flour (type 550), wholemeal flour, white bread and wholemeal bread.

The wheat grain was cleaned. Cleaning was done in three steps. First, the wheat was de-awned. In the second step impurities were removed with the use of sieves. In the final cleaning step, the grain was sorted by size grading (2.5 mm) to remove shrivelled or under-sized grain. The cleaned grain was sampled.

Conditioning was performed on a portion of cleaned grain that was moistened at room temperature. The grain after conditioning was sampled.

A part of the wheat sample was milled to clean the mill and to get normal milling conditions at the correct working temperature of the mill. This material was discarded. The mill delivered eight different fractions. Two of the fractions were the bran fractions (coarse bran and fine bran), two of them were the shorts fractions and the remaining four were the 'flour fractions without shorts'. Both shorts fractions were mixed and the shorts sample taken. The flour fractions flour without shorts and the remaining shorts were mixed together. Parts of fine bran and coarse bran were mixed (ratio 1:1) and the total bran was sampled.

The other parts of both bran fractions were used to get the toppings and middlings. Coarse bran was cleaned with a bran duster to get toppings 1. The fine bran was sieved to get toppings 2 and middlings. The middlings were sampled and toppings 1 and toppings 2 were mixed to get a toppings sample.

The toppings were mixed into the flour fraction to get refined flour (type 550) which was sampled.

For wholemeal flour, a hammer mill was used.

For preparation of white or wholemeal bread, all ingredients (white or wholemeal flour, salt, bread yeast, water) were placed in a kneading machine and mixed for 10 minutes. For fermentation, the dough was placed with moistened towels over the bowl or forms in environmental cabinets with a controlled climate for a total of 65 mins. The time was divided into three fermentation steps. After 25 min (first fermentation period) the dough was kneaded for 1 minute and transferred back again into the cabinet for a second fermentation period of 15 minutes. After the second fermentation the dough was manually divided into 4 loaves of comparable weights. These loaves were transferred into the baking forms. Four forms were placed in the cabinet again followed by a rest of 25 minutes (third fermentation). The forms were placed in the preheated oven with steam injection and baked at about 210 °C for 25 minutes. The bread (white bread or wholemeal bread) was sampled after cooling down.

The analytical method AGR/MOA/DIMETHOATE-1 was used to analyse dimethoate and its metabolites omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid and O-desmethyl iso-dimethoate. The analytical method AGR/MOA/DIMETHOATE-2 was used to analyse the dimethoate metabolites O-desmethyl omethoate, desmethyl dimethoate and O-desmethyl N-desmethyl omethoate. These methods were confirmed during the course of the study for dimethoate and its metabolites omethoate, dimethoate carboxylic acid, O-desmethyl omethoate carboxylic acid, O-desmethyl iso-dimethoate, O-desmethyl omethoate, desmethyl dimethoate and O-desmethyl N-desmethyl omethoate in coarse/total bran, refined flour (type 550), white bread and whole bread matrices with one exception. O-desmethyl omethoate carboxylic acid was not confirmed in refined flour (type 550). No analytical method was developed for the analysis of O-desmethyl omethoate carboxylic acid in refined flour (type 550), shorts and wholemeal flour.

The limit of quantification for dimethoate and its 7 metabolites in all wheat and its processed fractions was set at 0.01 mg/kg. Samples were analysed within 9 months of collection.

Table 163 Residues in wheat processed fractions from the foliar application of dimethoate to wheat in France (Tandy 2015, S14-01362, 1712 DMT)

Trial No., Location, Year, Variety	Sample	DAA	Residues (mg/kg)				Processing Factor	
			Dimethoate	Omethoate	ODOCA	DD		
S14-01362-01, Treatment 3 Meauzac, Tarn et Garonne, France, 2014 (Winter wheat-Soleilho) 1 application Growth stage at Application BBCH 69-71 1001 g ai/ha 200 L/ha	RAC grain, not cleaned	39	< 0.003	< 0.003	< 0.003	0.01	-	-
	Cleaned grain		< 0.003	< 0.003	< 0.003	0.01	-	-
	Grain after conditioning (before white flour)		< 0.003	< 0.003	< 0.003	0.01	-	-
	Coarse bran		< 0.01	< 0.003	< 0.003	0.02	-	-
	Fine bran		< 0.003	< 0.003	< 0.003	< 0.01	-	-
	Total bran		< 0.003	< 0.003	< 0.003	0.02	-	-
	Toppings		0.01	< 0.003	< 0.003	0.01	-	-
	Middlings		< 0.01	< 0.003	< 0.003	< 0.01	-	-
	Shorts		< 0.003	< 0.003	NA	< 0.01	-	-
	Refined flour (Type 550)		< 0.003	< 0.003	NA	< 0.003	-	-
	Dough (refined flour)		< 0.003	< 0.003	< 0.003	< 0.003	-	-
	White bread		< 0.003	< 0.003	< 0.003	< 0.003	-	-
	Grain after conditioning (before wholemeal flour)		< 0.003	< 0.003	< 0.003	< 0.01	-	-
	Wholemeal flour		< 0.003	< 0.003	NA	< 0.003	-	-

Trial No., Location, Year, Variety	Sample	DAA	Residues (mg/kg)				Processing Factor	
			Dimethoate	Omethoate	ODOCA	DD		
	Dough (wholemeal flour)		< 0.003	< 0.003	< 0.003	< 0.01	-	-
	Wholemeal bread		< 0.003	< 0.003	< 0.003	< 0.01	-	-

LOQ of dimethoate and metabolites = 0.01 mg/kg for grain and all processed commodities

LOD of dimethoate and metabolites = 0.003 mg/kg for grain and all processed commodities

ODOCA = O-desmethyl omethoate carboxylic acid

DD = Desmethyl dimethoate

Note: Residues of dimethoate carboxylic acid, O-desmethyl iso-dimethoate, O-desmethyl omethoate and O-desmethyl N-desmethyl omethoate were < 0.003 mg/kg in all samples

As residues observed in the RAC were <LOD it is not possible to calculate processing factors for the various processing fractions.

Residues of dimethoate and omethoate in animal commodities

Lactating cattle feeding study

A feeding study was conducted for lactating Holstein dairy cattle (Arndt – 2012b). 17 animals were selected for the study and randomly assigned to one of five groups – two untreated control animals, three each in the 1, 3.4, and 10.1 ppm dose groups, and six in the 33.2 ppm group. Treated animals were dosed orally daily for 28 days with dimethoate in gelatine capsules. Control animals received empty capsules. Cattle were milked twice daily through the acclimatisation period and treatment period, and the milk proportionately pooled to yield one composite milk sample per animal per day. Additional milk was collected on treatment days 13 and 28 for separation into skim milk and cream.

On day 28, all except one control animal and three of the highest dose group cattle were slaughtered (7.5-12 hours after the final dose) and samples of round, flank and loin muscle, liver, kidney, and omental, subcutaneous and perirenal fat were collected. The four animals not sacrificed on day 28 were continued without dosing in the depuration phase of the study, and further milk samples were collected on days 31, 35 and 42. On each of days 31, 35, and 42, one of the treated depuration animals was sacrificed, with the final control animal sacrificed on day 42. Samples of muscle, fat, liver and kidney were collected as before. Milk and tissues samples were frozen within a few hours of collection.

Feed and water consumption and milk production were monitored during the study, and tissues were analysed for gross abnormalities at sacrifice. No adverse effects on milk production, feed or water consumption, or body weight were noted.

Samples were analysed for dimethoate and omethoate using a QuEChERS based method (PTRL study no. 2080W), involving extraction with acetonitrile, with the addition of water in the case of fat, with cleanup by partitioning (by addition of salts to create an organic/aqueous partition) and dispersive solid phase extraction, followed by LC-MS/MS analysis (LOQ = 0.001 mg/kg). Good concurrent recoveries were achieved for milk and tissues. Milk samples were analysed within 35 days of collection. The maximum intervals between sampling and analysis were 260, 9, 6, and 19 days for fat, liver, kidney, and muscle samples respectively.

No residues of dimethoate were found in milk (either control or any of the treated samples) at levels above the LOQ. Low levels of omethoate (maximum 0.019 mg/kg) were found, only for some milk samples from the highest dose group. Omethoate residues in skim milk and cream were very similar, indicating no significant preferential partitioning. In muscle, again, no residues of dimethoate were found above the LOQ, while some low level residues of omethoate (up to 0.0051 mg/kg) were found only for the highest dose group. In liver, no residues were found above the LOQ for dimethoate, with low level detections of omethoate in the 10.1 and 33.2 ppm groups. For kidney, there was a single low level detection of omethoate for the highest dose group, and no detections of

dimethoate. For fat, there were some low level residues of both dimethoate and omethoate. The depuration data showed that clearance was rapid, with no detections above the LOQ.

Table 164 Residues of dimethoate and omethoate in whole milk (Arndt – 2012b)

Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
		230 → 199	230 → 125	214 → 183	214 → 155
1 (control)	All	All residues < 0.001			
2 (1 ppm)	All	All residues < 0.001			
3 (3.4 ppm)	All	All residues < 0.001			
4 (10.1 ppm)	All	All residues < 0.001			
5 (33.2 ppm)	-1	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	1	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	2	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	3	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	5	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	7	< 0.001 (6)	< 0.001 (6)	< 0.001 (4), 0.0010, 0.0012 (< 0.001)	< 0.001 (4), 0.0010, 0.0013 (< 0.001)
	10	< 0.001 (6)	< 0.001 (6)	0.0040, 0.0012, 0.0029, 0.0040, < 0.001 (2) (0.0022)	0.0039, 0.0012, 0.0029, 0.0400, < 0.001 (2) (0.0022)
	13	< 0.001 (6)	< 0.001 (6)	0.0064, 0.0036, 0.0014, 0.0046, 0.0023, 0.0050 (0.0039)	0.0063, 0.0036, 0.0013, 0.0048, 0.0024, 0.0050 (0.0039)
	16	< 0.001 (6)	< 0.001 (6)	0.0096, 0.0066, 0.0018, 0.0054, 0.0064, 0.0056 (0.0059)	0.0096, 0.0065, 0.0019, 0.0055, 0.0070, 0.0058 (0.0061)
	19	< 0.001 (6)	< 0.001 (6)	0.0111, 0.0062, 0.0022, 0.0056, 0.0056, 0.0050 (0.0060)	0.0110, 0.0061, 0.0021, 0.0055, 0.055, 0.0049 (0.0059)
	22	< 0.001 (6)	< 0.001 (6)	0.0087, 0.0065, 0.0023, 0.0075, 0.0049, 0.0055 (0.0059)	0.0086, 0.0066, 0.0023, 0.0074, 0.0049, 0.0056 (0.0059)
	25	< 0.001 (6)	< 0.001 (6)	0.0086, 0.0061, 0.0016, 0.0067, 0.0046, 0.0065 (0.0057)	0.0086, 0.0062, 0.0016, 0.0068, 0.0047, 0.0064 (0.0057)
	28	< 0.001 (6)	< 0.001 (6)	0.0187, 0.00147, 0.0054, 0.0124, 0.0129, 0.0135 (0.0129)	0.0189, 0.0146, 0.0054, 0.0126, 0.0132, 0.0136 (0.0131)
	31 (+3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)
	35 (+7)	< 0.001 (2)	< 0.001 (2)	< 0.001 (2)	< 0.001 (2)
	42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001

Table 165 Residues of dimethoate and omethoate in skim milk and cream (Arndt – 2012b)

Matrix	Dose group	Study day	Dimethoate residues (mg/kg)	Omethoate residues (mg/kg)

			230 → 199	230 → 125	214 → 183	214 → 155
Skim milk	1 (control)	All	All residues < 0.001			
	2 (1 ppm)	All	All residues < 0.001			
	3 (3.4 ppm)	All	All residues < 0.001			
	5 (33.2 ppm)	13	< 0.001 (3)	< 0.001 (3)	0.0062, 0.0031, 0.0012 (0.0035)	0.0063, 0.0031, 0.0013 (0.0036)
		28	< 0.001 (3)	< 0.001 (3)	0.0155, 0.013, 0.0061 (0.0117)	0.0152, 0.0135, 0.0061 (0.0116)
Cream	1 (control)	All	All residues < 0.001			
	2 (1 ppm)	All	All residues < 0.001			
	3 (3.4 ppm)	All	All residues < 0.001			
	5 (33.2 ppm)	13	< 0.001 (3)	< 0.001 (3)	0.0049, 0.0026, 0.0011 (0.0023)	0.0049, 0.0026, < 0.001 (0.0027)
		28	< 0.001 (3)	< 0.001 (3)	0.0152, 0.0129, 0.0051 (0.0111)	0.0152, 0.0128, 0.0051 (0.0110)

Table 166 Residues of dimethoate and omethoate in tissues (Arndt – 2012b)

Matrix	Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
			230 → 199	230 → 125	214 → 183	214 → 155
Loin muscle	1 (control)	28, 42	All residues < 0.001			
	3 (3.4 ppm)	28	All residues < 0.001			
	5 (33.2 ppm)	28	< 0.001 (3)	< 0.001 (3)	0.0049, 0.0021, < 0.001 (0.0025)	0.0048, 0.0021, < 0.001 (0.0025)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Flank muscle	1 (control)	28, 42	All residues < 0.001			
	3 (3.4 ppm)	28	All residues < 0.001			
	5 (33.2 ppm)	28	< 0.001 (3)	< 0.001 (3)	0.0049, 0.0020, < 0.001 (0.0025)	0.0051, 0.0020, < 0.001 (0.0025)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Round muscle	1 (control)	28, 42	All residues < 0.001			
	3 (3.4 ppm)	28	All residues < 0.001			
	5 (33.2 ppm)	28	< 0.001 (3)	< 0.001 (3)	0.0049, 0.0020, < 0.001 (0.0025)	0.0048, 0.0019, < 0.001 (0.0024)

Matrix	Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
			230 → 199	230 → 125	214 → 183	214 → 155
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Liver	1 (control)	28, 42	All residues < 0.001			
	2 (1 ppm)	28	All residues < 0.001			
	3 (3.4 ppm)	28	All residues < 0.001			
	4 (10.1 ppm)	28	< 0.001 (3)	< 0.001 (3)	0.0018, 0.0012, 0.0010 (0.0014)	0.0018, 0.0015, 0.0011 (0.0014)
	5 (33.2 ppm)	28	< 0.001 (3)	< 0.001 (3)	0.0038, 0.0054, 0.0039, (0.0044)	0.0042, 0.0059, 0.0039 (0.0047)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Kidney	1 (control)	28, 42	All residues < 0.001			
	2 (1 ppm)	28	All residues < 0.001			
	3 (3.4 ppm)	28	All residues < 0.001			
	4 (10.1 ppm)	28	All residues < 0.001			
	5 (33.2 ppm)	28	< 0.001 (3)	< 0.001 (3)	0.0047, < 0.001 (2) (0.0019)	0.0047, < 0.001 (2) (0.0019)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Subcutaneous fat	1 (control)	28, 42	All residues < 0.001			
	2 (1 ppm)	28	< 0.001 (2), 0.0025 (0.0012)	< 0.001 (2), 0.0026 (0.0012)	< 0.001 (3)	< 0.001 (3)
	3 (3.4 ppm)	28	0.0018, 0.0019, < 0.001 (0.0014)	0.0019, 0.0019, < 0.001 (0.0014)	< 0.001 (3)	< 0.001 (3)
	4 (10.1 ppm)	28	< 0.001, 0.0098, < 0.001 (0.0036)	< 0.001, 0.0100, < 0.001 (0.0037)	< 0.001 (3)	< 0.001 (3)
	5 (33.2 ppm)	28	< 0.001 (2), 0.0019 (< 0.001)	< 0.001 (2), 0.0019 (< 0.001)	0.004, < 0.001 (2) (0.0017)	0.004, < 0.001 (2) (0.0017)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Perirenal fat	1 (control)	28, 42	All residues < 0.001			
	2 (1 ppm)	28	0.0268,	0.0270,	< 0.001 (3)	< 0.001 (3)

Matrix	Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
			230 → 199	230 → 125	214 → 183	214 → 155
			0.0046, < 0.001 (0.011)	0.0046, < 0.001 (0.011)		
	3 (3.4 ppm)	28	0.0014, < 0.001 (2) (< 0.001)	0.0014, < 0.001 (2) (< 0.001)	< 0.001 (3)	< 0.001 (3)
	4 (10.1 ppm)	28	< 0.001 (2), 0.0027 (0.0012)	< 0.001 (2), 0.0028 (0.0013)	< 0.001 (3)	< 0.001 (3)
	5 (33.2 ppm)	28	0.0044, < 0.001, 0.0019 (0.0023)	0.0046, < 0.001, 0.0020 (0.0024)	0.0022, < 0.001 (2) (0.0011)	0.0021, < 0.001 (2) (0.0010)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001
Omental fat	1 (control)	28, 42	All residues < 0.001			
	2 (1 ppm)	28	0.0023, < 0.001 (2) (0.0011)	0.0023, < 0.001 (2) (0.0011)	< 0.001 (3)	< 0.001 (3)
	3 (3.4 ppm)	28	0.0025, < 0.001 (2) (0.0012)	0.0026, < 0.001 (2) (0.0012)	< 0.001 (3)	< 0.001 (3)
	4 (10.1 ppm)	28	< 0.001, 0.0174, < 0.001 (0.0061)	< 0.001, 0.0176, < 0.001 (0.0062)	< 0.001 (3)	< 0.001 (3)
	5 (33.2 ppm)	28	0.0053, < 0.001, 0.0016 (0.0025)	0.0055, < 0.001, 0.0016 (0.0025)	0.0012, < 0.001 (2) (< 0.001)	0.0013, < 0.001 (2) (< 0.001)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
		42 (+14)	< 0.001	< 0.001	< 0.001	< 0.001

Laying hen feeding study

A feeding study was conducted for laying white Leghorn hens (Arndt – 2012c). Sixty birds were selected for the study and randomly assigned to one of five groups – ten untreated control birds, ten each in the 0.15, 0.40, and 1.2 ppm dose groups, and 20 in the 4.0 ppm group. Treated birds were dosed orally daily for 28 days with dimethoate in gelatine capsules. Control birds received empty capsules. Eggs were collected twice daily through the acclimatisation period and treatment period, and the PM and the following AM eggs being pooled to form a single composite egg sample per bird per day.

On day 28, 46 birds were slaughtered approximately 24 hours after the final dose (6 control birds, all of the 0.15, 0.40, and 1.2 ppm birds, and 10 of the 4.0 ppm birds) and samples of thigh and breast muscle, liver, and subcutaneous and abdominal fat were collected. The birds not sacrificed on day 28 were continued without dosing in the depuration phase of the study, and further egg samples were collected on days 29 and 31. On each of days 31 and 35, three of the 4.0 ppm birds were sacrificed, and then on day 42, the remaining four 4.0 ppm and the four control birds were sacrificed.

Samples of muscle, fat, and liver were collected as before. Egg and tissues sample were frozen within a few hours of collection.

Feed and water consumption and egg production were monitored during the study, and tissues were analysed for gross abnormalities at sacrifice. No adverse effects on egg production, feed or water consumption, or body weight were noted.

Samples were analysed for dimethoate and omethoate using a QuEChERS based method (PTRL study no. 2080W), involving extraction with acetonitrile, with the addition of water in the case of fat, with cleanup by partitioning (by addition of salts to create an organic/aqueous partition) and dispersive solid phase extraction, followed by LC-MS/MS analysis (LOQ = 0.001 mg/kg). Good concurrent recoveries were achieved for eggs and tissues. Egg samples were analysed within 15 days of collection. The maximum intervals between sampling and analysis were 13, 4 and 13 days for fat, liver, and muscle samples respectively.

No residues of dimethoate or omethoate were found above the LOQ in any of the egg or tissue samples.

Table 167 Residues of dimethoate and omethoate in eggs (Arndt – 2012c)

Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
		230 → 199	230 → 125	214 → 183	214 → 155
1 (control)	All	All residues < 0.001			
2 (0.15 ppm)	All	All residues < 0.001			
3 (0.40 ppm)	All	All residues < 0.001			
4 (1.2 ppm)	All	All residues < 0.001			
5 (4.0 ppm)	-1	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	1	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	2	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	3	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	5	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	7	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	10	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	13	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	16	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	19	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	22	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	28	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)	< 0.001 (6)
	29 (+1)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)
	31 (+3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)

Table 168 Residues of dimethoate and omethoate in tissues (Arndt – 2012c)

Matrix	Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
			230 → 199	230 → 125	214 → 183	214 → 155
Muscle (composite breast and thigh)	1 (control)	28, 42	All residues < 0.001			
	2 (0.15 ppm)	28	All residues < 0.001			

Matrix	Dose group	Study day	Dimethoate residues (mg/kg)		Omethoate residues (mg/kg)	
			230 → 199	230 → 125	214 → 183	214 → 155
	3 (0.40 ppm)	28	All residues < 0.001			
	4 (1.2 ppm)	28	All residues < 0.001			
	5 (4.0 ppm)	28	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
Liver	1 (control)	28, 42	All residues < 0.001			
	2 (0.15 ppm)	28	All residues < 0.001			
	3 (0.40 ppm)	28	All residues < 0.001			
	4 (1.2 ppm)	28	All residues < 0.001			
	5 (4.0 ppm)	28	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001
Fat (composite subcutaneous and abdominal)	1 (control)	28, 42	All residues < 0.001			
	2 (0.15 ppm)	28	All residues < 0.001			
	3 (0.40 ppm)	28	All residues < 0.001			
	4 (1.2 ppm)	28	All residues < 0.001			
	5 (4.0 ppm)	28	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)
		31 (+3)	< 0.001	< 0.001	< 0.001	< 0.001
		35 (+7)	< 0.001	< 0.001	< 0.001	< 0.001

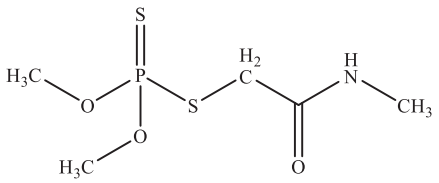
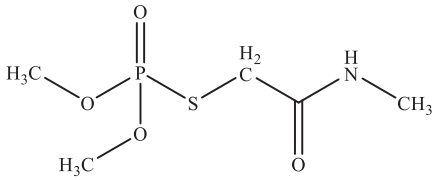
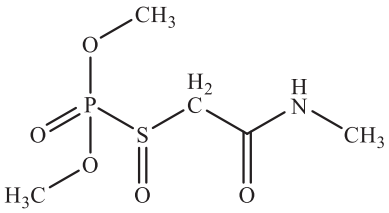
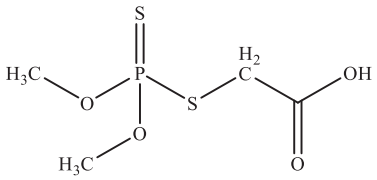
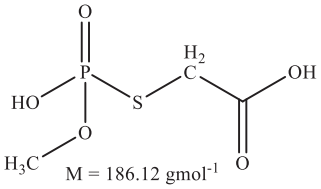
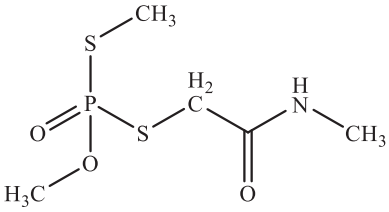
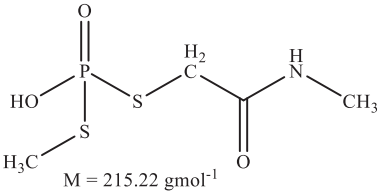
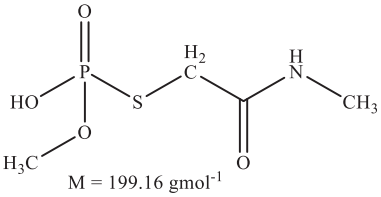
APPRAISAL

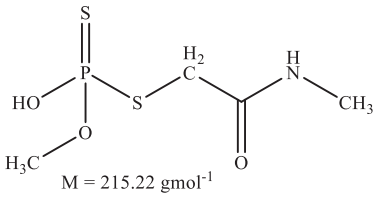
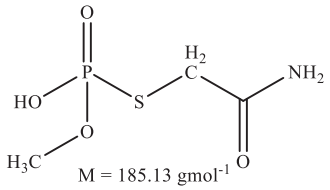
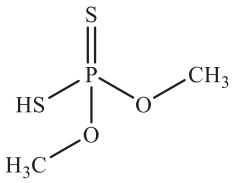
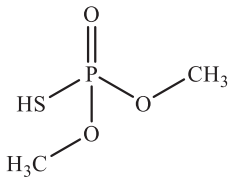
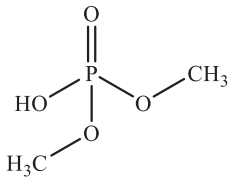
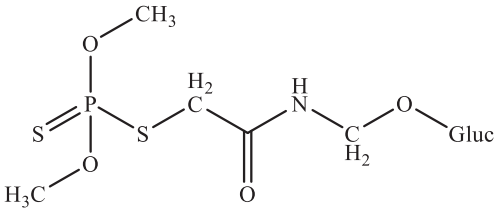
Dimethoate is an organophosphate insecticide which acts through acetylcholinesterase inhibition. It was scheduled by the Fiftieth Session of the CCPR (2018) for periodic review evaluation by the 2019 JMPR. Dimethoate has been evaluated on numerous occasions by the JMPR commencing in 1963. The most recent periodic review was in 1996 (toxicology) and 1998 (residues), with a subsequent evaluation for toxicology and residues in 2003 to establish an acute reference dose and consider additional plant metabolism studies. Residue data for additional uses was evaluated in 2006 and 2008.

The Meeting considered information supplied by the sponsor on identity, physicochemical properties, metabolism and environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing, and animal feeding studies. Additional supervised residue trial data was supplied by Australia for mandarin, oranges, avocados, mangoes, capsicum and pulses, and by Thailand for yard-long bean.

Table 1 Metabolites of dimethoate

Component name	Structure	Origin

Component name	Structure	Origin
Dimethoate		Parent compound
Omethoate		Potato, olives, wheat, rat, goat, hen
Omethoate sulfoxide		Goat (intermediate), hen (intermediate)
Dimethoate carboxylic acid	 M = 216.21 gmol^{-1}	Potato, olives, wheat, rat, goat, hen
O-desmethyl omethoate carboxylic acid	 M = 186.12 gmol^{-1}	Potato, wheat
Isodimethoate		Olives (intermediate), wheat (probable intermediate)
O-desmethyl isodimethoate	 M = 215.22 gmol^{-1}	Potato, olives, wheat
O-desmethyl omethoate	 M = 199.16 gmol^{-1}	Potato, olives, wheat

Component name	Structure	Origin
Desmethyl dimethoate	 M = 215.22 gmol^{-1}	Potato, hydrolysis, soil (minor component)
O-desmethyl N-desmethyl omethoate	 M = 185.13 gmol^{-1}	Potato, olives, wheat
Dimethyl dithiophosphate and conjugates		Potato, wheat, rat, goat, hen
Dimethyl thiophosphate		Hydrolysis
Dimethyl phosphate		Rat
Hydroxy dimethoate glucose conjugate		Potato, wheat

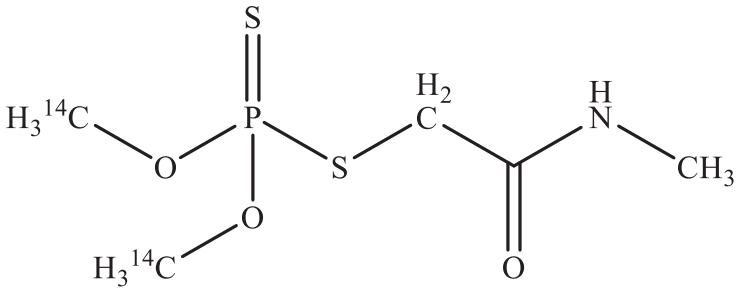
Physicochemical properties

Dimethoate is moderately water soluble (28.2 g/L in purified water at 20 °C), and very soluble in polar organic solvents such as dichloromethane, acetonitrile, and methanol. With an octanol/water partition coefficient (log10POW) of 0.75, dimethoate is not expected to partition into fat. Hydrolysis is slow at acidic pH (t1/2 = 156 days at pH 5), becoming more rapid as the pH is increased (t1/2 = 4.4 days at pH 9).

Omethoate is highly soluble in water (> 500 g/L) and has a very low octanol/water partition coefficient at -0.74, indicating that it is not expected to partition into fat.

Plant Metabolism

Metabolism studies were conducted using dimethoate labelled with carbon-14 at both methoxy groups.



The Meeting received plant metabolism studies conducted on olives, potatoes, and wheat.

An olive tree was treated with 4 × 0.72 kg ai/ha foliar applications at retreatment intervals of 57, 30, and 44 days. The first application was made at BBCH 51–69 (inflorescence emergence to end of flowering) with the final application at BBCH 75–89 (fruit at 50% final size and above), 28 days before harvest maturity. Fruit was sampled just before and just after the third application, at two intervals between the third and fourth applications, and at 0-, 0+ (green and black olives), 14, 21 and 28 days (green and black olives) after the final application. Leaves were sampled after the first application, 43 days after the third application and 14, 21 and 28 days after the fourth application.

In leaves collected on the day of the first application, residues were easily removed by washing or simple solvent extraction, with 70% TRR (30 mg eq/kg) removed by the surface washes, and 30% TRR (13 mg eq/kg) extracted with acetonitrile and acetonitrile water, leaving only 0.2% TRR (0.08 mg eq/kg) unextracted. Most of the residue was present as unchanged parent compound (96% TRR).

Olives were surface washed with acetonitrile then separated into flesh and stone fractions. For olive flesh collected between 30 days after application 2 and 28 days after the final application, small fractions of radioactivity were removed in surface washes (1.5–17% TRR or 0.05–1.0 mg eq.kg), with the majority being extracted with hexane, acetonitrile, and acetonitrile/water (73–88% TRR or 2.5–4.3 mg eq/kg). Harsher extractions removed further proportions of the radioactive residues, with base extractions (1 M NaOH followed by 6 M NaOH) being the most successful, removing up to 10% TRR, reflecting the incorporation of some radioactivity into fatty acids. Much lower proportions of the residue were readily extractable from olive stone samples, with acetonitrile, acetonitrile/water and water extractions removing only 29–44% TRR (0.58–1.2 mg eq/kg), with unextracted residues correspondingly comprising 56–71% TRR (1.3–1.9 mg eq/kg).

Parent dimethoate was a major residue component in olive flesh shortly after an application, at 31% TRR (1.5 mg eq/kg) on the day of the third application, and 33% TRR (1.9 mg eq/kg) on the day of the fourth application. At other intervals, the largest component of the residue was O-desmethyl-N-desmethyl omethoate, at 36–60% TRR (1.5–2.9 mg eq/kg). The next largest component was OL-L4, which comprised mainly radioactivity incorporated into triglycerides and sterols, at 3.5–9.7% TRR (0.16–0.42 mg eq/kg). Other components identified in olive flesh were O-desmethyl isodimethoate, at 1.5–6.9% TRR (0.07–0.27 mg eq/kg), omethoate, at 0.4–3.2% TRR (0.02–0.16 mg eq/kg), dimethoate carboxylic acid, at < 0.1–2.7% TRR (< 0.01–0.10 mg eq/kg), O-desmethyl omethoate, at < 0.1–1.7% TRR (< 0.01–0.09 mg eq/kg), and isodimethoate, at < 0.1–2.5% TRR (< 0.01–0.08 mg eq/kg).

Potatoes were treated with 2 × 0.34 kg ai/ha foliar applications of ^{14}C-methoxy dimethoate at 14-day intervals at BBCH 45–47. Foliage and tuber samples were collected at intervals from 0 to 28 days after the second application. Foliage was surface washed with acetonitrile, then homogenised and extracted with acetonitrile, acetonitrile/water and water. Tubers were homogenised and extracted with acetonitrile and acetonitrile/water. Unextractable residues were further investigated by treating subsamples in parallel with acid (0.1 M HCl for 20 hours at 37 °C), base (0.1 M NaOH for 20 hours at 37 °C), or enzyme (bacterial protease type VIII digestion in phosphate buffer at 37 °C for 20 hours).

Total radioactive residues in foliage were 12.30 mg eq/kg at day 0 (after the second application), declining to 1.3 mg eq/kg by day 14, with a subsequent increase to 3.5 mg eq/kg day 28 due to drying of the foliage. Tuber residues were lower at 0.30 mg eq/kg at day 0, declining only

slightly to 0.19 mg eq/kg at day 14 and 0.24 mg eq/kg at day 28. Solvent extractability was 85% in foliage at day 0, decreasing to 56% at day 14 and 46% at day 28, and 90% in tubers at day 0, remaining high at 85% and 83% at days 14 and 28 respectively.

In foliage, dimethoate and omethoate accounted for 68% and 6% of the TRR respectively (8.4 and 0.73 mg eq/kg) on day 0. By day 14, dimethoate and omethoate accounted for 15% and 9% of the TRR in foliage respectively (0.2 and 0.12 mg eq/kg). Other residue components in foliage were O-desmethyl N-desmethyl omethoate (1.8% TRR, 0.22 mg eq/kg at day 0 and 8.7% TRR, 0.11 mg eq/kg at day 14), followed by O,O-dimethyl dithiophosphoric acid (0.3% TRR, 0.04 mg eq/kg at day 0 and 4.5% TRR, 0.06 mg eq/kg at day 14), O-desmethyl omethoate (3.2% TRR, 0.39 mg eq/kg at day 0 and 3.2% TRR, 0.04 mg eq/kg at day 14), co-eluting O-desmethyl isodimethoate and dimethoate carboxylic acid (0.8% TRR, 0.10 mg eq/kg at day 0 and 3.1% TRR, 0.04 mg eq/kg at day 14), hydroxy dimethoate glucose conjugate (2.3% TRR, 0.28 mg eq/kg at day 0 and 4.8% TRR, 0.06 mg eq/kg at day 14), desmethyl dimethoate (1.2% TRR, 0.02 mg eq/kg at day 14), and O-desmethyl omethoate carboxylic acid (1.5% TRR, 0.02 mg eq/kg at day 14).

No dimethoate or omethoate was detected in the tubers. The components identified in potato tubers were O-desmethyl N-desmethyl omethoate (0.23 mg eq/kg, 76% TRR at day 0, 0.09 mg eq/kg, 46% TRR at day 14, and 0.10 mg eq/kg, 44% TRR at day 28), O-desmethyl omethoate, (< 0.01 mg eq/kg at day 0, 0.02 mg eq/kg, 12% TRR at day 14 and 0.04 mg eq/kg, 15% TRR at day 28), and O-desmethyl omethoate carboxylic acid (0.02 mg eq/kg, 6.1% TRR at day 0, 0.03 mg eq/kg, 18% TRR at day 14), and 0.03 mg eq/kg, 12% TRR at day 28). Neither dimethoate nor omethoate are translocated from the foliage to the tubers and metabolism occurs mainly in the foliage.

Wheat (grown outdoors) was treated with two foliar applications, at BBCH 24 and 69. A 1× trial was carried out with application rates of 0.68 and 0.40 kg ai/ha for the first and second applications, and a 5× trial at rates of 3.4 and 2.0 kg ai/ha respectively. Samples were collected after the first application (day 0) and after 14, 26 and 39 days. Samples were also taken after the second application (day 41), and after 62 (early harvest) and 73 days (normal harvest). For the exaggerated rate trial, only grain, hull and straw were collected at 73 days. Depending on the growth stage of the plant, samples consisted of whole plant, ear, remaining plant, grain, hull or straw. Samples were extracted with acetonitrile and acetonitrile/water. Where significant radioactivity remained in the post-extraction solids (PES), further harsher extraction techniques were employed-dilute acid (0.1 M HCl), dilute base (0.1 M or 1 M NaOH), strong base (6 M NaOH) or enzymes (protease, cellulose/hemicellulose, and amylase).

Residues were highest in plant material immediately after application, with TRRs of 30 mg eq/kg in whole plant at day 0 after one application at 0.68 kg ai/ha, declining to 0.90 mg eq/kg in remaining plant at day 39 (before the second application). At harvest maturity, TRRs were 2.3 mg eq/kg at day 62 (21 days after application 2) and 4.3 mg eq/kg at day 73 (32 DAA2) in grain, 23 and 34 mg eq/kg at 21 and 32 DAA2 in hulls, and 6.4 and 7.8 mg eq/kg at 21 and 32 DAA2 in straw. Solvent extractability was generally high in plant material, at 99.8% of TRR at day 0 after the first (0.68 kg ai/ha application, declining to 91% of TRR in ears and 78% of the TRR in remaining plant at 39 days after the first application. At harvest, extractability was 81% and 66% of TRR from grain at 21 and 32 days after 0.68 + 0.40 kg ai/ha applications, 92% and 84% from hulls respectively and 79% and 72% from straw respectively. Only grain at 32 days after the second application was analysed from the exaggerated rate trial, with a TRR of 20 mg eq/kg. 63% of TRR was extractable with acetonitrile/water.

In whole wheat plants immediately after the first application at 0.68 kg ai/ha, the residues was mainly unmetabolized dimethoate at 98% TRR (29 mg eq/kg), with small amounts of omethoate (0.7% TRR, 0.21 mg eq/kg), and O-desmethyl N-desmethyl omethoate (0.4% TRR, 0.12 mg eq/kg). Dimethoate was rapidly metabolized in wheat. At 14 days after the first application, the residue components were dimethoate (4.1% TRR, 0.07 mg eq/kg), omethoate (7.8% TRR, 0.13 mg eq/kg), O-desmethyl isodimethoate (30% TRR, 0.49 mg eq/kg), O-desmethyl N-desmethyl omethoate (26% TRR, 0.44 mg eq/kg), O,O-dimethyl dithiophosphate (4.5% TRR, 0.08 mg eq/kg), and O-desmethyl omethoate carboxylic acid (1.1% TRR, 0.02 mg eq/kg). At 39 days, residue components in plant

material after removal of ears were O-desmethyl N-desmethyl omethoate (42% TRR, 0.37 mg eq/kg), O-desmethyl isodimethoate (22% TRR, 0.20 mg eq/kg), O-desmethyl omethoate carboxylic acid (5.7% TRR, 0.05 mg eq/kg), and O,O-dimethyl dithiophosphate (2.6% TRR, 0.02 mg eq/kg).

At harvest, no residues of dimethoate or omethoate were found in grain. The residue components in grain sampled at 21 days after the second application (0.68 + 0.40 kg ai/ha) were O-desmethyl N-desmethyl omethoate (54% TRR, 1.2 mg eq/kg), O-desmethyl isodimethoate (11% TRR, 0.26 mg eq/kg), and O-desmethyl omethoate carboxylic acid (3.8% TRR, 0.09 mg eq/kg). In straw at 21 days, residue components were dimethoate (6.2% TRR, 0.40 mg eq/kg), omethoate (3.4% TRR, 0.22 mg eq/kg), O-desmethyl N-desmethyl omethoate (36% TRR, 2.3 mg eq/kg), O-desmethyl isodimethoate (20% TRR, 1.3 mg eq/kg), O-desmethyl omethoate carboxylic acid (4.6% TRR, 0.30 mg eq/kg), and O,O-dimethyl dithiophosphoric acid (2.8% TRR, 0.18 mg eq/kg). At 32 days, the composition of the residue in grain and straw were similar.

In the exaggerated rate grain samples, residue components were O-desmethyl N-desmethyl omethoate (39% TRR, 8.0 mg eq/kg), O-desmethyl isodimethoate (16% TRR, 3.1 mg eq/kg), O-desmethyl omethoate carboxylic acid (6.4% TRR, 1.3 mg eq/kg), dimethoate (0.5% TRR, 0.10 mg eq/kg), and omethoate (0.3% TRR, 0.06 mg eq/kg).

The metabolism of [^{32}P]dimethoate in lemons, sugar beet, maize, cotton, peas, potatoes and beans was reported. The reports were summaries which did not provide the level of detail given in a contemporary metabolism study. Generally, the main components of the radiolabelled residue were dimethoate, omethoate, dimethoate carboxylic acid, dimethyl hydrogen phosphate and O,O-dimethyl hydrogen phosphorodithioate, indicating oxidation to omethoate, omethoate carboxylic acid and dimethoate carboxylic acid, and cleavage of the P-S linkage either before or after oxidation. The metabolic pathways in these studies were similar to the olive, potato and wheat study.

In the bean study, after foliar treatment with [^{14}C-methoxy]-dimethoate, parent compound comprised 40% of the foliage TRR, omethoate 1.6% TRR, while N-desmethyl dimethoate, dimethoate carboxylic acid, and O-desmethyl dimethoate carboxylic acid all comprised less than 1% of TRR.

In a study of the metabolism of [^{32}P]-dimethoate in excised cotton leaves with the cut petioles immersed in an aqueous solution of the radiolabel, dimethoate comprised 70% of the TRR on day 1, declining to 1.9% on day 14. The components identified were dimethoate carboxylic acid (15–50%), O,O-dimethyl dithiophosphoric acid (4–11%), omethoate (approximately 6%), dimethyl phosphate (2.5–11%), O,O-dimethyl thiophosphoric acid (1.6–12%), O-desmethyl dimethoate carboxylic acid (no fraction given), and phosphoric acid (no fraction given).

After foliar application of [^{32}P]-dimethoate to lemons, residue components identified were dimethoate, omethoate, dimethyl phosphoric acid, phosphoric acid, O,O-dimethyl thiophosphoric acid, and desmethyl dimethoate.

Summary of plant metabolism

The major metabolic pathways observed in plants treated by foliar application were:

- Oxidation to omethoate.
- O-Demethylation of omethoate to give O-desmethyl omethoate, and either subsequent N-demethylation to yield O-desmethyl N-desmethyl omethoate or amide hydrolysis to give O-desmethyl omethoate carboxylic acid.
- O-Demethylation and rearrangement to yield des-O-methyl isodimethoate.
- Hydroxylation of the N-methyl group in potatoes, with subsequent glucose conjugation.
- Hydrolysis of the amide bond and subsequent degradation to give dimethoate carboxylic acid, followed by dimethyl dithiophosphate.
- Incorporation into fatty acids was observed in olives.

Environmental Fate

The Meeting received studies of aerobic soil metabolism, soil surface photolysis, hydrolysis, aqueous photolysis, and laboratory and field soil dissipation of dimethoate, as well as field soil dissipation of omethoate.

Hydrolysis

Hydrolysis of ^{14}C-methoxydimethoate at 25 ± 1 °C was rapid at pH 9, with a half-life of 4.4 days, and significantly slower under neutral and acid conditions, with half-lives of 68 and 156 days at pH 7 and 5, respectively. The main hydrolysis products were *O*-desmethyldimethoate and *O,O*-dimethylphosphorothioic acid, which comprised 62.1% and 36.0% of the applied radioactivity at day 30 in the pH 9 study, while parent comprised only 1.1% of AR. Hydrolysis is not a significant degradation pathway under environmental conditions.

Aqueous photolysis

Photolysis is not a significant environmental degradation pathway for dimethoate, as shown by an aqueous photolysis study which determined a half-life of > 175 days (pH 5, 25 ± 1 °C, irradiation equivalent to at least 30 days of natural equatorial sunlight).

Soil surface photolysis

Photolysis at the soil surface did not occur, with essentially there being no difference in the degradation rate in the irradiated and dark control samples (half-lives of 10.5 and 7.9 days respectively (SFO model)).

Aerobic soil degradation

In aerobic soil incubated at 20 ± 2 °C in the dark, degradation of radiolabelled dimethoate was rapid, with parent being less than 1% of AR at day 21. The DT50 and DT90 were calculated at 2.6 and 8.6 days respectively by the SFO model. Mineralisation was the major degradation pathway, with $^{14}CO_2$ at 53% of AR at day 21, with significant fractions of the applied radioactivity being found as fluvic acids, humins, and humic acids (25%, 13%, and 4.5%, respectively). Levels of other degradation products were low, with desmethyl-dimethoate being found at < 5%, and omethoate not being detected.

In a laboratory study conducted at 20 ± 3 °C with three different UK soils with unlabeled compound, dimethoate was rapidly degraded, with DT50 values of 2.0–4.1 days, and D90 values of 6.8–13.5 days.

Field soil dissipation-dimethoate

In field studies conducted in Europe (one site each in Germany, Italy, the Netherlands and Spain) and the USA (one site in each of California, New York and Texas), degradation of dimethoate was rapid, with half-lives ranging from 2.2 to 9.8 days.

Field soil dissipation-omethoate

Field soil dissipation studies were conducted at four sites in Europe where omethoate was applied directly to soil. Degradation was very rapid, with half-lives ranging from 1.3–2.9 days, and DT_{90} values from 4.4–9.8 days. Similar results were obtained from some of the US dimethoate dissipation studies, where an omethoate half-life was calculated after measurement of both components, with half-lives for omethoate being < 1 day.

Neither dimethoate nor omethoate are persistent in soil.

Confined Rotational Crops

A confined rotational cropping study on wheat, lettuce and turnips was conducted using [^{14}C-methoxy]dimethoate. Planting boxes containing a sandy loam soil (pH 6.4, organic matter 1.6%) were treated with the test substance at 0.56 kg ai/ha. The test crops were planted in the ^{14}C-dimethoate treated soil at 30 and 120 days after treatment.

Wheat forage was harvested 62 and 168 days after application for the first and second rotation respectively. Lettuce was harvested 78 and 174 days after application, turnip (root and foliage) at 100 and 208 days after application, wheat hay at 97 and 216 days after application, while wheat grain and straw was harvested 141 and 272 days after application for each rotation.

TRRs were low, ranging from 0.008 mg eq/kg in turnip root to 0.045 mg eq/kg in wheat straw at the 30-day PBI and 0.001 mg eq/kg in turnip root to 0.020 mg eq.kg in wheat straw at the 120-day PBI. Extractability with acetonitrile and acetonitrile/HCl was variable, ranging from 3.1% in 120-day PBI grain, to 74% in 30-day PBI lettuce. Neither dimethoate nor omethoate could be identified. The extractable residue was characterized as consisting of multiple highly polar components.

Residues of dimethoate in rotational crops are not expected to be significant.

Animal Metabolism

The Meeting received animal metabolism studies for dimethoate in rats, hens and goats.

Rats

Evaluation of the metabolism studies in rats was carried out by the WHO Core Assessment Group.

Residue components observed in rat metabolism were dimethoate, omethoate, dimethoate carboxylic acid, dimethyl dithiophosphate, dimethyl thiophosphate, and dimethyl phosphate.

Goats

Lactating goats were treated with [^{14}C-methoxy]dimethoate by capsule once daily for 3 consecutive days at a dose equivalent to 30 ppm in the diet (1.6 mg/kg bw per day). Milk was collected twice daily. The animals were sacrificed 23 hours after the final dose for tissue collection.

Recovery of the administered dose was good, at 92–96% in excreta, milk and tissues. Total residues in milk ranged from 0.035–0.23 mg eq/kg, and were higher in samples collected within 8 hours post treatment, compared to samples collected 8 to 24 hours post treatment each day, suggesting rapid elimination of dimethoate from milk.

Total residues in tissues were 1.2 mg eq/kg in liver, 0.15 mg eq/kg in kidney, 0.070 mg eq/kg in muscle, and 0.045 mg eq/kg in fat.

Solvent extractability (acetonitrile, acetonitrile/water, acetone, and methanol/1 M NH_4OH) was variable, ranging from 28% in fat, to 90% in milk. The liver PES was subjected to harsher extraction techniques, with protease removing a further 22% of TRR, 6 M HCl at 90 °C removing 14% of TRR, and 3 M NaOH removing 1.8% of TRR.

Dimethoate was rapidly metabolized. In tissues and milk, the bulk of the residue comprised radioactivity incorporated into natural products (released by the solvent extraction, protease and acid extraction). Phosphorylated natural products comprised 62% TRR/0.76 mg eq/kg in liver, 87% TRR/0.13 mg eq/kg in kidney, 70% TRR/0.049 mg eq/kg in muscle, and 65% TRR/0.15 mg eq/kg in milk. Residues in fat were not further analysed due to the low levels. Anionic species (mainly dimethyl phosphate, dimethyl thiophosphate and dimethyl dithiophosphate) comprised 6.2% TRR/0.076 mg eq/kg in liver, 13% TRR/0.02 mg eq/kg in kidney, 2.9% TRR/0.002 mg eq/kg in muscle, and 2.2% TRR/0.005 mg eq/kg in milk. No parent compound was identified. Omethoate was found at 9.8% TRR/0.12 mg eq/kg in liver, while dimethoate carboxylic acid was found at 2.5% TRR/0.031 mg eq/kg in liver, and 8.3% TRR/0.019 mg eq/kg in milk. Both dimethoate carboxylic acid and omethoate were released from liver by protease digestion.

Hens

Three groups of laying White Leghorn hens were administered [^{14}C-methoxy]dimethoate by capsule once daily for 7 consecutive days at a dose rate equivalent to 10 ppm in the diet (approximately 0.9 mg/kg bw per day). Eggs were collected daily and separated into yolks and white for analysis. The birds were sacrificed approximately 23 hours after the final dose for tissue collection.

Mean cumulative radioactivity recovered in excreta (including cage wash) was 75%, in GI tract < 1% and in bile < 1%. Mean cumulative radioactivity recovered in eggs accounted for < 1% of the administered dose. Mean daily total residues in yolks ranged from 0.018 to 0.34 mg eq/kg, and in whites ranged from 0.090 to 0.180 mg eq/kg. A plateau in egg residues was not reached during dosing. Total residues in tissues were 0.62–0.69 mg eq/kg in liver, 0.079–0.10 mg eq/kg in muscle, 0.024–0.061 mg eq/kg in fat, and 0.042–0.066 mg eq/kg in skin.

Extractability of radioactivity from tissues and eggs with acetonitrile and water ranged from 8.9% in liver to 50% in egg white. Significant proportions were released by harsher treatments, with 10–44% of TRR released by protease digestion, 1–14% by weak base extraction, 2.4–18% by strong base reflux, and 1.2–27% by strong acid reflux.

Dimethoate was not detected in any of the tissues, egg, excreta or blood extracts, indicating rapid metabolism. Omethoate (3.1% TRR/0.004 mg eq/kg in egg white and 9.9% TRR/0.081 mg eq/kg in liver) and dimethoate carboxylic acid (3.9% TRR/0.005 mg eq/kg in egg white and 16% TRR/0.13 mg eq.kg in liver) were identified by HPLC. The largest fractions of these metabolites were released from liver and egg white by protease treatment.

The bulk of the residue in all matrices was characterized as phosphorylated natural products, at essentially 100% TRR/0.10 mg eq/kg in breast muscle, 92% TRR/0.072 mg eq/kg in thigh muscle, 70% TRR/0.027 mg eq/kg in skin, 62% TRR/0.51 mg eq/kg in liver, 94% TRR/0.18 mg eq/kg in egg yolk, 81% TRR/0.10 mg eq/kg in egg white, and 58% TRR/0.016 mg eq/kg in fat.

Summary of animal metabolism

The metabolic pathways in animal are qualitatively similar in rats, goats and hens.

The major metabolic reactions in animals are oxidation to give omethoate, and hydrolysis of the amide functional group giving dimethoate carboxylic acid, followed by further hydrolysis to anionic (thio)phosphate species, such as O,O-dimethyldithiophosphate, which can then be further oxidised to O,O-dimethylthiophosphate and O,O-dimethylphosphate. Formation of adducts of these anions with natural components, such as lipids and proteins was observed in goats and hens.

Methods of analysis

Plant matrices

The Meeting received a number of validated methods for determination of dimethoate, omethoate, and other plant metabolites in plant matrices.

A method for determination of dimethoate and omethoate involved extraction with dichloromethane followed by cleanup by liquid-liquid partition and dSPE and column chromatography, with analysis by GC-FPD or LC-MS. The method was successfully validated for both dimethoate and omethoate with an LOQ of 0.01 mg/kg in orange (GC-FPD) and a range of plant matrices (LC-MS).

A second GC-FPD method involved extraction with acetonitrile (olive oil) or ethyl acetate (other matrices). Samples were cleaned up by gel permeation chromatography. The method was validated with an LOQ of 0.01 mg/kg for both dimethoate and omethoate in olives, olive oil, orange, lettuce and wheat grain.

QuEChERS-based methods involve extraction with acetonitrile or acetonitrile/water, with cleanup by liquid/liquid partition and dSPE, followed by LC-MS/MS analysis. QuEChERS methods

were validated in an extensive range of plant matrices for analysis of dimethoate and omethoate with an LOQ of 0.001 mg/kg and an LOQ of 0.01 mg/kg for dimethoate carboxylic acid in olives, wheat green plants, grain and straw, and sugar beet roots and tops.

An LC-MS/MS method was developed and validated for determination of O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, desmethyl dimethoate, O-desmethyl omethoate, and O-desmethyl N-desmethyl omethoate in olives, wheat green plants, grain and straw, and sugar beet roots and tops. Samples were extracted with methanol/water and cleanup by column SPE before LC-MS/MS analysis. Good recoveries were achieved at 0.01 and 0.10 mg/kg for all analytes.

Animal matrices

A QuEChERS-based method was reported for determination of dimethoate and omethoate in animal matrices. Samples were extracted with acetonitrile (with the addition of water for fat samples), cleaned up by liquid/liquid partition and dSPE, and analysed by LC-MS/MS. Good recoveries were achieved at the LOQ (0.001 mg/kg), 0.005 and 0.5 mg/kg for both dimethoate and omethoate in milk, eggs, and tissues.

Stability of pesticide residues in stored analytical samples

Plant matrices

Residues of dimethoate and omethoate were shown to be stable for 27 months frozen storage in a range of plant matrices, including high starch (sorghum grain and potato), high oil (cottonseed), and high acid (orange) commodities.

The stability of additional metabolites was studied in wheat, olive and sugar beet matrices.

Dimethoate carboxylic acid was stable for at least 12 months in olives, wheat grain, straw and forage, and sugar beet roots and tops.

O-Desmethyl omethoate carboxylic acid was stable for up to 12 months in wheat straw, 3 months in olives, and wheat forage and grain, 1 month in sugar beet roots, and < 1 month in sugar beet tops.

O-Desmethyl isodimethoate was stable for up to 12 months in olives, and wheat grain and straw, 3 months in wheat forage, 4 months in sugar beet roots and 2 months in sugar beet tops.

O-Desmethyl omethoate was stable for up to 14 months in wheat grain, 3 months in olives, 2 weeks in wheat straw, 1 month in sugar beet roots, and < 1 month in sugar beet tops.

Desmethyl dimethoate was stable for up to 14 months in wheat grain, at least 6 months in wheat straw, 3 months in olives, 12 months in sugar beet roots, and 2 months in sugar beet tops.

O-Desmethyl N-desmethyl omethoate was stable for up to 8 weeks in olives, 6 weeks in wheat straw, 2 weeks in wheat forage, 1 month in wheat grain, and < 1 month in sugar beet roots or tops.

Animal matrices

A stability study in tissues, milk and eggs showed that dimethoate was stable on frozen storage for up to 12 months in muscle, fat, milk, and egg, and 6 months in liver and kidney. Omethoate was stable for up to 12 months in milk and fat, 9 months in muscle, 2 months in eggs, and only 2 weeks in liver and 1 week in kidney. Samples in the feeding studies were analysed within the verified period of stability.

Residue Definition

Plant commodities

Metabolism studies in olives, potatoes and wheat were submitted to the Meeting, along with a number of older studies with limited supporting information (mainly published papers) previously submitted to JMPR which included summarized metabolism data on lemons, sugar beet, maize, cotton, peas, potatoes and beans.

In the olive, wheat and potato studies, the major components of the residue in matrices treated directly with dimethoate at shorter sampling intervals (0–14 days) were: dimethoate (15–68% TRR, 0.20–8.4 mg/kg in potato foliage; 4.1–98% TRR, 0.07–29 mg/kg in wheat whole plants; 31–33% TRR, 1.9 mg/kg in olive flesh), omethoate (5.9–16% TRR, 0.12–0.73 mg eq/kg in potato foliage; 0.7–7.8% TRR, 0.13–0.42 mg eq/kg in wheat whole plants; 1.8–3.2% TRR, 0.11–0.16 mg eq/kg in olive flesh), O-desmethyl N-desmethyl omethoate (1.8–8.7% TRR, 0.11–0.24 mg eq/kg in potato foliage; 0.4–26% TRR, 0.12–0.71 mg eq/kg in wheat whole plants; 35–36% TRR, 0.18–2.1 mg eq/kg in olive flesh), and O-desmethyl isodimethoate (< 0.2–3.7% TRR, < 0.01–0.17 mg eq/kg in potato foliage; < 0.1–30% TRR, < 0.03–0.60 mg eq/kg in wheat whole plants; 1.5–3.2% TRR, 0.07–0.19 mg eq/kg in olive flesh).

In matrices to which residues are translocated, and at longer intervals after application, the metabolite pattern is different and dimethoate and omethoate are no longer present, or are only present at low levels. Total residues in these matrices are generally lower than in the foliage and fruit matrices closer to application. In olive flesh 28–43 days after 3 or 4 applications, the major residues were O-desmethyl N-desmethyl omethoate (53–60% TRR, 2.2–2.9 mg eq/kg) and O-desmethyl isodimethoate (4.0–6.9% TRR, 0.21–0.27 mg eq/kg). In wheat grain at 21–32 days after the second application, the major residues were O-desmethyl N-desmethyl omethoate (46–54% TRR, 1.2–2.0 mg eq/kg), O-desmethyl isodimethoate (6.8–11% TRR, 0.26–0.29 mg eq/kg), and O-desmethyl omethoate carboxylic acid (3.6–3.8% TRR, 0.09–0.15 mg eq/kg). In potato tubers at 0–28 days after the second application the major residues were O-desmethyl N-desmethyl omethoate (40–76% TRR, 0.09–0.23 mg eq/kg), O-desmethyl omethoate (< 0.1–28% TRR, < 0.01–0.07 mg eq/kg), and O-desmethyl omethoate carboxylic acid (6.1–18% TRR, 0.02–0.03 mg eq/kg).

Dimethoate was a significant residue component in bean forage, and omethoate was a significant component in lemons. In excised cotton leaves, dimethoate carboxylic acid was a major component.

In a number of residue trials in wheat, olives and sugar beet, dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl omethoate, O-desmethyl N-desmethyl omethoate, O-desmethyl omethoate carboxylic acid, O-desmethyl isodimethoate, and desmethyl dimethoate were analysed.

Table 2 Summary of residue components in field trials for olives, sugar beet and wheat

Component	Maximum residues (mg/kg)					
	Olives	Sugar beet roots	Sugar beet tops	Wheat grain	Wheat forage/hay	Wheat straw
Dimethoate	6.3	< 0.01	3.6	< 0.01	8.3	0.76
Omethoate	1.1	< 0.01	0.33	< 0.01	0.32	0.07
Dimethoate carboxylic acid	0.02	< 0.01	0.01	< 0.01	0.08	0.12
O-desmethyl isodimethoate	0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
O-desmethyl omethoate carboxylic acid	0.35	< 0.01	< 0.01	< 0.01	< 0.01	0.05
Desmethyl dimethoate	0.33	0.03	0.60	< 0.01	0.22	0.21
O-desmethyl omethoate	0.13	< 0.01	0.18	< 0.01	0.02	0.08
O-desmethyl N-	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

Component	Maximum residues (mg/kg)					
	Olives	Sugar beet roots	Sugar beet tops	Wheat grain	Wheat forage/hay	Wheat straw
desmethyl omethoate						

No residues of any of the components were found above the LOQ in wheat grain, apart from a single detection of dimethoate at the LOQ in one of the 16 samples. In sugar beet roots, all components other than desmethyl dimethoate were below the LOQ, while low levels of desmethyl dimethoate (maximum 0.03 mg/kg) were found in 8 out of 48 samples. In olives, dimethoate and omethoate were the most significant residue components, with dimethoate reaching a maximum of 6.3 mg/kg and omethoate a maximum of 1.1 mg/kg, with O-desmethyl omethoate carboxylic acid, desmethyl dimethoate, and O-desmethyl omethoate reaching maximum levels of 0.35, 0.33 and 0.13 mg/kg.

Based on the metabolism studies and field trials, dimethoate and omethoate are good marker compounds particularly for shorter pre-harvest intervals, and in directly treated commodities such as leafy vegetables and fruits. In the olive residue studies, dimethoate and omethoate were by far the most significant residue components in olive fruit. For commodities to which dimethoate residues are translocated, such as grain, bulbs or tubers, total residues are generally low. Levels of dimethoate, omethoate, and six other metabolites were mostly undetectable in sugar beet roots and wheat grain in the field residue studies discussed above.

Residues of dimethoate or its metabolites are unlikely to occur in rotational crops based on the confined rotational crop metabolism study.

Therefore, in practice, the most suitable marker residues in plant commodities are considered to be dimethoate and omethoate. The Meeting noted that suitable validated methods were available for dimethoate and omethoate in an extensive range of plant commodities.

The Meeting further noted that in residue trials for some commodities, for example cherries and olives, omethoate was often present at higher levels than dimethoate, particularly around harvest. Therefore, inclusion of both dimethoate and omethoate in the definition for compliance with MRLs is warranted. The Meeting noted that omethoate is itself a pesticide and should therefore be measured separately from dimethoate.

The Meeting considered that a suitable residue definition for compliance with MRLs in plant commodities is *dimethoate and omethoate, measured and reported separately*.

The Meeting considered that dimethoate and omethoate were expected to have similar bioavailability to livestock, and determined that the *sum of dimethoate and omethoate* would be used to estimate median and highest residues in feed commodities for estimation of livestock dietary burden.

As a result of concerns relating to the genotoxicity of omethoate and other related metabolites, a conclusion was unable to be reached on a residue definition for dietary risk assessment.

Animal commodities

In goats, dimethoate carboxylic acid was identified at 0.019 mg eq/kg in milk and 0.031 mg eq/kg in liver, while omethoate was identified in liver at 0.12 mg eq/kg. The bulk of the radioactivity in goat milk and tissues was comprised of phosphorylated natural products. A similar pattern was observed in hens, with only dimethoate carboxylic acid (0.13 mg eq/kg in liver and 0.005 mg eq/kg in egg white) and omethoate (0.004 mg eq/kg in egg white and 0.081 mg eq/kg in liver) identified, and the major component of the residue incorporated into natural products.

In the lactating cattle feeding study, no residues of dimethoate were found above the LOQ in milk, muscle, liver, or kidney, while low levels of omethoate were detected in milk, kidney, and muscle for the highest dose group, in liver for the highest and second highest dose groups, while low

levels of dimethoate were detected in fat at all doses, without any clear correlation between dose and residue level. Omethoate was detected in fat at higher dose levels.

In the laying hen feeding study, no residues of dimethoate or omethoate were detected in tissues or eggs at any dose level.

Dimethoate and omethoate, are considered to be the most suitable marker residues for enforcement in animal commodities. The Meeting noted that suitable validated methods were available for dimethoate and omethoate in milk, eggs and tissues.

The Meeting considered that a suitable residue definition for compliance with MRLs in animal commodities is *dimethoate and omethoate, measured and reported separately*.

The octanol-water partition coefficients of dimethoate and omethoate ($log_{10}K_{OW}$ values) are 0.75 and -0.74 respectively, indicating that neither compound is lipophilic. The Meeting further noted that residues of dimethoate and omethoate did not preferentially partition into cream rather than skim milk in the cattle feeding study. Residues of dimethoate and omethoate are not fat-soluble.

As for plant commodities, the Meeting was unable to reach a conclusion on a residue definition for dietary risk assessment in animal commodities.

Results of supervised residue trials on crops

The residue trial tables include values for the sum of dimethoate and omethoate for used livestock dietary burden calculation where applicable.

Where residues were reported below the LOQ, the following conventions were adopted for summing residues (using an LOQ of 0.01 mg/kg as an example):

Table 3 Convention adopted for summing of residues

Dimethoate (mg/kg)	Omethoate (mg/kg)	Sum of dimethoate and omethoate (mg/kg)
0.30	0.04	0.34
0.30	< 0.01	0.31
< 0.01	< 0.01	< 0.02

As a conclusion could not be reached on a residue definition for dietary risk assessment, the Meeting withdrew all previous recommendations for maximum residue limits for dimethoate and omethoate, including the spice MRLs.

Citrus fruit

There are two GAPs for citrus fruit in Australia:

- 3 × 0.03 kg ai/100 L dilute foliar applications (14-day intervals), with a 7-day PHI OR
- A post-harvest dip or flood spraying application at a concentration of 0.04 kg ai/100 L (40 ppm), with no withholding period required. Use instructions in Australia preclude both the foliar application and the post-harvest application being made to the same fruit.

Residue trial data from Australia were conducted in accordance with the Australian post-harvest GAP.

Residues of dimethoate in mandarin (whole fruit) were (n = 4) 0.58, 0.70, 0.71, and 0.82 mg/kg.

Residues of omethoate in mandarin (whole fruit) were (n = 4) < 0.01 (4) mg/kg.

Residues of dimethoate in orange (whole fruit) were (n = 6) 0.51, 0.59, 0.60, 0.63, 0.66, and 0.67 mg/kg.

Residues of omethoate in orange (whole fruit) were (n = 6) 0.003 (3), 0.004 (2), and 0.005 mg/kg.

No residue data in accordance with the Australian foliar application GAP were available to the Meeting.

Residue trials for a foliar GAP in Brazil (dilute foliar applications at 0.04 kg ai/100 L with a 3-day PHI) are available. The Meeting noted that this GAP is in excess of the Australian foliar treatment GAP. Based on the residue levels in the Brazilian trials and in the Australian post-harvest trials, the Meeting considered that the post-harvest GAP in Australia was the critical GAP and that maximum residue levels based on the post-harvest GAP would cover residues arising from the alternative foliar Australian GAP.

The Meeting noted that the GAPs for post-harvest treatment of oranges and mandarins in Australia are the same and investigated combining the data sets for mutual support. The mandarin and orange data were likely to come from the same population (Mann-Whitney).

Combined mandarin and orange dataset for dimethoate: (n = 10) 0.51, 0.58, 0.59, 0.60, 0.63, 0.65, 0.67, 0.70, 0.71, and 0.82 mg/kg.

Combined mandarin and orange dataset for omethoate: (n = 10) 0.003 (3), 0.004 (2), 0.005, and < 0.01 (4) mg/kg.

The Meeting considered that mandarins and oranges were representative crops for the subgroups of mandarins and oranges respectively.

Based on the combined dataset, the Meeting estimated maximum residue levels of 2 mg/kg for dimethoate in the subgroup of mandarins and the subgroup of oranges.

The Meeting estimated maximum residue levels of 0.02 mg/kg for omethoate in the subgroup of mandarins and the subgroup of oranges.

Cherries

The critical GAP for cherries is in the USA (a single application at 1.5 kg ai/ha with a 21-day PHI). No trials were available matching that GAP.

The GAP for cherries in the Czech Republic is a single application at 0.4 kg ai/ha with a 28-day PHI. Trials were conducted in northern Europe in accordance with this GAP.

Residues of dimethoate in cherries after treatment in accordance with the Czech GAP were (n = 4) < 0.01 (3), and 0.01 mg/kg.

Residues of omethoate in cherries were (n = 4) < 0.01, 0.04, 0.05, and 0.08 mg/kg.

The Meeting concluded that there was insufficient data matching GAP to estimate a maximum residue level for cherries.

Olives

The critical GAP for olives is in Greece, with a single bait spray application applied to the trunk of 100 trees per hectare at a rate of 0.05 kg ai/ha followed after 10 days by 2 × 0.48 kg ai/ha foliar applications at a 14-day retreatment interval, with a 28-day PHI.

There were no residue trials available to the Meeting involving both the bait spray and the foliar applications being made to the same trees.

Residue data were available from trials in southern France, Greece, Italy and Spain in accordance with the 2 × 0.48 kg ai/ha foliar use pattern.

Residues of dimethoate in olives (whole fruit) were (n = 28) < 0.01 (8), 0.02, 0.04 (2), 0.05, 0.06 (2), 0.07, 0.11 (2), 0.12, 0.22, 0.34, 0.41, 0.55, 0.63, 0.74, 0.76, 1.2, and 1.5 (2) mg/kg. .

Residues of omethoate were (n = 28) < 0.01 (2), 0.03, 0.05, 0.07, 0.11, 0.15, 0.20, 0.21 (2), 0.22, 0.24, 0.26, 0.27 (2), 0.28 (2), 0.29 (2), 0.30, 0.34, 0.35 (2), 0.36, 0.50, 0.69, 0.78, and 0.88 mg/kg.

Two trials in accordance with the bait spray application use pattern were conducted in Greece. The latest sampling interval in these trials was 42 days after application. At 42 days, residues of dimethoate were < 0.01 (2) mg/kg and residues of omethoate were < 0.01 and 0.01 mg/kg.

Residues from the bait spray application are therefore expected to be much lower than those arising from the foliar application and therefore not expected to make a significant contribution to the residue in mature olives. The maximum residue level parameters estimated based on the foliar use pattern trials are therefore considered to accommodate residues from both the bait and foliar use patterns.

Based on the foliar application dataset, the Meeting estimated maximum residue levels of 3 mg/kg for dimethoate in table olives and olives for oil production.

The Meeting estimated maximum residue levels of 1.5 mg/kg for omethoate in table olives and olives for oil production.

Tropical fruit – inedible peel

Avocados

The critical GAP for avocados in Australia is dilute foliar applications at 0.03 kg ai/100 L as required, with a 7-day PHI, followed by a post-harvest dipping application at 0.04 kg ai/100 L for 1 minute followed by packing the fruit after allowing to drain. A withholding period is not required for the post-harvest application.

Trials were conducted in avocados in Australia at GAP, with avocado orchards being treated with three dilute foliar spray applications at 0.03 kg ai/100 L at a 21- then a 7-day interval, fruit being harvested 7 days after the last application and then given a post harvest dip at 0.04 kg ai/100 L.

Residues of dimethoate in whole avocados were (n = 4) 0.41, 0.44, 0.71, and 0.75 mg/kg.

Residues of omethoate in whole avocados were (n = 4) 0.016, 0.025, 0.042, and 0.067 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg for dimethoate in avocado.

The Meeting estimated a maximum residue level of 0.15 mg/kg for omethoate in avocado.

Mangoes

The critical GAP for mangoes in Australia is dilute foliar applications at 0.03 kg ai/100 L as required, with a 3-day PHI, followed by a post-harvest dipping application at 0.04 kg ai/100 L for 1 minute followed by packing the fruit after following draining. A withholding period is not required for the post-harvest application.

Trials were conducted in mangoes in Australia at GAP, with mango orchards being treated with three dilute foliar spray applications at 0.03 kg ai/100 L at a 7-day interval, fruit being harvested 3 days after the last application and then given a post harvest dip at 0.04 kg ai/100 L.

Residues of dimethoate in whole mangoes were (n = 4) 0.17, 0.24, 0.34, and 0.43 mg/kg.

Residues of omethoate in whole mangoes were (n = 4) < 0.02, 0.03, 0.05, and 0.06 mg/kg.

The Meeting considered that there were insufficient data to estimate a maximum residue level for mangoes.

Onion, bulb

The critical GAP for onions is in Australia, with applications at 0.3 kg ai/ha as required, with a 7-day PHI. However, no trials matching that GAP were available to the Meeting.

The GAPs for onions in the Czech Republic and Greece is a 2 × 0.24 kg ai/ha applications, with a 14-day PHI.

Trials were conducted in France, Germany, Greece, Italy and the UK in accordance with the Czech and Greek GAPs.

Residues of dimethoate in onion bulbs were (n = 8) < 0.001 (7), and 0.005 mg/kg.

Residues of omethoate were (n = 8) < 0.001 (7), and 0.001 mg/kg.

The Meeting estimated maximum residue levels of 0.01 mg/kg for dimethoate and omethoate in onion (bulb).

Brassica vegetables

Subgroup of flowerhead brassicas

There were no trials available to the Meeting matching the US GAPs for broccoli, or cauliflower (3 × 0.56 kg ai/ha applications, and a 7-day PHI).

The GAP in Estonia for broccoli, and cauliflower is 2 × 0.24 kg ai/ha applications at a 7-day retreatment interval and with a 21-day PHI.

Trials in broccoli and cauliflower conducted in Denmark, France, Germany, and the UK in accordance with the Estonian GAP were available to the Meeting.

Residues of dimethoate in broccoli were (n = 4) < 0.01 (4) mg/kg.

Residues of omethoate in broccoli were (n = 4) < 0.01 (4) mg/kg.

Residues of dimethoate in cauliflower were (n = 9) < 0.01 (5), 0.01 (3), and 0.13 mg/kg.

Residues of omethoate in cauliflower were (n = 9) < 0.01 (9) mg/kg.

The Meeting noted that the GAPs were the same for broccoli and cauliflower and that the data were likely to come from the same population (Mann-Whitney). The Meeting agreed to combine the broccoli and cauliflower data sets for estimating a subgroup maximum residue level.

Residues of dimethoate in flowerhead brassicas: (n = 13) < 0.01 (9), 0.01 (3), and 0.13 mg/kg.

Residues of omethoate in flowerhead brassicas: (n = 13) < 0.01 (13) mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for dimethoate in the subgroup of flowerhead brassicas.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg for omethoate in the subgroup of flowerhead brassicas.

Subgroup of head brassicas

The GAP in the Czech Republic for Brussels sprouts is 2 × 0.24 kg ai/ha applications with a 21-day PHI.

Trials in Brussels sprouts conducted in Denmark, France, Germany, and the UK in accordance with the Estonian GAP were available to the Meeting.

Residue of dimethoate in Brussels sprouts were: (n = 9) < 0.01 (3), 0.01, 0.02 (3), and 0.03 (2) mg/kg.

Residues of omethoate in Brussels sprouts were: (n = 9) < 0.01 (8), and 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg for dimethoate in Brussels sprouts.

The Meeting estimated a maximum residue level of 0.015 mg/kg for omethoate in Brussels sprouts.

The GAP in Estonia for cabbage, head is 2 × 0.24 kg ai/ha applications with a 10-day retreatment interval and a 14-day PHI.

Residue data from trials in northern Europe in accordance with the Estonian GAP was available to the Meeting.

Residues of dimethoate in cabbage, head (with wrapper leaves) were (n = 4) < 0.01 (4) mg/kg.

Residues of omethoate in cabbage, head (with wrapper leaves) were (n = 4) < 0.01 (4) mg/kg.

The Meeting considered that there were insufficient data to estimate a maximum residue level for cabbage, head.

Melons

There were no trials available to the Meeting matching the US GAP (2 × 0.56 kg ai/ha applications with a 3-day PHI), or the Australian GAP (applications at 0.3 kg ai/ha, or 0.03 kg ai/100 L with a 7-day PHI).

The GAP in Greece is 2 applications before flowering at 0.24 kg ai/ha, with a harvest PHI not required.

Residues trials conducted in Greece, Italy, and Spain were available to the Meeting, but the trials did not match GAP, as the applications were conducted too late in the season (last application between BBCH 71 and 82).

Fruiting vegetables, other than Cucurbits

Peppers, sweet (capsicum)

There were no trials available to the Meeting matching the US GAP (5 × 0.37 kg ai/ha applications with a 0-day PHI).

The GAP in Australia for peppers, sweet (capsicum) is dilute foliar application at 0.03 kg ai/100 L up to 0.3 kg ai/ha with a 3-day PHI.

Trials were conducted at GAP in Australia.

Residues of dimethoate were (n = 5) 0.04, 0.08, 0.14, 0.15, and 0.32 mg/kg.

Residues of omethoate were (n = 5) < 0.02, 0.02, < 0.04, 0.04, and 0.11 mg/kg.

The Meeting concluded that there was insufficient trial data to estimate a maximum residue level.

Tomato

No trials were available matching the Brazilian or US GAPs.

The critical GAP for tomatoes is in Australia is 2 × 0.3 kg ai/ha applications at a 14-day retreatment interval and with a 21-day PHI.

Trials conducted in southern Europe involved 2 × 0.6 kg ai/ha applications, with a 21-day harvest PHI and these matched the Australian GAP with the exception of the higher application rate. The Meeting considered that the proportionality principle could be applied.

Residues of dimethoate were (n = 8) < 0.01 (8) mg/kg.

Residues of omethoate were (n = 8) < 0.01 (6), and 0.01 (2) mg/kg.

Applying a scaling factor (0.5×) for the Australian GAP for tomatoes:

Residues of dimethoate: < 0.005 (8) mg/kg

Residues of omethoate: < 0.005 (6) and 0.005 (2) mg/kg

The Meeting estimated a maximum residue level of 0.01(*) mg/kg for dimethoate in tomatoes.

The Meeting estimated a maximum residue level of 0.01 mg/kg for omethoate in tomatoes.

Eggplant

The GAP for eggplant in Greece is 2 × 0.24 kg ai/ha foliar applications with a 21-day PHI.

Two trials were conducted in Australia, however these did not match GAP as the PHI was too short. The Meeting considered there was insufficient data to estimate a maximum residue level for eggplant.

Leafy vegetables

Lettuce

No trials were available matching the US GAP (3 × 0.28 kg ai/ha with a 14-day PHI).

The critical GAP for lettuce in the Czech Republic is 2 × 0.24 kg ai/ha foliar applications with a 21-day PHI.

Trials were conducted in leaf lettuce varieties in Denmark, France, Poland, and the UK, in accordance with the Czech GAP.

Residues of dimethoate in lettuce, leaf were (n = 8) < 0.01 (6), 0.01, and 0.09 mg/kg.

Residues of omethoate in lettuce, leaf were (n = 8) < 0.01 (7), and 0.04 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for dimethoate in lettuce, leaf.

The Meeting estimated a maximum residue level of 0.06 mg/kg for omethoate in lettuce, leaf.

Turnip greens

The GAP in the USA in turnips is 7 × 0.28 kg ai/ha applications at 3-day intervals with a 14-day PHI.

Trials were conducted in turnips in the USA, however, these did not match the maximum GAP, as only three applications were made, at 7-day intervals.

Legume vegetables

Peas (succulent without pods)

The GAP in the USA for peas without pods is 2 applications at 0.20 + 0.36 kg ai/ha (maximum individual rate of 0.36 kg ai/ha and maximum seasonal rate of 0.56 kg ai/ha), with a 0-day PHI.

A number of trials from the USA was available to the Meeting, however these did not match GAP.

Peas (succulent with pods)

The GAP for peas with pods is 3 × 0.18 kg ai/ha with a 0-day PHI.

A number of trials from the USA was available to the Meeting, however these did not match GAP.

Yard long bean

The GAP in Thailand for yard long bean is 4 × 0.6 kg ai/ha foliar applications with a 7-day PHI.

Trials were conducted in Thailand, with either 3 or 4 × 0.6 kg ai/ha applications being made. There was no significant difference in residues at a 7-day PHI after either three or four applications at

the label rate, and trials with both three and four applications are considered representative of the residues expected after treatment in accordance with the GAP.

Residues of dimethoate in yard long bean were (n = 6) < 0.05 (5) and 0.05 mg/kg.

Residues of omethoate in yard long bean were (n = 6) < 0.05 (6) mg/kg.

The Meeting estimated a maximum residue level of 0.07 mg/kg for dimethoate in yard long bean.

The Meeting estimated a maximum residue level of 0.05 mg/kg for omethoate in yard long bean.

Pulses

Peas (dry)

The US GAP for peas (dry) is 2 applications at 0.20 + 0.36 kg ai/ha (maximum individual rate of 0.36 kg ai/ha and maximum seasonal rate of 0.56 kg ai/ha), with a 0-day PHI.

Residue trials were conducted in the USA, however, only two independent trials were available. Further, only dimethoate residues were tested and the trials did not match GAP. The Meeting concluded that there was insufficient data to estimate a maximum residue level for peas (dry).

Beans (dry)

The critical GAP for beans (dry), including adzuki beans, cowpeas, mung beans, navy beans, Borlotti beans, and field beans in Australia is an unspecified number of applications at a rate of 0.32 kg ai/ha and 14-day intervals, with a PHI of 14 days for both grazing and harvest.

Residue data matching the Australian GAP for beans except soya beans (3 × 0.32 kg ai/ha applications at 14-day intervals) was available for mung beans and navy beans.

Residues of dimethoate in dry beans (mung and navy beans) were (n = 6) < 0.05 (4), 0.066, and 0.40 mg/kg.

Residues of omethoate in dry beans (mung and navy beans) were (n = 6) < 0.05 (5), and 0.064 mg/kg.

Residues for livestock dietary burden estimation (sum of dimethoate and omethoate) in dry beans were (n = 6) < 0.10 (4), 0.17, and 0.46 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg for dimethoate in the subgroup of dry beans.

The Meeting estimated a maximum residue level of 0.08 mg/kg for omethoate in the subgroup of dry beans.

The Meeting estimated a median residue of 0.10 mg/kg.

Soya bean (dry)

The critical GAP for soya beans in Australia is an unspecified number of applications at a rate of 0.136 kg ai/ha and 14-day intervals, with a PHI of 14 days for both grazing and harvest.

Residues of dimethoate in soya bean, dry were < 0.05 (3) mg/kg.

Residues of omethoate in soya bean, dry were < 0.05 (3) mg/kg.

The Meeting considered that there were insufficient data to estimate a maximum residue level for soya bean.

Root and tuber vegetables

Carrot

The critical GAP for carrots is in Estonia, with 3 × 0.24 kg ai/ha applications at a 7-day retreatment interval and with a 28-day PHI.

Two series of residue trials were conducted in Europe, one with 3 × 0.24 kg ai/ha foliar applications and the other with 4 × 0.24 kg ai/ha foliar applications. Decline data was available from a number of the trial sites, and a median half-life of 9.9 days for the sum of dimethoate and omethoate (residue definition for chronic risk assessment) was calculated from eight trials. Based on a re-treatment interval of 7 days and a PHI of 28 days, residues at harvest after four applications would be approximately 1.1× those expected after three applications. The Meeting therefore considered that the data from trials with four applications could be combined with the data from trials with three applications for the purpose of estimated a maximum residue level and dietary intake parameters for carrots.

Residues of dimethoate were (n = 16) < 0.001 (8), 0.001 (3), 0.002 (2), 0.004, 0.006, and 0.008 mg/kg.

Residues of omethoate were (n = 16) < 0.001 (4), 0.001, 0.0013 (2), 0.002, 0.003 (3), 0.004, 0.005, 0.006, 0.008, and 0.013 mg/kg.

Residues of dimethoate + omethoate for estimation of livestock dietary burden were (n = 16) < 0.002 (5), 0.002 (2), 0.003, 0.004, 0.005, 0.006 (2), 0.007, 0.012, 0.014, and 0.016 mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg for dimethoate in carrots.

The Meeting estimated a maximum residue level of 0.02 mg/kg for omethoate in carrots.

The Meeting estimated median and highest residues for livestock dietary burden calculation of 0.0035 and 0.016 mg/kg respectively.

The Meeting noted that the GAPs in Estonia for parsnip and parsley, turnip rooted were the same as those for carrot. The Meeting agreed to extrapolate the estimations for carrots to parsnips and parsley, turnip rooted.

Sugar beet

The critical GAPs for sugar beet are in the Czech Republic and Greece, 2 × 0.24 kg ai/ha applications with a 28-day PHI.

An extensive data set from northern and southern Europe, matching the Czech and Greek GAPs, was available to the Meeting.

Residues of dimethoate in sugar beet roots were (n = 17) < 0.001 (17) mg/kg.

Residues of omethoate in sugar beet roots were (n = 17) < 0.001 (17) mg/kg.

The Meeting estimated maximum residue levels of 0.001(*) mg/kg for both dimethoate and omethoate in sugar beet.

Turnip

The GAP in the USA in turnips is 7 × 0.28 kg ai/ha applications at 3-day intervals with a 14-day PHI.

Trials were conducted in turnips in the USA, however, these did not match the maximum GAP, as only three applications were made, at 7-day intervals.

Asparagus

Two Italian trials in asparagus were available to the Meeting, however they did not match any of the GAPs available.

Cereals

Barley

The GAP for barley in Estonia is a single 0.20 kg ai/ha application up to BBCH 59 (end of heading, inflorescence fully emerged), with a PHI not required.

Trials were conducted in barley in Europe in accordance with the Estonian GAP.

Residues of dimethoate in barley were (n = 7) < 0.001 (6) and 0.016 mg/kg.

Residues of omethoate in barley were (n = 7) < 0.001 (6) and 0.003 mg/kg.

Residues for livestock dietary burden estimation (sum of dimethoate and omethoate) were (n = 7) < 0.002 (6), and 0.019 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg for dimethoate in barley.

The Meeting estimated a maximum residue level of 0.01 mg/kg for omethoate in barley.

The Meeting estimated a median residue of 0.002 mg/kg.

The Meeting noted that the same GAP was authorized for oats in Estonia and considered that the residue potential was similar for barley and oats. The Meeting agreed that the estimations for barley could be extrapolated to oats, and estimated maximum residue levels of 0.03 and 0.01 mg/kg for dimethoate and omethoate respectively, together with a median residue of 0.002 mg/kg.

Wheat

The GAP for wheat in the Czech Republic and Estonia is a single 0.20 kg ai/ha application up to BBCH 69 (end of flowering), with a PHI not required.

Residue trials were conducted in wheat in Europe in accordance with the Czech and Estonian GAPs.

Residues of dimethoate in wheat were (n = 32) < 0.001 (13), 0.002 (2), 0.005, < 0.01 (15), and 0.01 mg/kg.

Residues of omethoate in wheat were (n = 32) < 0.001 (15), 0.001, and < 0.01 (16) mg/kg.

Residues for livestock dietary burden estimation (sum of dimethoate and omethoate) were (n = 32) < 0.002 (12), 0.002, 0.003 (2), 0.006, < 0.02 (15), and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg for dimethoate in wheat.

The Meeting estimated a maximum residue level of 0.03 mg/kg for omethoate in wheat.

The Meeting estimated a median residue of 0.013 mg/kg.

The Meeting noted that the same GAP was authorized for rye and triticale in the Czech Republic and Estonia, considered that the residue potential was similar for wheat, rye and triticale and agreed that the estimations for wheat could be extrapolated to rye and triticale.

The Meeting estimated maximum residue levels of 0.03 mg/kg for dimethoate and omethoate in rye and triticale, together with median residues of 0.013 mg/kg.

Rape seed (canola)

The Australian GAP for rape (canola) is a single application at 0.14 kg ai/ha with a PHI of 7 days for both harvest and grazing.

Trials were conducted in canola in Australia according to GAP.

Residues of dimethoate in rape seed were (n = 8) < 0.02, 0.02, 0.026, 0.027, 0.028, 0.051, 0.066, and 0.084 mg/kg.

Residues of omethoate in rape seed were (n = 8) < 0.02 (7) and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for dimethoate in rape seed.

The Meeting estimated a maximum residue level of 0.03 mg/kg for omethoate in rape seed.

Animal feeds

Sugar beet tops

Data for sugar beet tops from trials in accordance with the Czech and Greek GAPs was available to the Meeting.

Residues for estimation of livestock dietary burden in sugar beet tops were: (n = 17) < 0.02 (12), 0.03, 0.04 (2), and 0.06 (2) mg/kg (as received basis) or < 0.13 (12), 0.20, 0.27 (2), and 0.40 (2) mg/kg (dry weight basis, converted using the default dry matter content of 15% from the OECD livestock feed tables).

The Meeting estimated a median and a highest residue of 0.13 and 0.40 mg/kg respectively (dry weight basis).

Barley straw and fodder

Residue data for barley hay and straw was available from the European trials.

Residues of dimethoate in barley hay were (n = 4) < 0.012, < 0.014 (2), and 0.14 mg/kg (dry weight basis).

Residues of omethoate in barley hay were (n = 4) < 0.012, < 0.014 (2), and 0.05 mg/kg (dry weight basis).

Residues in accordance with GAP for livestock dietary burden calculation were (n = 4) < 0.024, < 0.028 (2), and 0.19 mg/kg (dry weight basis).

Residues of dimethoate in barley straw were (n = 7) < 0.01 (6), and 0.05 mg/kg (as received basis).

Residues of omethoate in barley straw were (n = 7) < 0.01 (7) mg/kg.

Residues in accordance with GAP for livestock dietary burden calculation were (n = 7) < 0.02 (6), and 0.06 mg/kg (as received basis) or < 0.022 (6) and 0.067 mg/kg (adjusted for dry weight using the default dry matter content of 89% from the OECD livestock feed tables)).

Based on the hay data, the Meeting estimated a maximum residue level of 0.3 mg/kg (dw) for dimethoate in barley straw and fodder, dry.

The Meeting estimated at maximum residue level of 0.1 mg/kg (dw) for omethoate in barley straw and fodder, dry (based on the hay data).

The Meeting estimated a median and a highest residue of 0.028 and 0.19 mg/kg respectively for dietary burden calculations (dry weight basis) for barley hay.

The Meeting estimated a median and a highest residue of 0.022 and 0.067 mg/kg respectively for dietary burden calculations (dry weight basis) for barley straw.

The Meeting noted that the same GAP was authorized for oats in Estonia and considered that the residue potential was similar for barley and oats. The Meeting agreed that the estimations for barley straw and fodder could be extrapolated to oats, and estimated the following values:

- Maximum residue level for dimethoate in oat straw and fodder, dry: 0.3 mg/kg;
- Maximum residue level for omethoate in oat straw and fodder, dry: 0.1 mg/kg;
- Median and highest residue of 0.028 and 0.19 mg/kg respectively for oat hay;

- Median and highest residue of 0.022 and 0.067 mg/kg respectively for oat straw.

Wheat straw and fodder

Residue data for wheat hay and straw was available from the European trials.

Residues of dimethoate in wheat hay were (n = 8) < 0.012 (2), < 0.013, 0.023, 0.033, 0.21, 0.37, and 1.4 mg/kg (dry weight basis).

Residues of omethoate in wheat hay were (n = 8) < 0.011, < 0.012 (3), < 0.013, 0.022, 0.033, and 0.077 mg/kg (dry weight basis).

Residues for livestock dietary burden calculation were (n = 8) < 0.024 (2), < 0.026, 0.034, 0.055, 0.22, 0.40, and 1.5 mg/kg (dry weight basis)

Residues of dimethoate in wheat straw were (n = 32) < 0.01 (17), 0.01 (3), 0.02 (3), 0.04, 0.05 (3), 0.08, 0.19, 0.65, 0.68, and 0.83 mg/kg (as received basis).

Residues of omethoate in wheat straw were (n = 32) < 0.01 (27), 0.01, 0.02, 0.05, 0.06, and 0.074 mg/kg (as received basis).

Residues for livestock dietary burden calculation were (n = 32) < 0.02 (17), 0.02 (3), 0.03 (3), 0.05, 0.06 (3), 0.09, 0.24, 0.70, 0.71, and 0.90 mg/kg (as received basis) or < 0.023 (17), 0.023 (3), 0.034 (3), 0.057, 0.068 (3), 0.10, 0.27, 0.80, 0.81, and 1.0 mg/kg (adjusted for dry weight using the default dry matter content of 88% from the OECD livestock feed tables).

Based on the hay data, the Meeting estimated maximum residue levels of 3 and 0.15 mg/kg for dimethoate and omethoate respectively in wheat straw and fodder, dry.

The Meeting estimated a median and a highest residue of 0.0455 and 1.5 mg/kg (dry weight basis) for wheat hay.

The Meeting estimated a median and a highest residue of 0.023 and 1.0 mg/kg for wheat straw (dry weight basis).

The Meeting noted that the same GAP was authorized for rye and triticale in the Czech Republic and Estonia, considered that the residue potential was similar for wheat, rye and triticale, and agreed that the estimations for wheat straw and fodder could be extrapolated to rye and triticale.

The Meeting estimated maximum residue levels of 3 mg/kg for dimethoate in rye straw and fodder, dry and triticale straw and fodder, dry.

The Meeting estimated maximum residue levels of 0.15 mg/kg for omethoate in rye straw and fodder, dry and triticale straw and fodder, dry.

The Meeting estimated a median and a highest residue of 0.0455 and 1.5 mg/kg (dry weight basis) for triticale hay.

The Meeting estimated a median and a highest residue of 0.023 and 1.0 mg/kg for rye straw and triticale straw.

Bean forage

Residue data in soya bean, mung bean, and navy bean forage was available from the Australian trials.

The GAP for beans except soya beans is an unspecified number of applications at a rate of 0.32 kg ai/ha and 14-day intervals, with a grazing interval of 14 days.

The GAP for soya beans is an unspecified number of applications at a rate of 0.136 kg ai/ha and 14-day intervals, with a PHI of 14 days for both grazing and harvest.

The data for forage did not match GAP, as samples were only collected at intervals of 0 and 7 days after application.

The Meeting therefore did not estimate median or highest residues for bean forage.

Fate of residues during processing

High temperature hydrolysis

Under conditions simulating pasteurization (90 °C/pH 4/20 minutes), no significant hydrolysis of dimethoate or omethoate occurred (> 90% of AR remained as parent). Under conditions simulating baking/boiling/brewing (100 °C/pH 5/60 minutes) and sterilization (120 °C/pH 6/20 minutes), significant hydrolysis, mainly of the O-methyl groups took place, with 28%/36% of AR as the O-desmethyl metabolite for dimethoate/omethoate after baking/boiling/brewing, and 60%/63% after sterilization). No conversion from dimethoate to omethoate was observed under any of the conditions.

Processing data for oranges, olives, cabbage and wheat were available to the Meeting. Estimated processing factors are summarized in the tables below.

The Meeting noted that separate residue definitions have been established for enforcement for dimethoate and omethoate, and therefore, determination of the levels of these components in processed commodities is required in order to estimate maximum residue levels for processed commodities. The Meeting noted that that no conversion of dimethoate to omethoate was observed during the high temperature hydrolysis study. The Meeting further noted that, in the processing studies available to the Meeting, both dimethoate and omethoate were present in the raw agricultural commodities and omethoate generally transferred into processed commodities to a lesser extent than dimethoate, further indicating that conversion of dimethoate to omethoate during processing is not significant in the processed commodities considered by the present Meeting. Therefore, separate processing factors for dimethoate and omethoate have been calculated for each processed commodity, with the levels of dimethoate and omethoate (and the summed levels for dietary risk assessment) then being calculated for the processed commodities individually from each supervised residue trial. This in turn allows the estimation of processed commodity maximum residue levels for dimethoate and omethoate where required, and STMR and HR values.

Oranges

Table 4 Processing factors for oranges

Processed commodity	Dimethoate	Omethoate
Juice	0.14	0.20
Dry pulp	2.1	1.6
Molasses	5.8	5.9
Orange oil	0.20	< 0.07

Table 5 Calculation of residues of dimethoate and omethoate in orange juice

Raw orange dimethoate residue (mg/kg)	PF	Orange juice dimethoate residue (mg/kg)	Raw orange omethoate residue (mg/kg)	PF	Orange juice omethoate residue (mg/kg)
0.66	0.14	0.092	0.004	0.20	0.0008
0.59		0.083	0.003		0.0006
0.67		0.094	0.005		0.001
0.60		0.084	0.004		0.0008
0.51		0.071	0.003		0.0006
0.63		0.088	0.003		0.0006

Table 6 Calculation of residues of dimethoate and omethoate in orange dried pulp

Raw orange dimethoate residue (mg/kg)	PF	Orange dried pulp dimethoate residue (mg/kg)	Raw orange omethoate residue (mg/kg)	PF	Orange dried pulp omethoate residue (mg/kg)	DM + OM (mg/kg)
0.66	2.1	1.39	0.004	1.6	0.0064	1.39
0.59		1.24	0.003		0.0048	1.24
0.67		1.41	0.005		0.008	1.42
0.60		1.26	0.004		0.0064	1.27
0.51		1.07	0.003		0.0048	1.08
0.63		1.32	0.003		0.0048	1.33
					Median:	1.30

The Meeting estimated a maximum residue level of 4 mg/kg for dimethoate in citrus pulp, dry.

The Meeting estimated a maximum residue level of 0.02 mg/kg for omethoate in citrus pulp, dry.

The Meeting estimated a median residue of 1.3 mg/kg for citrus pulp, dry for livestock dietary burden calculation.

Table 7 Calculation of residues of dimethoate and omethoate in orange oil

Raw orange dimethoate residue (mg/kg)	PF	Orange oil dimethoate residue (mg/kg)	Raw orange omethoate residue (mg/kg)	PF	Orange oil omethoate residue (mg/kg)
0.66	0.20	0.13	0.004	0.07	0.00028
0.59		0.12	0.003		0.00021
0.67		0.13	0.005		0.00035
0.60		0.12	0.004		0.00028
0.51		0.10	0.003		0.00021
0.63		0.13	0.003		0.00021

Olives

Table 8 Processing factors for olives

Processed commodity	Dimethoate		Omethoate	
	Processing factors	Best estimate PF	Processing factors	Best estimate PF
Crude (hot pressed) oil	0.25, 0.25, 0.32, 0.33, 0.42, 0.44, 1.5	0.33	0, 0, 0, 0, < 0.01, < 0.02, < 0.04, < 0.05	0.005
Virgin (cold pressed) oil	0.5, 1.5	1.0	< 0.02, < 0.02	0.02
Refined oil	0.007, < 0.03, < 0.05, 0.12, 0.25, 0.26, 0.29, < 0.50	0.185	0, 0, 0, 0, < 0.01, 0.02, < 0.02, < 0.04, < 0.05	0.01
Canned olives in brine (sterilized)	0.03, 0.05, 0.065, 0.07, 0.08, 0.08, 0.11, 0.12, 0.31	0.08	0, 0, 0.007, 0.02, 0.02, 0.02, 0.03, < 0.04, 0.04, 0.19	0.02
Canned olives in brine (not sterilized)	0.12, 0.24, 0.28, 0.28, 0.31, 0.37, 0.74, 0.92,	0.37	< 0.02, 0.02, < 0.05, 0.05, 0.07, 0.08, 0.14,	0.11

Processed commodity	Dimethoate		Omethoate	
	Processing factors	Best estimate PF	Processing factors	Best estimate PF
	1.0, 1.1, 1.3		0.15, 0.73, 0.84, 0.89, 1.1	

Table 9 Calculation of residues of dimethoate and omethoate in canned (table) olives

Raw olive dimethoate residue (mg/kg) [a]	PF	Canned (table) olives dimethoate residue (mg/kg)	Raw olive omethoate residue (mg/kg) [a]	PF	Canned (table) olives omethoate residue (mg/kg)
1.2	0.37	0.44	0.35	0.11	0.039
0.41		0.15	0.35		0.039
0.01		0.0037	0.01		0.0011
0.63		0.23	0.2		0.022
0.55		0.20	0.24		0.026
0.11		0.041	0.29		0.032
0.11		0.041	0.3		0.033
0.06		0.022	0.69		0.076
0.07		0.026	0.28		0.031
0.02		0.0074	0.15		0.017
0.01		0.0037	0.29		0.032
0.01		0.0037	0.21		0.023
0.06		0.022	0.22		0.024
0.12		0.044	0.07		0.0077
0.22		0.081	0.28		0.031
0.74		0.27	0.78		0.086
0.34		0.13	0.5		0.055
0.76		0.28	0.26		0.029
1.5		0.56	0.36		0.040
1.5		0.56	0.88		0.097
0.01		0.0037	0.11		0.012
0.01		0.0037	0.01		0.0011
0.04		0.015	0.27		0.030
0.01		0.0037	0.05		0.0055
0.05		0.019	0.34		0.037
0.01		0.0037	0.03		0.0033
0.01		0.0037	0.21		0.023
0.04		0.015	0.27		0.030

[a] Residues below the LOQ in the RAC are shown at the LOQ for calculation purposes.

Table 10 Calculation of residues of dimethoate and omethoate in olive oil, virgin

Raw olive dimethoate residue (mg/kg) [a]	PF	Olive oil dimethoate residue (mg/kg)	Raw olive omethoate residue (mg/kg) [a]	PF	Olive oil omethoate residue (mg/kg)
1.2	1.0	1.2	0.35	0.02	0.00077
0.41		0.41	0.35		0.00077
0.01		0.01	0.01		0.000022
0.63		0.63	0.2		0.00044
0.55		0.55	0.24		0.00053
0.11		0.11	0.29		0.00064
0.11		0.11	0.3		0.00066
0.06		0.06	0.69		0.0015
0.07		0.07	0.28		0.00062
0.02		0.02	0.15		0.00033
< 0.01		< 0.01	0.29		0.00064
0.01		0.01	0.21		0.00046
0.06		0.06	0.22		0.00048
0.12		0.12	0.07		0.00015
0.22		0.22	0.28		0.00062
0.74		0.74	0.78		0.0017
0.34		0.34	0.5		0.0011
0.76		0.76	0.26		0.00057
1.5		1.5	0.36		0.00079
1.5		1.5	0.88		0.0019
0.01		0.01	0.11		0.00024
0.01		0.01	0.01		0.000022
0.04		0.04	0.27		0.00059
0.01		0.01	0.05		0.00011
0.05		0.05	0.34		0.00075
0.01		0.01	0.03		0.000066
0.01		0.01	0.21		0.00046
0.04		0.04	0.27		0.00059

[a] Residues below the LOQ in the RAC are shown at the LOQ for calculation purposes.

The Meeting estimated a maximum residue level of 3 mg/kg for dimethoate in olive oil, virgin.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg for omethoate in olive oil, virgin.

Table 11 Calculation of residues of dimethoate and omethoate in olive oil, refined

Raw olive dimethoate residue (mg/kg)[a]	PF	Olive oil dimethoate residue (mg/kg)	Raw olive omethoate residue (mg/kg)[a]	PF	Olive oil omethoate residue (mg/kg)
1.2	0.185	0.22	0.35	0.01	0.0035

Raw olive dimethoate residue (mg/kg)[a]	PF	Olive oil dimethoate residue (mg/kg)	Raw olive omethoate residue (mg/kg)[a]	PF	Olive oil omethoate residue (mg/kg)
0.41		0.076	0.35		0.0035
0.01		0.002	0.01		0.0001
0.63		0.12	0.2		0.002
0.55		0.10	0.24		0.0024
0.11		0.020	0.29		0.0029
0.11		0.020	0.3		0.003
0.06		0.011	0.69		0.0069
0.07		0.013	0.28		0.0028
0.02		0.004	0.15		0.0015
< 0.01		0.002	0.29		0.0029
0.01		0.002	0.21		0.0021
0.06		0.011	0.22		0.0022
0.12		0.022	0.07		0.0007
0.22		0.041	0.28		0.0028
0.74		0.14	0.78		0.0078
0.34		0.063	0.5		0.005
0.76		0.14	0.26		0.0026
1.5		0.28	0.36		0.0036
1.5		0.28	0.88		0.0088
0.01		0.002	0.11		0.0011
0.01		0.002	0.01		0.0001
0.04		0.007	0.27		0.0027
0.01		0.002	0.05		0.0005
0.05		0.009	0.34		0.0034
0.01		0.002	0.03		0.0003
0.01		0.002	0.21		0.0021
0.04		0.007	0.27		0.0027

[a] Residues below the LOQ in the RAC are shown at the LOQ for calculation purposes.

Wheat

Table 12 Processing factors for wheat

Processed commodity	Dimethoate		Omethoate	
	Processing factors	Best estimate PF	Processing factors	Best estimate PF
Wholemeal flour	0.22, 0.62, 0.70, 3.2	0.66	< 0.3, < 0.5, 1.0	0.5
White flour	0.06, 0.10, 0.31, 0.80	0.21	< 0.3, < 0.5, < 0.5	0.5
Bran	1.4, 4.0, 4.8, 15	4.4	1.3, 3.5, 5.0	3.5
Wheat germ	0.83, 2.8, 3.0, 9.6	2.9	0.67, 2.0, 4.0	2.0
Wholemeal bread	0.72, 1.8, 1.9, 5.4	1.85	0.33, 1.5, 2.5	1.5

Table 13 Calculation of residues of dimethoate and omethoate in wheat bran

Raw wheat dimethoate residue (mg/kg)^	PF	Wheat bran dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wheat bran omethoate residue (mg/kg)	DM + OM (mg/kg)
< 0.001	4.4	< 0.0044	< 0.001	3.5	< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
0.002		0.0088	< 0.001		< 0.0035	0.0123
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
0.002		0.0088	< 0.001		< 0.0035	0.0123
< 0.001		< 0.0044	< 0.001		< 0.0035	< 0.0079
0.005		0.022	< 0.001		< 0.0035	0.0255
< 0.001		< 0.0044	0.001		0.0035	0.0079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.044
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
< 0.01		< 0.044	< 0.01		< 0.035	< 0.079
0.01		0.044	< 0.01		< 0.035	< 0.079
					Median	0.05225

Table 14 Calculation of residues of dimethoate and omethoate in wheat germ

Raw wheat dimethoate residue (mg/kg)^	PF	Wheat germ dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wheat germ omethoate residue (mg/kg)
< 0.001	2.9	< 0.0029	< 0.001	2.0	< 0.002

Raw wheat dimethoate residue (mg/kg)^	PF	Wheat germ dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wheat germ omethoate residue (mg/kg)
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
0.002		0.0058	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
0.002		0.0058	< 0.001		< 0.002
< 0.001		< 0.0029	< 0.001		< 0.002
0.005		0.0145	< 0.001		< 0.002
< 0.001		< 0.0029	0.001		0.002
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
< 0.01		< 0.029	< 0.01		< 0.02
0.01		0.029	< 0.01		< 0.02

Table 15 Calculation of residues of dimethoate and omethoate in wheat wholemeal bread

Raw wheat dimethoate residue (mg/kg)^	PF	Wholemeal bread dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wholemeal bread omethoate residue (mg/kg)
< 0.001	1.85	< 0.00185	< 0.001	1.5	< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015

Raw wheat dimethoate residue (mg/kg)^	PF	Wholemeal bread dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wholemeal bread omethoate residue (mg/kg)
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
0.002		0.0037	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
0.002		0.0037	< 0.001		< 0.0015
< 0.001		< 0.00185	< 0.001		< 0.0015
0.005		0.00925	< 0.001		< 0.0015
< 0.001		< 0.00185	0.001		0.0015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
< 0.01		< 0.0185	< 0.01		< 0.015
0.01		0.0185	< 0.01		< 0.015

Table 16 Calculation of residues of dimethoate and omethoate in wholemeal flour

Raw wheat dimethoate residue (mg/kg)^	PF	Wholemeal flour dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wholemeal flour omethoate residue (mg/kg)
< 0.001	0.66	< 0.00066	< 0.001	0.5	< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005

Raw wheat dimethoate residue (mg/kg)^	PF	Wholemeal flour dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	Wholemeal flour omethoate residue (mg/kg)
< 0.001		< 0.00066	< 0.001		< 0.0005
0.002		0.0013	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
0.002		0.0013	< 0.001		< 0.0005
< 0.001		< 0.00066	< 0.001		< 0.0005
0.005		0.0033	< 0.001		< 0.0005
< 0.001		< 0.00066	0.001		0.0005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
< 0.01		< 0.0066	< 0.01		< 0.005
0.01		0.0066	< 0.01		< 0.005

Table 17 Calculation of residues of dimethoate and omethoate in white flour

Raw wheat dimethoate residue (mg/kg)^	PF	White flour dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	White flour omethoate residue (mg/kg)
< 0.001	0.21	< 0.00021	< 0.001	0.5	< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
0.002		0.00042	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005

Raw wheat dimethoate residue (mg/kg)^	PF	White flour dimethoate residue (mg/kg)	Raw wheat omethoate residue (mg/kg)^	PF	White flour omethoate residue (mg/kg)
< 0.001		< 0.00021	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
0.002		0.00042	< 0.001		< 0.0005
< 0.001		< 0.00021	< 0.001		< 0.0005
0.005		0.0010	< 0.001		< 0.0005
< 0.001		< 0.00021	0.001		0.0005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
< 0.01		< 0.0021	< 0.01		< 0.005
0.01		0.0021	< 0.01		< 0.005

The Meeting estimated a maximum residue level of 0.1 mg/kg for dimethoate in wheat bran.

The Meeting estimated a maximum residue level of 0.09 mg/kg for omethoate in wheat bran.

The Meeting estimated a median residue of 0.05225 mg/kg for wheat bran for livestock dietary burden calculations.

The Meeting estimated a maximum residue level of 0.07 mg/kg for dimethoate in wheat germ.

The Meeting estimated a maximum residue level of 0.05 mg/kg for omethoate in wheat germ.

The Meeting estimated a maximum residue level of 0.05 mg/kg for dimethoate in bread and other cooked cereal products.

The Meeting estimated a maximum residue level of 0.04 mg/kg for omethoate in bread and other cooked cereal products.

Farm animal dietary burden

Farm animal feeding studies in lactating cattle and laying hens were provided to the Meeting.

Lactating cattle

Lactating Holstein dairy cattle were dosed daily with dimethoate for 28 days at 1, 3.4, 10.1 or 33.2 ppm. Composite morning and afternoon milk samples were collected daily, along with skim milk and

cream on days 13 and 28. The animals not required for the depuration phase were slaughtered on day 28 (7.5–12 hours after the final dose) and samples of round, flank and loin muscle, liver, kidney, and omental, subcutaneous and perirenal fat were collected. Three highest dose group animals were retained for the depuration phase, with one sacrificed on each of days 31, 35, and 42 (3, 7 and 14 days depuration respectively).

No residues of dimethoate were found in milk (either control or any of the treated samples) at levels above the LOQ. Low levels of omethoate (maximum 0.019 mg/kg) were found, only for some milk samples from the highest dose group. Omethoate residues in skim milk and cream were very similar, indicating no significant preferential partitioning. In muscle, again, no residues of dimethoate were found above the LOQ, while some low level residues of omethoate (up to 0.0051 mg/kg) were found only for the highest dose group. In liver, no residues were found above the LOQ for dimethoate, with low level detections of omethoate in the 10.1 and 33.2 ppm groups. For kidney, there was a single low level detection of omethoate for the highest dose group, and no detections of dimethoate. For fat, there were some low level residues of both dimethoate and omethoate, without a clear relationship between dose and residue level. The depuration data showed that clearance was rapid, with no detections above the LOQ.

Laying hens

Laying white Leghorn hens were dosed daily with dimethoate for 28 days at 0.15, 0.40, 1.2 or 4.0 ppm. Composite morning and afternoon egg samples were collected daily. The birds not required for the depuration phase were slaughtered on day 28 (approximately 24 hours after the final dose) and samples of thigh and breast muscle, liver, and subcutaneous and abdominal fat were collected. Ten of the highest dose group birds were retained for the depuration phase, with three or four birds sacrificed on each of days 31, 35 and 42 (3, 7 and 14 days depuration respectively).

No residues of dimethoate or omethoate were found above the LOQ in any of the egg or tissue samples.

Livestock dietary burden

Dietary burden calculations for cattle and poultry are provided below. The dietary burdens were estimated using the 2018 OECD Feed diets listed in Appendix XIV Electronic attachments to the 2016 edition of the FAO manual.

Table 18 Summary of livestock dietary burden (ppm dimethoate)

	USA-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.39	0.18	0.51	0.15	1.5 [a]	0.50 [b]	0.038	0.038
Dairy cattle	0.50	0.18	0.74	0.36	1.5 [c]	0.48 [d]	0.03	0.030
Broiler hens	0.037	0.037	0.055	0.045	0.093	0.093	0.003	0.003
Laying hens	0.037	0.037	0.22 [e]	0.054	0.093	0.093 [f]	0.018	0.018

[a] Highest maximum dietary burden for beef cattle suitable for estimation of MRLs for mammalian meat and offal.

[b] Highest mean dietary burden for beef cattle suitable for estimation of STMRs for mammalian meat and offal.

[c] Highest maximum dietary burden for dairy cattle suitable for estimation of MRLs for milk.

[d] Highest mean dietary burden for dairy cattle suitable for estimation of STMRs for milk.

[e] Highest maximum dietary burden for broiler and layer poultry suitable for estimation of MRLs for poultry meat, offal and eggs.

[f] Highest mean dietary burden for broiler and layer poultry suitable for estimation of STMRs for poultry meat, offal and eggs.

Animal commodity maximum residue levels

Mammals

The maximum dietary burden for dairy cattle was 1.5 ppm, while the highest mean burden was 0.48 ppm.

In a lactating cow feeding study, when cattle were dosed with dimethoate daily for 28 days at levels up to 33 ppm, no residues of dimethoate were found above the LOQ (0.001 mg/kg) in milk. Low levels of omethoate residue were found above the LOQ (at 0.019 mg/kg maximum) only in animals from the 33 ppm feeding group.

The Meeting therefore estimated maximum residue levels of 0.001(*) mg/kg for both dimethoate and omethoate in milk.

The maximum dietary burden for beef cattle was 1.5 ppm, while the highest mean burden was 0.50 ppm.

In liver and kidney, residues were below the LOQ (0.001 mg/kg) in all samples from feeding levels bracketing the maximum dietary burden (1 and 3.4 ppm). Some low level residues of omethoate were found at the 10 and 33 ppm feeding levels in liver and kidney.

The Meeting therefore estimated maximum residue levels of 0.001(*) mg/kg for both dimethoate and omethoate in mammalian edible offal.

In muscle, residues were below the LOQ (0.001 mg/kg) in all samples from the 3.4 ppm feeding level, with low level detections of omethoate in the 33 ppm feeding level samples.

The Meeting therefore estimated maximum residue levels of 0.001(*) mg/kg for both dimethoate and omethoate in mammalian meat.

In fat, no residues of omethoate were found above the LOQ (0.001 mg/kg) in feeding levels bracketing the maximum dietary burden (1 and 3.4 ppm dose groups), with some detections for highest dose groups. Residues of dimethoate were observed in subcutaneous, perirenal and omental fat, with levels highest in perirenal fat. In the 1 ppm feeding group, the highest residue was 0.0268 mg/kg, with the mean at 0.011 mg/kg. For the 3.4 ppm feeding group, the highest residue was 0.0014 mg/kg, while the mean was < 0.001 mg/kg.

As a conservative estimate, the residue levels from the 1 ppm dose group were used to estimate maximum residue levels for fat.

Scaling for the maximum dietary burden of 1.5 ppm, the estimated maximum dimethoate residue in fat is 1.5 × 0.0247 mg/kg = 0.037 mg/kg. The Meeting estimated a maximum residue level of 0.04 mg/kg for dimethoate in mammalian fats.

As the residues of omethoate in all fat samples were below the LOQ (0.001 mg/kg) for the 1 and 3.4 ppm feeding levels, the Meeting estimated a maximum residue level of 0.001(*) mg/kg for omethoate in mammalian fats.

Poultry

The maximum dietary burden for poultry for both meat and egg production was 0.22 ppm, while the highest mean dietary burden was 0.093 ppm.

In a poultry feeding study, when hens were fed dimethoate daily for 28 days at up to 4.0 ppm, no residues of dimethoate or omethoate were found above the LOQ (0.001 mg/kg) in any of the egg or tissue samples.

The Meeting therefore estimated maximum residue levels of 0.001(*) mg/kg for both dimethoate and omethoate in poulty meat, poultry fats, poultry, edible offal of, and eggs.

RECOMMENDATIONS

The residue definition for compliance with the MRL in plant and animal commodities is: *dimethoate and omethoate (measured and reported separately).*

The Meeting was unable to recommend a residue definition for dietary risk assessment.

The residue is not fat-soluble.

Table 19 Residue levels suitable for establishing maximum residue limits and for IEDI and IESTI assessments

CCN	Commodity name	Recommended maximum residue level, mg/kg			
		Dimethoate		Omethoate	
		New	Previous	New	Previous
VS 0620	Artichoke, globe	W	0.05		
VS 0621	Asparagus	W	0.05(*)		
GC 0640	Barley	W	2		
VB 0402	Brussels sprouts	W	0.2		
VB 0403	Cabbage, Savoy	W	0.05(*)		
MO 0812	Cattle, Edible offal of	W	0.05(*)		
VB 0404	Cauliflower	W	0.2		
VS 0624	Celery	W	0.5		
FS 0013	Cherries	W	2		
FC 0001	Citrus fruits	W	5		
PE 0112	Eggs	W	0.05(*)		
VL 0482	Lettuce, Head	W	0.3		
MF 0100	Mammalian fats (except milk fats)	W	0.05(*)		
FI 0345	Mango	W	1		
MM 0096	Meat of cattle, goats, horses, pigs and sheep	W	0.05(*)		
ML 0107	Milk of cattle, goats and sheep	W	0.05(*)		
FP 0230	Pear	W	1		
VP 0063	Peas (pods and succulent=immature seeds)	W	1		
HS 0444	Peppers Chili, dried	W	3		
VO 0445	Peppers, sweet (including pimento or pimiento)	W	0.5		
VR 0589	Potato	W	0.05		
PF 0111	Poultry fats	W	0.05(*)		
PM 0110	Poultry meat	W	0.05(*)		
PO 0111	Poultry, edible offal of	W	0.05(*)		
MO 0822	Sheep, edible offal of	W	0.05(*)		
HS 0191	Spices, fruits and berries	W	0.5	W	0.01
HS 0193	Spices, roots and rhizomes	W	0.1(*)	W	0.05

CCN	Commodity name	Recommended maximum residue level, mg/kg			
		Dimethoate		Omethoate	
		New	Previous	New	Previous
HS 0190	Spices, seeds	W	5		
VR 0596	Sugar beet	W	0.05		
FT 0305	Table olives	W	0.5		
VL 0506	Turnip greens	W	1		
VR 0506	Turnip, Garden	W	0.1		
GC 0654	Wheat	W	0.05		
AS 0654	Wheat straw and fodder, dry	W	1		

Table 20 Median and highest residues (where required) values for livestock feeds (for livestock dietary burden)

CCN	Commodity	Median residue (mg/kg)	Highest residue (mg/kg)
GC 0640	Barley	0.002	
AS 0640	Barley straw and fodder, dry	Hay 0.028 (l, dw) Straw 0.022 (l, dw)	Hay 0.19 (l, dw) Straw 0.067 (l, dw)
AB 0001	Citrus pulp, dry	1.3	
VR 0577	Carrot culls	0.0035	0.016
VD 2065	Dry beans	0.10	
GC 0647	Oats	0.002	
AS 0647	Oat straw and fodder, dry	Hay 0.028 (l, dw) Straw 0.022 (l, dw)	Hay 0.19 (l, dw) Straw 0.067 (l, dw)
AV 0596	Sugar beet leaves or tops (dry)	0.13	0.40
GC 0650	Rye	0.013	
AS 0650	Rye straw and fodder, dry	Hay 0.0455 (l, dw) Straw 0.023 (l, dw)	Hay 1.5 (l, dw) Straw 1.0 (l, dw)
GC 0653	Triticale	0.013	
AS 0653	Triticale straw and fodder, dry	Hay 0.0455 (l, dw) Straw 0.023 (l, dw)	Hay 1.5 (l, dw) Straw 1.0 (l, dw)
GC 0654	Wheat	0.013	
CF 0654	Wheat bran, processed	0.05225	
AS 0654	Wheat straw and fodder, dry	Hay 0.0455 (l, dw) Straw 0.023 (l, dw)	Hay 1.5 (l, dw) Straw 1.0 (l, dw)

l = livestock feed, dw = dry weight

DIETARY RISK ASSESSMENT

The WHO panel recommended an ADI of 0–0.001 mg/kg bw/day and an ARfD of 0.02 mg/kg bw for dimethoate.

The WHO panel was unable to recommend an ADI or an ARfD for omethoate, due to concerns regarding its genotoxicity.

As a result of concerns relating to the genotoxicity of omethoate and other related metabolites, a conclusion was unable to be reached on a residue definition for dietary risk assessment. Long-term and acute dietary risk assessments could not be conducted.

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