Australian Government

Australian Pesticides and Veterinary Medicines Authority

DIMETHOATE

RESIDUES AND DIETARY RISK ASSESMENT REPORT

APPENDIXES 2, 3, 4, 5 AND 6

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LIST OF APPENDIXES

APPENDIX 2: RESULTS OF PESTICIDE MONITORING PROGRAMS

The APVMA received the results of various residues monitoring programs and considered these results when assessing the chronic exposure to dimethoate. These results are not reproduced in full in this report.

These surveys included:

- Surveys of fresh fruit and vegetables purchased from Sydney markets between February 2002 and February 2005, comprising 1,497 samples of 48 different fresh fruit and vegetables that were analysed for various pesticides including dimethoate and omethoate.
- 336 samples comprising 44 different fresh fruit and vegetables purchased from Sydney markets in the period between November 2000 and June 2001 and analysed for various pesticides.
- Monitoring data for dimethoate in fruit and vegetables exported from a quarantine zone in north Queensland from November 1995 to June 1996 following an outbreak of papaya fruit fly.
- Monitoring data for dimethoate in a range of fruit and vegetables collected as part of the FreshTestTM residue monitoring program. The period of the survey was not indicated but is likely to pre-date 2004.
- A summary of residue survey data for grains for July 2005 to November 2010 were provided by the National Residue Survey (NRS).
- The NRS also provided limited survey data for dimethoate and omethoate on apples, blueberries, almonds, onions and pears for the period 2005 – 2010.

Overall, the available survey data show limited detections of dimethoate and omethoate, with only one case exceeding current MRLs. In particular for cereals, oilseeds and pulses, there were no detections above the limit of reporting. For blueberries sampled by the NRS, there were a significant number of detections of dimethoate and omethoate but the detections were generally between the limit of reporting and one fifth of the MRL. There were also detections of dimethoate in capsicums, mangoes, pawpaws, passionfruit, peaches and strawberries from Queensland, although none exceeded the relevant MRLs.

Food Standards Australia New Zealand regularly conducts the Australian Total Diet Study (ATDS) that regularly includes testing for pesticides in foods. The 20th ATDS included the analysis of dimethoate in 65 foods. Dimethoate residues were observed in 2 samples at low levels.

APPENDIX 3: METABOLISM DATA

No. 8340; Corden, M T; 14C-Dimethoate metabolism in potatoes; 28/6/2000

Dimethoate labelled with 14C in the methoxy group was mixed with an EC formulation containing non-labelled material. The mixture was applied to potatoes (BBCH 45–47) as a foliar spray at a target rate of 2× 340 g 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. At least two plants were taken and separated into foliage and tubers at each sampling interval. The 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 with acid, base or enzyme.

Total radioactive residues and concentrations of metabolites in foliage and tuber are summarised in Table 77.

Table 77: Metabolites observed in the foliage and tubers of potato plants

Component/ fraction	Foliage (mg equiv/kg)				Tuber (mg equiv/kg)				
	Day 0	Day 2	Day 7	Day 14	Day 0	Day 7	Day 14	Day 21	Day 28
Dimethoate	8.38	2.39	1.89	0.20	<0.01	<0.01	<0.01	<0.01	<0.01
Omethoate	0.73	0.32	0.72	0.12	<0.01	<0.01	<0.01	<0.01	<0.01
Desmethyl dimethoate	<0.02	<0.01	<0.01	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
O-desmethyl omethoate	0.39	0.03	0.21	0.04	<0.01	0.07	0.02	0.04	0.04
Dimethyldithiophosphate	0.04	<0.01	<0.01	0.06	<0.01	<0.01	<0.01	<0.01	<0.01
O-desmethyl omethoate carboxylic acid	<0.02	<0.01	<0.01	0.02	0.02	0.02	0.03	0.03	0.03
O-desmethyl N-desmethyl omethoate	0.22	0.18	0.24	0.11	0.23	0.12	0.09	0.09	0.10
Dimethoate carboxylic acid/des-O-methyl isodimethoate	0.10	<0.01	0.17	0.04	<0.01	<0.01	<0.01	<0.01	<0.01
Others	0.62	0.45	0.63	0.13	0.02	0.01	0.02	0.02	0.02
(Major other)	(0.28)	(0.19)	(0.36)	(0.06)	(0.02)	(0.01)	(0.01)	(0.01)	(0.01)
Water extractable	na	na	0.14	0.08	na	<0.01	<0.01	0.01	0.01
Protease extractable	0.53	0.41	0.25	0.22[a]	0.01	<0.01	<0.01	<0.01	<0.01
Base extractable	na	na	na	0.20[b]	na	na	0.01	0.01	0.01

Component/ fraction	Foliage (mg equiv/kg)				Tuber (mg equiv/kg)				
	Day 0	Day 2	Day 7	Day 14	Day 0	Day 7	Day 14	Day 21	Day 28
Unextractable	1.29	0.59	0.37	0.09	0.02	0.03	0.02	0.03	0.03
Total	**12.30**	**4.37**	**4.63**	**1.32**	**0.30**	**0.25**	**0.19**	**0.23**	**0.24**

NA Not applicable

a Mainly polar material.

b Composed of polar baseline material (0.13 mg/kg) and two minor unknowns representing 0.04 and 0.03 mg/kg.

Total radioactive residues were highest in foliage (12.30 mg/kg) and tuber (0.30 mg/kg) immediately after the second application (day 0). No dimethoate or omethoate was detected in the tubers at any time. Although dimethoate is a systemic insecticide 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.

Based on these results the following metabolic pathway was proposed for potatoes.

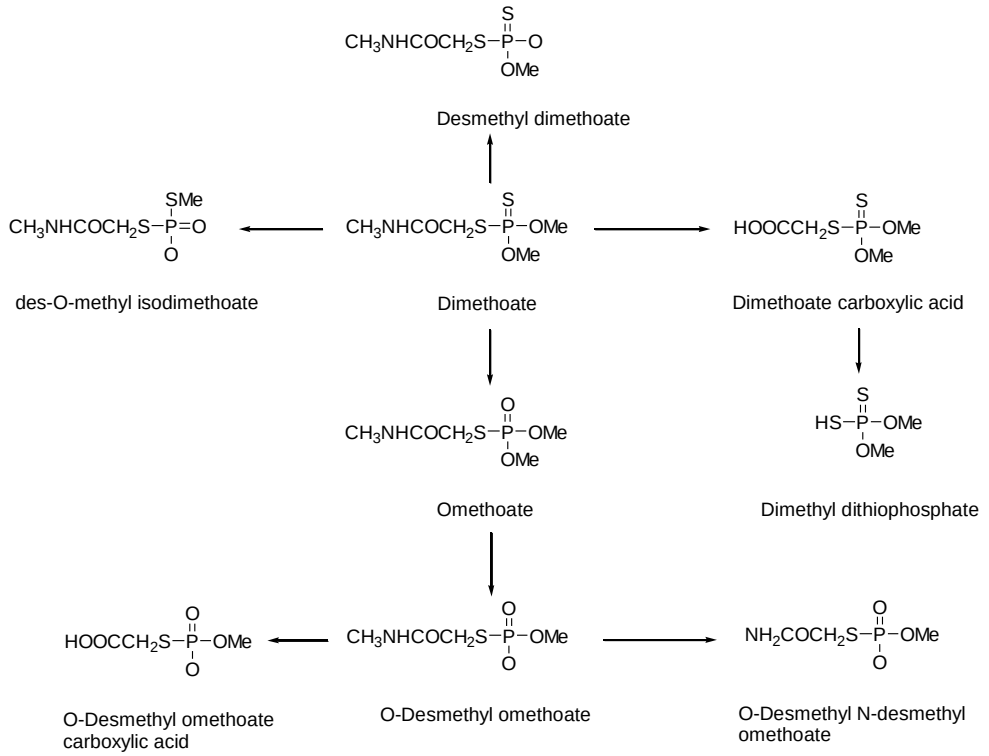

Figure 3: Proposed metabolic pathway for dimethoate in potatoes

No. 8341; Corden, M T; 14C-Dimethoate metabolism in potatoes: investigation of components A, G and K; 11/12/2001

The purpose of this study was to identify three unknown components characterised initially in the potato metabolism study:

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.

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 as shown below:

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.

Based on these results a revised metabolic pathway for dimethoate in potatoes was proposed, including the conjugate of hydroxy dimethoate. This is shown on the next page.

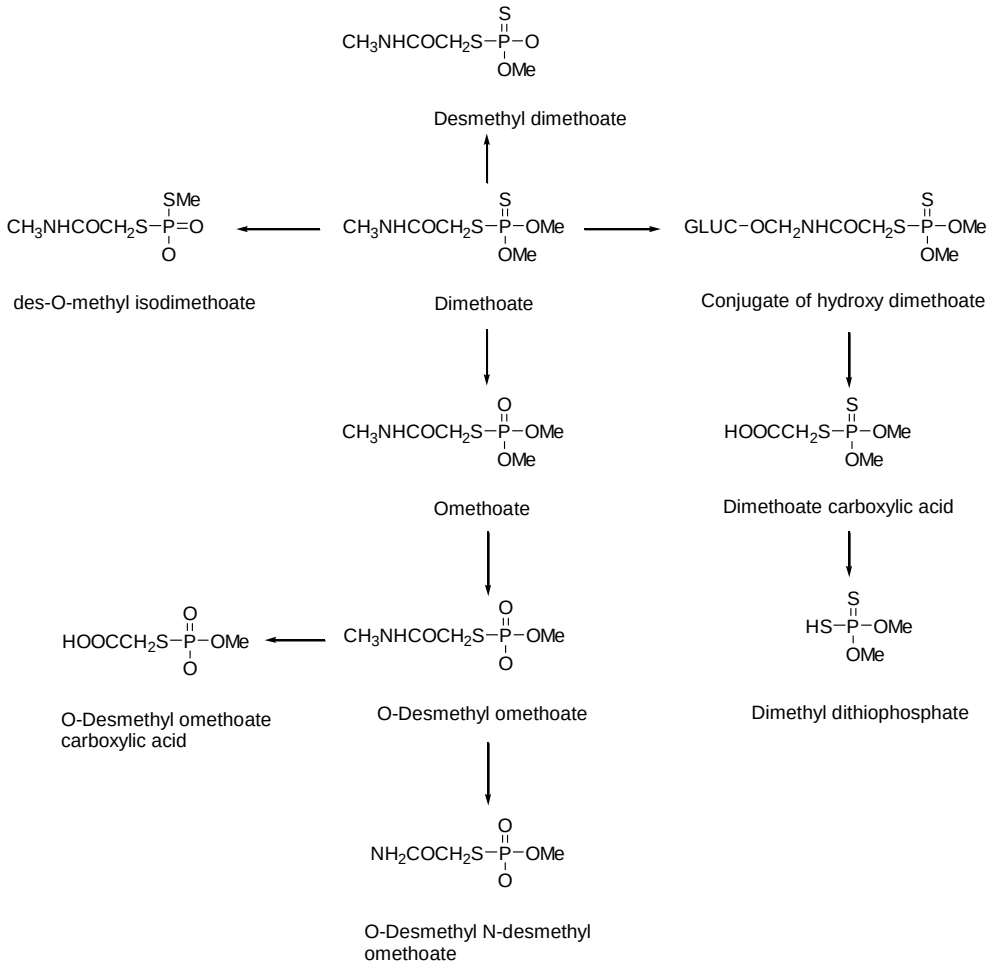

Figure 4: Revised metabolic pathway for dimethoate in potatoes

No. 8342; Corden, M T; 14C-Dimethoate metabolism in wheat; 10 December 2001

Dimethoate labelled with 14C in both methoxy groups was mixed with an EC formulation containing non-labelled material. The mixture was applied to wheat as a foliar spray at 680 g/ha at BBCH 24 followed by 400 g/ha at BBCH 69. The experiment was also performed using an exaggerated application rate (5×). 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). Depending on the growth stage of the plant, samples consisted of whole plant, ear, remaining plant, grain, hull or straw. Radioactivity was determined in homogenised samples. Samples were extracted with acetonitrile/water. Extracts containing significant radioactivity were analysed by HPLC and TLC with comparison against reference substances. Unextractable residues were further investigated by treating with acid, base or enzyme. Total radioactive residues and concentrations of metabolites (in mg/kg) in crop parts are summarised in Table 78 (1x rate).

Table 78: Concentration of metabolites (in mg equiv/kg) observed in wheat after 2 applications of dimethoate at 680 and 400 g ai/ha respectively

Component/ fraction	Day 0	Day 14	Day 26		Day 39 (before 2nd application)		Day 41 (after 2nd application)		Day 62			Day 73		
	Whole plant	Whole plant	Ear	Remain-ing plant	Ear	Remain-ing plant	Ear	Remain-ing plant	Grain	Hull	Straw	Grain	Hull	Straw
Dimethoate	29.00	0.07	<0.01	0.02	<0.01	<0.01	20.96	13.40	<0.01	1.21	0.40	<0.01	1.01	0.27
Omethoate	0.21	0.13	<0.01	0.03	<0.01	<0.01	0.43	0.42	<0.01	2.37	0.22	<0.01	1.85	0.28
Dimethyldithio-phosphate	<0.03	0.08	<0.01	<0.01	<0.01	0.02	0.11	0.32	<0.01	0.77	0.18	<0.01	0.71	0.16
Des-O-methyl isodimethoate	<0.03	0.49	<0.01	0.28	0.07	0.20	0.32	0.60	0.26	7.21	1.29	0.29	3.00	0.28
O-desmethyl N desmethyl omethoate	0.12	0.43	0.18	0.56	0.30	0.35	0.39	0.71	0.97	7.07	2.01	1.50	15.23	3.17
O-desmethyl omethoate carboxylic acid	<0.03	0.02	0.01	0.05	0.01	0.05	<0.02	<0.02	0.09	<0.02	0.30	0.15	1.01	0.28
Component A[a]	<0.03	0.01	<0.01	0.03	<0.01	0.02	<0.02	<0.02	0.28	0.44	0.28	0.49	2.43	0.53
Others[b]	0.35	0.15	<0.01	0.06	0.01	0.08	0.36	0.23	0.25	2.21	0.37	0.38	3.23	0.68
(Major other)	(0.12)	(0.03)	(<0.01)	(<0.01)	(<0.01)	(0.02)	(<0.07)	(0.11)	(0.08)	(0.58)	(0.08)	(0.04)	(<0.03)	(<0.01)
Base extractable	na	0.24	0.02	0.14	na	0.15	na	na	0.25	1.09	1.02	1.47	3.60	2.18
Unextractable	0.06	0.05	0.01	0.06	0.04	0.04	0.16	0.42	0.19	0.88	0.35	<0.01	1.62	<0.01
Total	**29.74**	**1.67**	**0.22**	**1.23**	**0.43**	**0.90**	**22.73**	**16.10**	**2.29**	**23.26**	**6.42**	**4.28**	**33.69**	**7.83**

a Component A was subsequently shown to be mainly O-desmethyl-N-desmethyl omethoate which was retained at the point of application during TLC.

b Individual components represent <10% of the total radioactive residue.

Dimethoate and omethoate were not detected in grain samples at harvest after application at the 1× rate. Low levels of dimethoate (0.10 mg/kg) and omethoate (0.06 mg/kg) were detected in day 73 grain samples after treatment at the exaggerated rate (5×). 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. Intermediates in brackets were not detected in the wheat study, but have been proposed based on the potato metabolism study.

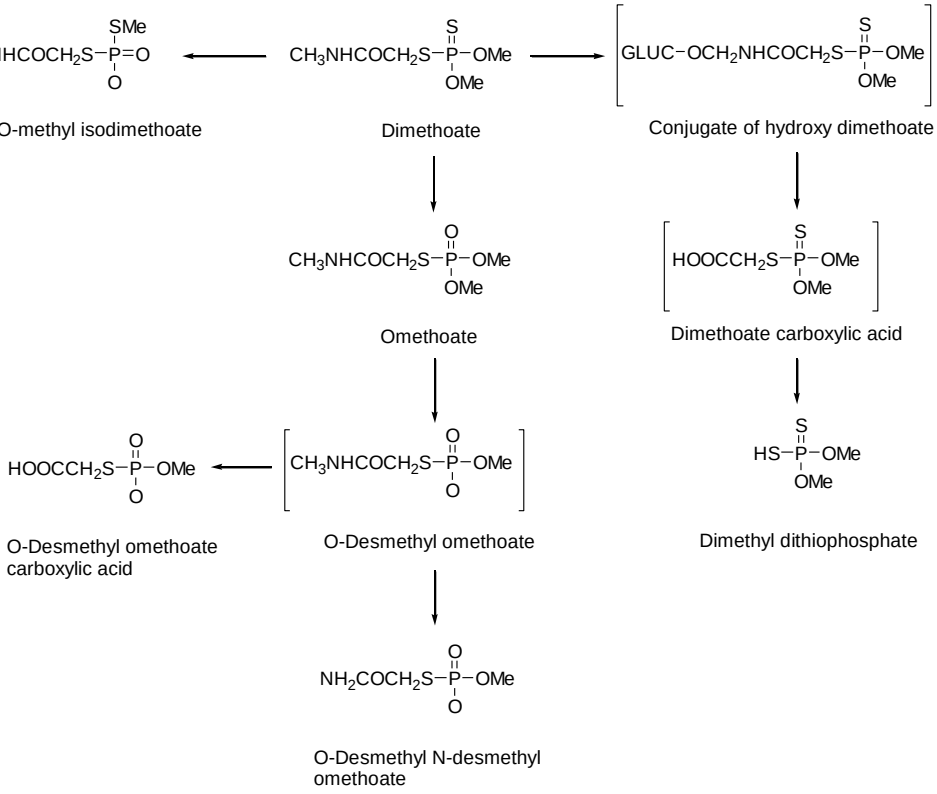

Figure 5: Proposed metabolic pathway for dimethoate in wheat

No. 8343; Pistel, F; Summary on and assessment of plant metabolism and residue behaviour; 13 March 1992

Only selected pages of this document were translated. The document referenced several literature studies but did not provide any data. It was reported that dimethoate in plants is degraded by oxidases, hydrolases and amidases. Final products are phosphate, C1-, C2- or N1- fractions being used by the plant for production of cell-related compounds. Hence, bound residues may be interpreted by formation of cell-related compounds.

No. 8344, Pistel, F; The metabolism of dimethoate in plants; 31 March 1993

This report summarises the available literature on the metabolism of dimethoate in plants. A large number of publications are available describing the behaviour in the following crops: sugar beet, potato, wheat, sorghum, maize, bean, pea, onion, cucumber, tomato, cotton, citrus, olives and rice.

The original publications were also provided; however, in many cases they were not in English.

Sugar beet

^{32}P-Dimethoate was applied to sugar beet plants at their fifth day after emergence (rate not stated). Only small amounts of radioactivity were recovered from the roots, mainly from vascular tissues. It was shown that dimethoate metabolises by formation of omethoate or by hydrolysis and formation of O-desmethyl dimethoate, O,O-dimethyl thiophosphoric acid, O,O-dimethyl phosphoric acid and phosphoric acid. Four additional metabolites were not identified. At long intervals between application and sampling the highest concentration of radioactivity in the leaves was associated with O,O-dimethyl thiophosphoric. The concentration of omethoate was initially far below that of dimethoate, but was at similar levels after 13–30 days. By 37 days after application neither dimethoate nor omethoate were found in leaves and roots.

Maize, cotton, peas and potatoes

After foliar application to these plants, ^{32}P-dimethoate was rapidly absorbed and degraded on the leaf surfaces as well as in the plant. The main metabolite was omethoate carboxylic acid (8.8 to 94.1% on leaf surfaces and 4.1 to 10.4% in the leaf). Additional degradation products in leaves were phosphoric acid (main product in peas), O,O-dimethyl thiophosphoric acid, O,O-dimethyl phosphoric acid and O-desmethyl dimethoate. Main metabolites in the plant were O-desmethl dimethoate (17.7 to 69.2%) as well as phosphoric acid, O,O-dimethyl phosphoric acid, O,O-dimethyl thiophosphoric acid and omethoate carboxylic acid. Omethoate was found in negligible amounts at a maximum of 0.7 to 1.7% of applied radioactivity. By 12 days after application, only traces of dimethoate and omethoate were detectable.

Wheat and sorghum grain

Grains were treated with a hexane solution of dimethoate at 2 and 10 ppm and stored at 20^{o}C in the dark. Samples were taken after 1, 4, 7, 11, 14 and 21 days and extracted by homogenisation in water/chloroform. Dimethoate was determined by gas chromatography, omethoate by an anticholinesterase technique. Degradation products were analysed by paper chromatography. Dimethoate residues were reported to degrade with a half-life of 3–4 days in wheat grain and 6–11 days in sorghum grain. Omethoate was only found in traces (0.1 µg—concentration not stated) after storage for 4 to 7 days. No omethoate was formed in studies with enzyme extracts. The following metabolites were formed at rates above the applied concentration: dimethoate carboxylic acid, O-desmethyl dimethoate, O-desmethyl dimethoate carboxylic acid and O,O-desmethyl thiophosphoric acid. Traces of O,O-dimethyl dithiophosphoric acid were also found.

In a further study on wheat grain, decarboxylation of dimethoate to O,O-dimethyl-phosphorodithioate was dependent on humidity and was only determined at water contents of 14 to 18%. The highest decarboxylase activity was associated with the embryo.

Beans

Four different application methods were used to investigate the metabolism of ^{32}P and ^{14}C-labelled dimethoate in beans:

- uptake via the stem of cut leaves
- stem injection
- uptake via roots
- foliar application.

Surface washes and plant extracts were analysed by thin layer or paper chromatography. Of 18 degradation products, the following 7 metabolites were identified: N-Desmethyl dimethoate, omethoate, dimethoate carboxylic acid, O-desmethyl dimethoate, O-desmethyl dimethoate carboxylic acid, O,O-dimethyl dithiophosphoric acid, O,O-dimethyl thiophosphoric acid. At 4 days after foliar application of ^{14}C-dimethoate, omethoate was present at a maximum of 0.81% of the applied radioactivity, with other metabolites between 0.03 and 0.72%. After surface application of ^{32}P-dimethoate, the maximum omethoate concentration was 4% of the applied radioactivity. After root application up to 10% (^{14}C-label) and 5% (^{32}P-label) of the applied radioactivity was found as omethoate. Degradation of dimethoate was most rapid after uptake by cut leaves, followed by stem injection and root application. Degradation was slowest after foliar application. The half-life of the total metabolite fraction was 1 to 4 days.

Glasshouse cucumbers

Residue samples were taken 3 to 12 days after application of pure or technical grade active ingredient (application rates were not stated). A maximum dimethoate residue of 1.3 mg/kg was detected 3 days after application. Traces of omethoate were detectable in all samplings with up to 0.2 mg/kg found at 7 days after application. After application in April residues degraded with a half-life of 4 days, with faster degradation in May and July due to vigorous plant growth.

Onions

Six days after application of ^{32}P-dimethoate, samples of onions were analysed for parent and omethoate. Dimethoate residues of 0.67 and 0.04% were found. Omethoate was found at up to 0.12 mg/kg, equivalent to about 18% of the total residue.

Tomatoes

A study on tomatoes used ^{32}P and ^{35}S dimethoate and also investigated various dimethoate analogues. Tomato plants (15–20 cm high) were cut above the root and put into aqueous solutions of the labelled substrates for 24 hours. Subsequently, the plants were grown in distilled water for 14 days. Soluble residues were extracted from leaves and their radioactivity determined. Besides dimethoate, the degradate omethoate could be detected by TLC reaching a maximum by 5 days after application. The half-lives of dimethoate and omethoate in the leaves were 3.2 and 9.3 days respectively. In contrast, in aqueous solution at pH 6 half-lives were 32.5 to 205 days, suggesting that, in plants, degradation is enzymatically catalysed.

Olives

Olive trees were extensively sprayed with ^{32}P-dimethoate (rate not stated). Olives were harvested up to 45 days after application. After extraction and paper chromatography, residues were separated and identified by paper chromatography and ion exchange. A maximum of 1.6 mg/kg of the metabolite omethoate was identified 9 days after application. Main metabolites were phosphoric acid and O-methyl phosphoric acid. The half-life of dimethoate was found to be in the order of <4 to 11 days. Processing of olives into edible olives or olive oil significantly reduced residues by 98–99% and 25–33% respectively.

Cotton

The metabolism of ^{32}P-dimethoate was investigated after uptake by cotton leaf cuttings. The radioactivity was extracted and metabolites separated by paper chromatography. Eleven metabolites were detected, of which 8 were identified. Besides dimethoate, the main metabolite was dimethoate carboxylic acid (up to 58% of applied radioactivity). Omethoate was only present at low levels (less than 6% of the applied radioactivity). Other metabolites were phosphoric acid, O,O-dimethyl-phosphoric acid, O-desmethyl dimethoate carboxylic acid, O,O-dimethyl thiophosphoric acid and O,O-dimethyl dithiophosphoric acid. The half-life of dimethoate was found to be 1.8 days.

Lemons

After application of ^{32}P-dimethoate to lemons the active ingredient was rapidly absorbed and acropetally translocated. Two degradation pathways were observed. An oxidative pathway resulted in the formation of omethoate, a hydrolytic pathway resulted in the formation of O,O-dimethyl phosphoric acid, phosphoric acid, O,O-dimethyl thiophosphoric acid and O-desmethyl dimethoate. Very high concentrations were found in leaves. Roots contained only very low amounts of ^{32}P substances.

Rice

A study is available on the in vitro metabolism of ^{32}P-dimethoate in a homogenate of rice leaves. Only metabolite O-desmethyl dimethoate was identified.

Conclusion

The report concluded that dimethoate acts systemically and is translocated within the plant after leaf application, stem injection and following uptake via the root system. The investigation on degradation and metabolism reveals uniform behaviour in all crops. The half-life of dimethoate and its metabolites in plants is in the range of 1 to 4 days (up to 11 days in stored crop protection trials in grain). Dimethoate and its transformation products undergo relatively rapid enzymatic degradation in plant tissues. In plant tissue, dimethoate is degraded by oxidases, hydrolases and amidases. Final products are phosphate, C1-, C2- or N1- fractions found in cell-related compounds. Generally, in most of the studies the toxicologically relevant metabolite omethoate was detected in low concentrations only. The following pathway was proposed for dimethoate in plants:

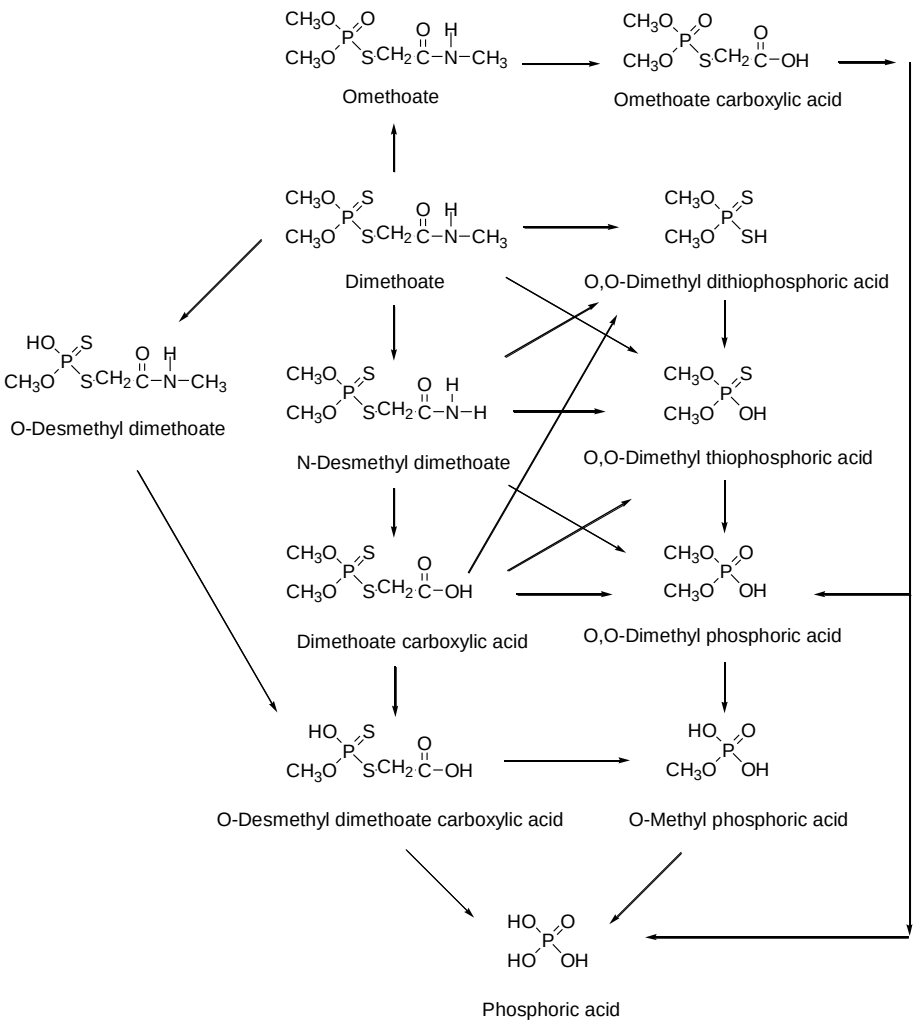

Figure 6: Metabolism of dimethoate in plants

No. 8345; Heidemann, A; The metabolism of dimethoate in plants; 2 February 1995

This report also reviews the available scientific literature on the metabolism of dimethoate in various plants. The studies are generally the same as those summarised in data No. 8344; the report draws the same conclusions and the same metabolic pathway has been proposed. Additional studies discussed in this review are outlined below.

Asparagus

Three trials on asparagus conducted in British Columbia in 1982 and 1983 involved 2 foliar applications of dimethoate at 0.25 to 1.12 kg ai/ha. Samples of fern and spears were taken from 6 hours to 66 days after treatment. In the first trial, residues of 12 mg/kg dimethoate plus omethoate were detected in the ferns 2 days after application. The highest omethoate residue of 1 mg/kg was detected after 10 days and accounted for about 50% of the total residue at that time. By 46 days after treatment only traces (<0.01 mg/kg) of dimethoate and omethoate were present. Similar results were obtained in the second trial, when 5.1 mg/kg dimethoate and 1.6 mg/kg omethoate were detected 5 days after application. After 39 days dimethoate was present at 0.1 mg/kg and only traces of omethoate were detected. In the third trial, residues ranged from 7.83 to 23.8 mg/kg by 6 hours after application (for rates of 0.25 to 1.0 kg/ha). At 6 hours after

application the omethoate residue in the ferns was 0.5 mg/kg after application at the higher rate. Omethoate residues peaked 2 days after application (level not stated) and decreased continually thereafter. Omethoate residues exceeded dimethoate residues after 13 days due to the more rapid decline of dimethoate. About 90% of the total residues disappeared in 7 days and only a trace (<0.01 mg/kg) to 0.03 mg/kg were present after 38 days.

Spinach

A study on winter spinach was briefly reviewed. Dimethoate at rates of 0.25 to 0.5 kg ai/ha was applied in September/October, December, January and March. Dimethoate residues were 0.93 to 6.4 mg/kg immediately after treatment, declining to 0.1 to 0.86 mg/kg by 28 days after treatment. It was stated that the difference in disappearance rates did not appear to be related to temperature.

Stone fruit

A study on the migration behaviour of dimethoate/omethoate in stone fruit trees (peach and cherry) was reviewed. The study was conducted in Italy in 1959–60 and involved the trunk application of ^{32}P dimethoate at 2.5 to 20 g ai/tree. Samples were taken 1–60 days after treatment. In cherry leaves the maximum dimethoate residue (25 mg/kg) was found relatively late (30 days after application) and declined slowly (at 50 days after application 20 mg/kg dimethoate was detected). For peaches, it was shown that both the total ^{32}P and the ^{32}P extracted into the chloroform phase (dimethoate + omethoate) maintained higher levels in the leaves than the fruit. The total ^{32}P content in leaves reached a maximum and then slowly decreased, while for fruit the increase in total ^{32}P continued until ripeness. The ^{32}P extracted into the chloroform phase reached a maximum in leaves and a little later in fruit, and then decreased rapidly in both. The ^{32}P content of the fruit was more concentrated in the half of the fruit near the stem, due to penetration of the radioactive compounds through the stem end. Autoradiograms of peach leaves showed that the labelled compounds penetrated through the petiole and the main vein, and were distributed initially in the tissues between secondary veins and then localised progressively along the borders of the leaf blade.

Pome fruit

The migration behaviour of dimethoate in apples after trunk application was also reviewed. The maximum dimethoate concentration in the leaves was found about 30 days after application. By 70 days after application only 1% of the maximum content was present. In fruit, maximum values were detected 10 days after application and were 100-fold lower than those in leaves. By 40 days after application, 30% of the maximum value in fruit was detected. Actual concentrations were not recorded in the review document.

Grapes

After foliar application to grapes it was reported that residues of some dimethoate metabolites (N-desmethyl dimethoate, N-desmethyl omethoate, N-hydroxymethyl omethoate and the O-glucoside of N-hydroxymethyl dimethoate) were below the sensitivity limit of the method used (0.05 mg/kg). Dimethoate and omethoate were present at 0.35 to 0.5 mg/kg and about 0.15 mg/kg respectively at the end of the sampling period (28 days after application). In processed wine, grape pomace and raisins, dimethoate and omethoate values ranged from 0.15 to 0.37 mg/kg and <0.05 to 0.13 mg/kg respectively.

Tea

Two weeks after application of ^{14}C-dimethoate to leaves, 45% of the radioactivity was recovered from the processed leaves. An aqueous infusion extracted 52% of the recovered radioactivity of which 15% was extracted with ether and 32% with chloroform. Dimethoate, omethoate, O,O-dimethyl methylthiophosphoric acid and N-desmethyl dimethoate were identified in the extracts.

Flowering plants

After application on excised flowering plant tops, ^{14}C-dimethoate and its metabolites could be detected in all floral organs and in the nectar. However, after application of dimethoate to flowering potted plants at field rates, no nectar contamination via the systemic route could be observed.

No. 8347; Jalali, K; Krautter, G R; Cassidy, J E; The metabolism of ^{14}C-Dimethoate in the lactating goat following oral administration for 3 consecutive days; 23 January 1995

Two lactating goats 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 about 23 hours after the final dose and total radioactive residue levels were determined in tissues by combustion analysis.

Urinary excretion was the main route of elimination of radioactivity. About 91 and 86% was excreted in the urine of the two treated animals. Cumulative radioactivity in the feces accounted for 3 and 4% of the dose. After sacrifice less than 1% of the dose remained unexcreted in the gastrointestinal tract. Cumulative radioactivity recovered in milk accounted for less than 0.1% of the administered dose. The TRR found in the tissues and milk is summarised in Table 79.

Table 79: Total radioactive residues in tissues and milk in mg/kg

Sample	Goat no. 651	Goat no. 658
Liver	1.221	1.012
Kidney	0.149	0.154
Muscle	0.070	0.047
Fat (composite)	0.045	0.057
Blood	0.076	0.079
Milk	0.035–0.228	0.052–0.135

The observed TRRs in milk were higher in samples collected within 8 hours post treatment, compared to samples collected 8 to 24 hours post treatment, suggesting rapid elimination in milk. The highest residues in milk were observed in the samples collected from 48–60 hours, decreasing again after 60 hours. Residue levels in milk during the course of the study are summarised in Table 80.

Table 80: Variation of residue levels in milk with time

Collection interval	Mean residue (mg/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

Dimethoate was rapidly metabolised. Metabolites identified in urine were dimethoate carboxylic acid, dimethyl thiophosphate and dimethyl phosphate. For tissues and milk, extractable radioactivity (in acetonitrile/water) was high only for muscle and milk (Table 81). Metabolites identified in tissue and liver extracts are summarised in Table 82.

Table 81: Extractability of radiocarbon from tissues and milk

Sample	TRR (ppm)	Percent ^{14}C (mg/kg)		Percent ^{14}C total recovered
		Extracted	Residual	
Liver	1.221	41.1 (0.505)	47.1 (0.575)	90.5
Kidney	0.149	68.1 (0.101)	34.3 (0.051)	107.5
Muscle	0.070	84.3 (0.059)	17.3 (0.012)	101.7
Fat	0.045	28.6 (0.013)	82.1 (0.037)	110.7
Milk 48–60 hr	0.228	90.0 (0.205)	9.9 (0.023)	99.9

Table 82: Metabolites identified in tissues and milk

Component	mg equiv/kg			
	Liver	Kidney	Muscle	Milk
Dimethoate carboxylic acid (by HPLC only)	0.031[a]	N/D	N/D	0.019[a]
Omethoate (by HPLC only)	0.120[a]	N/D	N/D	N/D
Anions such as dimethyl phosphate and dimethyl thiophosphate	0.076	0.020	0.002	0.005
Phosphorylated natural products	0.762	0.130	0.049	0.148
TRR	1.221	0.149	0.070	0.228
Accountability of ^{14}C	81%	100.7%	72.9%	75.4%

a Solubilised by protease treatment of the extracted sample.

Residues of dimethoate and its metabolites did not concentrate in fat. All extractable fractions of fat contained ≤0.005 mg equiv/kg. The non-extractable residue was 0.018 mg equiv/kg. For other tissues, dimethoate carboxylic acid was identified in liver and milk, and omethoate in liver. The report suggests that the poor extractablility and the HPLC characterisation of the extractable radiocarbon support the ($^{14}CH_3$-O)$_2$P=O phosphorylation of natural products. Low levels of anions were also present in the extracts of liver, kidney, muscle and milk.

The proposed metabolic pathway leading to the residues in tissues and milk is summarised in Figure 7. 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 (not shown).

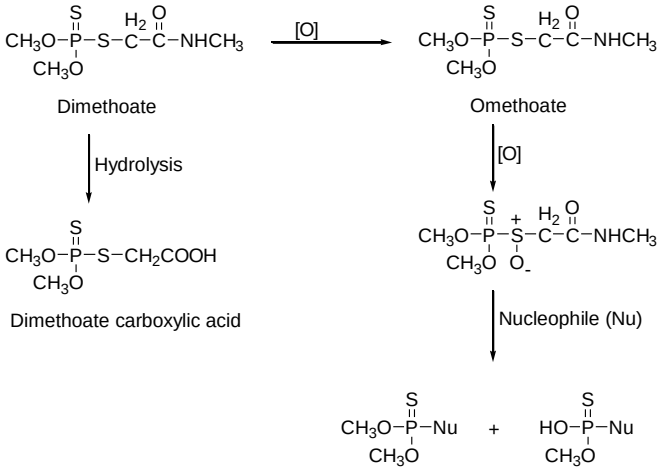

Figure 7: Proposed metabolic pathway for dimethoate in goats. (Note: Nu may be any electron-rich endogenous component. Thus the sulphoxide may phosphorylate proteins, lipids etc.)

No. 8348; Jalali, K; Hiler, R; Further characterisation of [^{14}C]-Dimethoate residues in the 48-60 hour milk extract from the study entitled "The metabolism of [^{14}C]-dimethoate in the lactating goat following oral administration for 3 consecutive days" (MRID 435 83301); 29 April 1997

The composition of the residue in a subsample of milk from the previous study was investigated further. The TRR in the sample was 0.210 mg/kg. A hexane extraction removed 7.5% (0.015 mg/kg) with a further 68.9% (0.145 mg/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 equiv/kg) eluted in water (neutral fraction) with a further 53% (0.077 mg equiv/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 equiv/kg. The underivatised solvent front band contained 0.032 mg equiv/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 equiv/kg) after derivatisation with PFBB. Underivatised material in the solvent front accounted for 0.063 mg equiv/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 report suggested that the polar anionic metabolites in the acidic fraction may be phosphorylated natural products.

No. 8349; Jalali, K; Krautter, G R; Cassidy, J E; The metabolism of [^{14}C]-Dimethoate in laying hens following oral administration for 7 consecutive days; 23 January 1995

Three groups of five white Leghorn laying hens 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 was 66%, in cage wash 9%, in gastrointestinal 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 0.339 mg/kg; levels in whites ranged from 0.090 to 0.180 mg/kg. A plateau was not reached during dosing. TRRs in tissues and eggs are summarised in Table 83.

Table 83: Residue levels in tissues and eggs

Sample	Collection	Mean TRR (mg equiv/kg)			
		Group B	Group C	Group D	Overall mean B,C,D
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 radiocarbon from tissues and eggs was poor (Table 84). Dimethoate was not detected in any of the tissues, egg, excreta or blood extracts, suggesting rapid metabolism. The major metabolites dimethyl phosphate and dimethyl thiophosphate were rapidly excreted. It was suggested that if these metabolites were present in the tissues or eggs then the extraction procedure would have efficiently extracted them. Omethoate and dimethaote carboxylic acid were characterised by HPLC. Most of these metabolites were released from liver and egg white following protease treatment (Table 85).

Table 84: Extractability of radiocarbon from tissues and eggs with acetonitrile-water (8:2)

Sample	TRR (mg/kg)	Percent ^{14}C (mg/kg)		Percent ^{14}C Recovered
		Extracted	Residual[A]	
Liver	0.705	8.9 (0.063)	90.5 (0.638)	99.6
Breast muscle	0.098	46.2 (0.045)	59.1 (0.058)	105.3
Thigh muscle	0.079	36.0 (0.029)	54.2 (0.043)	90.2
Egg whites	0.127	50.0 (0.064)	37.3 (0.047)	87.3
Egg yolks	0.192	29.4 (0.057)	57.5 (0.110)	86.8
Skin	0.038	26.7 (0.009)	54.6 (0.021)	81.3
Fat	0.028	9.8 (0.003)	29.3 (0.008)	69.7

a As determined by combustion of post extraction solids.

Table 85: Identification of metabolites in liver and egg white

Matrix	Metabolite	mg equiv/kg
Liver	Omethoate, HPLC only	0.081
	Dimethoate carboxylic acid, HPLC only	0.131
	^{14}C-Phosphorylated natural products	0.514
Egg white	Omethoate, HPLC only	0.004
	Dimethoate carboxylic acid, HPLC only	0.005
	^{14}C-Phosphorylated natural products	0.103
Matrix	**Metabolite**	**mg equiv/kg**
Liver	Omethoate, HPLC only	0.081
	Dimethoate carboxylic acid, HPLC only	0.131
	^{14}C-Phosphorylated natural products	0.514
Egg white	Omethoate, HPLC only	0.004
	Dimethoate carboxylic acid, HPLC only	0.005
	^{14}C-Phosphorylated natural products	0.103

For tissue protease, HCl and NaOH treatment were required to solubilise residual radiocarbon. No evidence of ^{14}C-formate was found. No evidence of ^{14}C amino acids were found after solubilisation of liver extracts with 6 N HCl. Anionic compounds in the excreta were readily derivatised by benzylation; derivatisation of metabolites extracted from the tissues was poor. Due to difficulties in extraction and analysis by C-18 HPLC, where ^{14}C eluted early and was poorly recovered, it was suggested the main path for metabolism in tissues was the $(^{14}CH_3\text{-}O)_2P{=}O$ phosphorylation of natural products. This is supported by the goat metabolism study. In egg yolk 31.8% of the TRR co-chromatographed with phospholipids. It was suggested that this indicates

either the radiocarbon has entered the carbon pool or that some phospholipids were phosphorylated and they co-chromatographed with natural phospholipids. A metabolic pathway for the production of radiocarbon residues in tissues was proposed (Figure 8). 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.

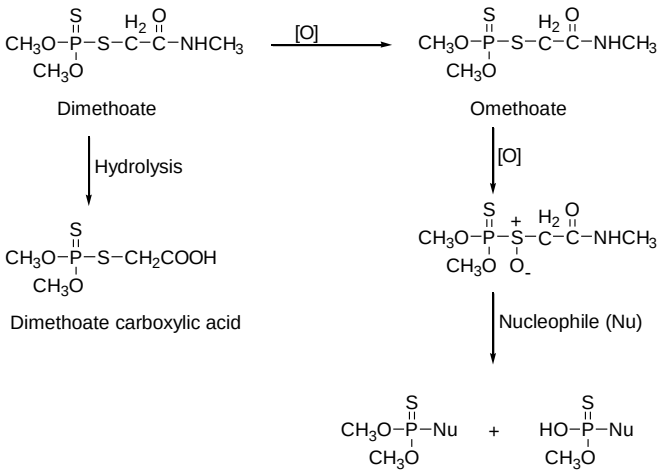

Figure 8: Proposed metabolic pathway for dimethoate in hens. (Note: Nu may be any electron-rich endogenous component. Thus the sulphoxide may phosphorylate proteins, lipids etc.)

No. 8379; Adair, T H; Sprinkle, R B; Toia, R F; A confined rotational crop study with ^{14}C-Dimethoate using lettuce, turnips and wheat; 21 June 1995

A confined rotational accumulation 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 0.5 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 screenhouses 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 summarised in Table 86.

Table 86: Total radioactive residues in rotational crops after treatment of soil with [^{14}C-methoxy]dimethoate

Crop	TRR (mg equiv/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 generally an order of magnitude 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 equiv/kg were subjected to solvent extraction (acetonitrile and acetonitrile/HCl, Table 87). Non-extractable fractions ranged from 0.006 to 0.031 mg/kg. Extracts containing >0.01 mg equiv/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 equiv/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 87: Extractable and non-extractable residues from crops grown in soil treated with ^{14}C-dimethoate

Crop	Planting group[A]	TRR (mg/kg)	Extracted radiocarbon				Non-extracted		Total %
			Method A[B]		Method B[C]				
			%	mg/kg	%	mg/kg	%	mg/kg	
Lettuce	I	0.030	74.2	0.022	–	–	26.0	0.008	100.2
Turnip foliage	I	0.037	55.3	0.020	–	–	44.8	0.017	100.1
Wheat forage	I	0.036	63.1	0.023	–	–	41.5	0.015	104.6
Wheat hay	I	0.037	34.8	0.013	–	–	83.8	0.031	118.6
Wheat straw	I	0.045	14.9	0.007	57.6	0.027	29.1	0.013	101.6
	II	0.020	28.8	0.006	59.0	0.012	28.2	0.006	116.0

Wheat grain	I	0.021	3.8	0.001	58.4	0.012	51.8	0.011	114.0
	II	0.012	3.1	0.000	24.3	0.003	81.9	0.010	109.3

a Group I planted 30 DAT, Group II planted 120 DAT.

b Extracted with acetonitrile.

c Extracted with acetonitrile/0.1 N HCl (1:1) following the method A extraction.

Crop samples were stored for 15–27 days between collection and analysis. Storage stability (Table 88) 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 88: 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	0.980	104.0	1.019	95.9	0.977
	42	0.966	100.9	0.975	99.5	0.970
Lettuce	14	1.024	101.5	1.039	91.4	0.950
	48	1.021	95.6	0.976	100.0	0.976
Wheat hay	6	1.039	97.4	1.012	99.3	1.005
	32	1.025	65	0.666	100.0	0.666
Turnip foliage	6	1.039	98.2	1.020	94.8	0.967
	32	0.976	100.3	0.979	88.1	0.862
Wheat grain	6	1.033	75.0	0.775	81.5	0.632
	53	1.026	95.7	0.982	97.7	0.959
Wheat straw	6	1.033	96.6	0.998	100.0	0.998
	53	1.029	108.1	1.112	99.1	1.102

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.

APPENDIX 4: PROCESSING STUDIES

No. 8350; Aikens, P J; Dimethoate and omethoate: hydrolysis under simulated processing conditions; 14 December 1999

The hydrolysis of [^{14}C-methoxy]-dimethoate and omethoate were investigated under simulated processing conditions. The test substances were added separately to aqueous buffer solutions at pH 4, 5 and 6. The concentration of the test substances was 1 µg/ml. The solutions were incubated in darkness under the following conditions:

- pH 4, 90^{o}C, 20 minutes, represents pasteurisation
- pH 5, 100^{o}C, 60 minutes, represents baking, brewing and boiling
- pH 6, 120^{o}C, 20 minutes, represents sterilisation.

Dimethoate incubations

At the end of the 90, 100 and 120^{o}C incubations, dimethoate represented 93.3, 66.4 and 29.5% of the applied radioactivity. The major degradate was desmethyl dimethoate accounting for 4.7, 28.1 and 59.5% of the applied radioactivity after incubation at 90, 100 and 120^{o}C respectively. The degradate des-O-methyl isodimethoate reached a maximum of 5.3% of the applied radioactivity after incubation a 120^{o}C. Three other reactive degradates of dimethoate were detected, none of which exceeded 2.5% of the applied radioactivity (Table 89).

Table 89: Proportions of radioactive components in aqueous buffer after incubation of ^{14}C-dimethoate

Component	pH 4, 90^{o}C, 20 mins	pH 5, 100^{o}C, 60 mins	pH 6, 120^{o}C, 20 mins
1	0.5	1.2	2.4
2	0.4	0.8	1.2
3	0.9	2.5	2.4
Des-O-methyl isodimethoate	0.3	0.9	5.3
Desmethyl dimethoate	4.7	28.1	59.5
Dimethoate	93.3	66.4	29.5

Omethoate incubations

At the end of the 90, 100 and 120^{o}C incubations, omethoate represented 92.9, 61.3 and 5.5% of the applied radioactivity respectively. The major degradate was O-desmethyl omethoate, representing 6.3, 36.2 and 62.6% of the applied radioactivity respectively. Only one other degradate exceeded 10% of the AR: dimethyl phosphoric acid represented 19.2% of the AR after incubation at 120^{o}C (Table 90).

Table 90: Proportions of radioactive components in aqueous buffer after incubation of ^{14}C-omethoate

Component	pH 4, 90°C, 20 mins	pH 5, 100°C, 60 mins	pH 6, 120°C, 20 mins
A	n.d.	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

No. 8366; Rice, F; Beckerman, J M; Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate in or on corn grain and its processed commodities; 28 June 1994

A processing study was conducted on corn grown in Iowa in 1993. An EC formulation of dimethoate was applied to corn at two rates: (1×) 0.5 lb ai/A (0.56 kg ai/ha) and (5×) 2.5 lb ai/A (2.8 kg ai/ha). Three applications were made at about 7-day intervals with the final application at 14 days before harvest. Application volumes were approximately 188 L/ha. Corn grain samples were harvested with a self-propelled combine at 14 days after the last application. Corn grain samples were processed using simulated commercial practices into grits, meal, flour, starch, and wet and dry milled oils (crude, refined and bleached deodorised). Grain samples were processed within 93 days of harvest. Samples were extracted within 23 days of processing and analysis took place within 8 days of extraction. Samples were stored frozen before analysis.

With the exception of corn oil, samples were homogenised with acetone, filtered, partitioned with methylene chloride and cleaned up with a charcoal/celite column. Oil samples were dissolved in 15:85 methylene chloride/cyclohexane and cleaned up by gel permeation chromatography. Residues were quantified by gas chromatography with a flame photometric detector in the phosphorus mode. Recoveries of dimethoate and omethoate from control samples of grain, grits, meal, flour, starch and oil fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 91.

Table 91: Recoveries of dimethoate and omethoate from fortified control samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Grain	80–92	87	5	77–100	86	10
Grits	87–94	90	2	83–110	92	9
Meal	76–100	88	8	74–100	88	8
Flour	91–100	96	4	82–120	101	15
Starch	80–90	86	4	80–100	86	6
Oil	78–91	87	5	66–100	78	13

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in grain, grits, meal, flour, starch and oil samples derived from the control plot. Residues found in samples derived from the treated plot are summarised in Table 92.

Table 92: Residues of dimethoate and omethoate in corn and its processed fractions

Matrix	Residue (mg/kg)	
	Dimethoate	Omethoate
Grain	0.06	<0.01
	0.07	<0.01
Grits	0.02	<0.01
	0.02	<0.01
Meal	0.02	<0.01
	0.02	<0.01
Flour	0.02	<0.01
	0.02	<0.01
Starch	<0.01	<0.01
	<0.01	<0.01
Oil – wet milled crude	<0.01	<0.01
	<0.01	<0.01
Oil – wet milled refined	<0.01	<0.01
	<0.01	<0.01
Oil – wet milled bleached	<0.01	<0.01
	<0.01	<0.01
Oil – dry milled crude	0.02	0.04
	0.02	0.01
Oil – dry milled refined	<0.01	<0.01
	<0.01	<0.01
Oil – dry milled bleached	<0.01	<0.01
	<0.01	<0.01

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in starch and refined or bleached corn oil samples derived from the treated plot. The maximum total dimethoate residue (expressed as parent equivalents) was 0.07 mg/kg in grain, 0.02 mg/kg in grits, meal and flour, and 0.06 mg/kg in crude corn oil. Residues of dimethoate and omethoate did not concentrate in processed field corn grain commodities.

No. 8367; Rice, F; Beckerman, J M; Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate, in or on raw agricultural and processed commodities of white potatoes; 10 June 1994

A processing study was conducted on potatoes grown in the Yakima Valley, Washington, USA in 1993. An EC formulation of dimethoate was applied to white poatoes at two rates: (1×) 0.5 lb ai/A (0.56 kg ai/ha) and (5×) 2.5 lb ai/A (2.8 kg ai/ha). Three applications were made 7 days apart in a spray volume of 185 L/ha. Potato tubers were harvested on the day of the final application and processed to granules, chips, wet peel and dry peel. Processing took place within 39 days of sampling. Samples were extracted within 26 days of processing and analysed within 6 days of extraction. Samples were stored frozen before analysis.

With the exception of chips, samples were extracted with acetone, filtered, partitioned with methylene chloride and cleaned up using a charcoal/celite column. Due to their high oil content, chip samples were extracted with EtOAc and cleaned up by gel permeation chromatography. Residues were quantified by gas chromatography with a flame photometric detector in phosphorus mode. Recoveries of dimethoate and omethoate from control samples fortified between 0.01 and 1.0 mg/kg are summarised in Table 93.

Table 93: Recoveries of dimethoate and omethoate from fortified control samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Tubers	90–98	93	3	100–130	114	10
Granules	90–106	98	6	101–130	112	9
Chips	91–120	100	11	77–130	96	21
Wet peel	80–97	89	5	91–120	101	13
Dry peel	80–93	83	5	76–100	87	8

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in tuber, granule, chip, wet peel and dry peel samples derived from the control plot. Residues in samples derived from the treated plot are summarised in Table 94.

Table 94: Residues of dimethoate and omethoate in white potatoes and processed fractions

Matrix	Residue (mg/kg)	
	Dimethoate	Omethoate
Tubers	0.09	<0.01
	0.06	<0.01
	0.08	<0.01
Granules	0.01	<0.01
	0.01	<0.01
Chips	<0.01	<0.01
	<0.01	<0.01
Wet peel	0.02	<0.01
	0.02	<0.01
Dry peel	0.06	<0.01
	0.06	<0.01

Residues of dimethoate and omethoate in chip samples from the treated plot were non-detectable (<0.01 mg/kg). The maximum total dimethoate residue (in parent equivalents) was 0.09 mg/kg in tubers, 0.01 mg/kg in granules, 0.02 mg/kg in wet peel and 0.06 mg/kg in dry peel.

No. 8368; Rice, F; Beckerman, J M; Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate, in or on wheat grain and its processed commodities; 10 June 1994

A processing study was conducted on wheat grown in Kansas in 1993. An EC formulation of dimethoate was applied to wheat 35 days before harvest at two rates: (1×) 0.38 lb ai/A (0.43 kg ai/ha) and (5×) 1.9 lb ai/A (2.1 kg ai/ha). The application volume was 19 gallons/A (178 L/ha). Wheat grain samples were harvested with a self-propelled combine at 37 days after application. Grain from the 5× treated plot was processed to bran, middlings, shorts and flour within 49 days of sampling. Processed samples were extracted within 76 days of processing and analysed within 7 days of extraction. Samples were stored frozen before analysis.

Samples were extracted with acetone, filtered, partitioned with methylene chloride and cleaned up with a charcoal/celite column. Residues were quantified by gas chromatography with a flame photometric detector in the phosphorus mode. Recoveries of dimethoate and omethoate from control samples of wheat grain, bran, middlings, shorts and flour fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 95.

Table 95: Recoveries of dimethoate and omethoate from fortified control samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Grain	90–96	92	3	103–120	110	7
Bran	90–98	93	3	96–109	103	5
Middlings	80–99	93	6	91–100	97	3
Shorts	90–102	95	5	92–120	106	11
Flour	70–140	92	23	73–124	93	17

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in grain, bran, middlings, shorts and flour samples from the untreated control plot. Residues found in samples derived from the 5× treated plot are summarised in Table 96.

Table 96: Residues of dimethoate and omethoate in wheat grain and processed fractions

Matrix	Residue (mg/kg)	
	Dimethoate	Omethoate
Grain	<0.01	<0.01
	<0.01	<0.01
Bran	<0.01	<0.01
	<0.01	<0.01
Middlings	<0.01	<0.01
	<0.01	<0.01
Shorts	<0.01	<0.01
	<0.01	<0.01
Patent flour	<0.01	<0.01
	<0.01	<0.01
Low grade flour	<0.01	<0.01
	<0.01	<0.01

Residue of dimethoate and omethoate were not detected (<0.01 mg/kg) in processed fractions derived from grain from the treated plot. As residues in grain from this plot were also <0.01 mg/kg, it is not possible to calculate processing factors.

No. 8369, CHA Doc. No. 177 DMT; Rice, F, Beckerman, J M, Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate, in or on oranges and its processed commodities, 1994

Orange plots located in southern Florida received 2 applications of a dimethoate EC formulation at 0.75 or 3.75 lb ai/100 gal (90 or 449 g ai/100 L). The dates of application were 09/12/93 and 23/12/93. Mature orange RAC samples were obtained from each plot 14 days after the last application. Orange samples from the 3.75 lb ai/100 gal treated plot and control plot were processed and analysed. Samples were stored frozen before analysis. Orange samples were processed within 18 days after sampling. Processed fractions were extracted within 25 days of processing and analysed within 9 days of extraction. Samples (except oils) were extracted with acetone, filtered, partitioned with methylene chloride and cleaned up with a charcoal/celite column. Oil samples were diluted with cyclohexane/methylene chloride and cleaned up by gel permeation chromatography. Samples were analysed by gas chromatography with a flame photometric detector in the phosphorus mode. The analytical method was validated for samples fortified at 0.01, 0.05 and 5 mg/kg. Recoveries are summarised in Table 97.

Table 97: Recoveries of dimethoate and omethoate from orange samples

matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Whole oranges	95–110	101	5	82–120	100	12
Juice	102–110	107	4	90–107	99	5
Dry pulp	69–84	79	5	63–80	72	6
Molasses	70–100	87	12	66–101	90	12
Oil	80–100	93	7	65–120	79	21

Residues of dimethoate and omethoate were <0.01 mg/kg in whole oranges, juice, dried pulp, molasses and oil samples from the control plot. Residues in treated samples and processed fractions are summarised in Table 98.

Table 98: Residues of dimethoate and omethoate in oranges

Matrix	Residue (mg/kg)		
	Dimethoate	Omethoate	Total[A]
Whole oranges—unwashed	1.07	0.12	1.20
	1.82	0.17	2.00
Whole oranges—washed	1.98	0.20	2.20
	1.03	0.12	1.16
Juice	0.20	0.03	0.23
	0.21	0.03	0.24
Dry pulp	3.18	0.24	3.44
	2.92	0.24	3.18
Molasses	8.14	0.71	8.90
	8.73	1.06	9.87
Oil	0.28	<0.01	0.28
	0.29	<0.01	0.29

a A value of 1.075 was used for the molecular weight ratio.

No. 8370; Rice, F; Beckerman, J M; Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate, in or on cottonseed and its processed commodities; 20 July 1994

A processing study was conducted on cottonseed grown in Texas in 1993. An EC formulation of dimethoate was applied to cotton at two rates: (1×) 0.5 lb ai/A (0.56 kg ai/ha) and (5×) 2.5 lb ai/A (2.8 kg ai/ha). Two applications were made 13 days apart, with the final application 14 days before harvest. The application volume was 15 gallons/A (140 L/ha). Cottonseed samples were harvested by hand or with a self-propelled cotton picker at 14 days after the second application. Cottonseed from the control and 5× treated plot were processed by simulated commercial practices to delinted seed, meal, hulls, oil (crude, refined and bleached/deodorised) and soapstock. Cottonseed samples were processed within 44 days of harvest. Samples were extracted within 80 days of processing and analysed within 9 days of extraction. Samples were stored frozen until analysis.

Cottonseed RAC samples were Soxhlet extracted using ethyl acetate as solvent. After extraction the ethyl acetate was evaporated and the sample cleaned up by gel permeation chromatography. Cottonseed meal and hull samples were extracted with acetone, filtered, partitioned with methylene chloride and cleaned up with a charcoal/celite column. Oil samples were dissolved in 15:85 methylene chloride/cyclohexane and cleaned up by gel permeation chromatography. Soapstock samples were first acidified and then treated the same as oil samples. Recoveries from control samples fortified at 0.01, 0.05 and 0.5 mg/kg (cottonseed, meal, hulls and oil) and 0.02 and 0.05 mg/kg for soapstock are summarised in Table 99. The method was not successfully validated for omethoate in soapstock.

Table 99: Recoveries of dimethoate and omethoate from fortified control samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Cottonseed	90–110	98	8	49–80	65	14
Meal	78–90	83	4	66–90	82	8
Hulls	83–90	87	3	81–100	90	8
Crude oil	90–120	105	13	99–140	116	17
Soapstock	70–82	76	6	24–40	31	7

Residues of dimethoate and omethoate were non-detectable in cottonseed, meal, hull, oil (<0.01 mg/kg) and soapstock (<0.02 mg/kg) in samples from the control plot. Residues found in samples derived from the 5× treated plot are summarised in Table 100.

Table 100: Residues found in cottonseed and processed fractions

Matrix	Residue (mg/kg)		
	Dimethoate	Omethoate	Total[A]
Cottonseed	0.02	<0.01	0.02
	0.03	<0.01	0.03
Meal	0.04	<0.01	0.04
	0.03	<0.01	0.03
Hulls	0.08	<0.01	0.08
	0.09	<0.01	0.09
Crude oil	0.02	<0.01	0.02
	0.02	<0.01	0.02
Refined oil	<0.01	<0.01	<0.01
	<0.01	<0.01	<0.01
Bleached oil/deodorised oil	<0.01	<0.01	<0.01
	<0.01	<0.01	<0.01
Soapstock	<0.02	–[b]	<0.02
	<0.02	–	<0.02

a A value of 1.075 was used for the molecular weight ratio of dimethoate to omethoate.

b Residues of omethoate were not detectable—the analytical method quantifies an average of 31% from samples fortified with omethoate at 0.02–0.05 mg/kg.

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in refined and bleached oil samples and non-detectable in soapstock (although the method for soapstock was not successfully validated for omethoate). For the other matrices, the maximum total dimethoate residues (in parent equivalents) were 0.03 mg/kg in cottonseed, 0.04 mg/kg in meal, 0.09 mg/kg in hulls and 0.02 mg/kg in crude oil samples.

No. 8371; Rice, F; Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate in or on raw agricultural and processed commodities of tomatoes; 18 January 1995

A processing study was conducted on tomatoes grown in California. An EC formulation of dimethoate was applied to tomatoes at three rates: (1×) 0.5 lb ai/A (0.56 kg ai/ha), (2×) 1.0 lb ai/A (1.1 kg ai/ha) and (5×) 2.5 lb ai/A (2.8 kg ai/ha). Four applications were made at 7-day intervals with the final application 7 days before harvest. Application volumes were about 188 L/ha. Tomato samples were collected from treated and untreated plots at 7 days after the last application. Tomato samples from the control and 5× treated plots were processed according to simulated commercial practices into juice, wet pomace, dry pomace, puree, paste and catsup. Samples were processed within 2 days of harvest. Processed products were stored frozen for up to 31 days before analysis.

Samples were extracted with acetone, filtered, partitioned with methylene chloride, and cleaned up with a charcoal/celite column. Residues were quantified by gas chromatography with FPD in phosphorus mode. Recoveries from samples of tomatoes and processed products fortified at 0.01 to 0.5 mg/kg are summarised in Table 101.

Table 101: Recoveries of dimethoate and omethoate from fortified control samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Whole tomatoes	80–96	89	7	100–116	110	5
Juice	97–100	99	2	104–110	107	4
Wet pomace	83–93	89	4	85–100	93	11
Dry pomace	70–104	87	11	70–114	97	17
Puree	80–97	89	12	80–114	97	24
Paste	90–110	98	7	60–107	88	18
Catsup	90–101	96	8	60–106	83	33

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in whole tomatoes, juice, wet pomace, dry pomace, puree, paste and catsup samples from the control plot. Residues in samples from the treated plot are summarised in Table 102.

Table 102: Residues of dimethoate and omethoate in tomatoes and processed fractions

Sample	Residue (mg/kg)			Concentration factor[B]
	Dimethoate	Omethoate	Total[A]	
Whole tomatoes	0.15	0.05	0.20	–
	0.24	0.07	0.32	
	0.13	0.06[c]	0.19	
	0.18	0.06[c]	0.24	
Juice	0.02	<0.01	0.02	–
	0.02	<0.01	0.02	
Wet pomace	0.10	0.01	0.11	–
	0.12	0.02	0.14	
Dry pomace	0.11	0.01	0.12	–
	0.09	<0.01	0.09	
Puree	0.29	0.06	0.35	1.5
	0.31	0.06	0.37	
Paste	0.50	0.08	0.59	2.6
	0.56	0.09	0.66	
Catsup	0.34	0.06	0.40	1.6
	0.31	0.05	0.36	

a A value of 1.075 was used for the molecular weight ratio of omethoate to dimethoate.

b Concentration factor = average total residue in matrix/average total residue in RAC.

c These samples were not analysed for dimethoate. For the purpose of calculation of total residues, the average omethoate residue from the other trials was used.

Maximum total residues were 0.32 mg/kg in whole tomatoes, 0.02 mg/kg in juice, 0.14 mg/kg in wet pomace, 0.12 mg/kg in dry pomace, 0.37 mg/kg in puree, 0.66 mg/kg in paste and 0.40 mg/kg in catsup. Residues of dimethoate and omethoate do not concentrate in tomato juice, wet pomace or dry pomace. Residues did appear to concentrate in puree (1.5×), paste (2.6×) and catsup (1.6×).

Moisture contents were reported to be 2.4% for dry pomace and 63.8% for wet pomace.

No. 8372; Judy, D; Williams, B B; Magnitude of the residues of dimethoate and its oxygen analog, omethoate, in or on raw agricultural and processed commodities of safflower; 13 April 1998

A processing study was conducted on safflower grown in California in 1997. An EC formulation of dimethoate was applied to safflower at two rates: (1×) 0.5 lb ai/A (0.56 kg ai/ha) and (5×) 2.5 lb ai/A (2.8 kg ai/ha). Two applications were made approximately 14 days apart, with the final application occurring 14–15 days before the estimated harvest. The application volume was approximately 188 L/ha. Safflower seed

samples were collected by hand at 14–15 days after the last application. Seed samples from the 5× treated plot were processed using simulated commercial practices into meal and refined oil. Samples were processed within 42–43 days of sampling. Seed samples were extracted within 33 days of sampling, oil and meal samples were extracted within 32 days of processing. Analysis was within 3 days of extraction. Samples were stored frozen prior to analysis.

Dimethoate and omethoate were extracted from seed by Soxhlet extraction with ethyl acetate. The extract was cleaned up by gel permeation chromatography. Residues were extracted from meal using acetone and an accelerated solvent extractor. The extract was cleaned up on a celite:activated charcoal column. Oil samples were diluted with solvent and cleaned up by gel permeation chromatography. Residues were quantified by gas chromatography with a flame photometric detector in the phosphorus mode. Recoveries of dimethoate and omethoate from control samples of seed, meal and oil fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 103.

Table 103: Recoveries of dimethoate and omethoate from fortified control samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Seed	50–86	73	14	40–50	45	4
Meal	88–94	91	2	68–90	80	8
Oil	70–115	101	15	80–110	98	10

Residues of dimethoate and omethoate were non-detectable (<0.01 mg/kg) in safflower seed, meal and refined oil samples from the control plot. Residues found in samples derived from the treated plot are summarised in Table 104.

Table 104: Residues of dimethoate and omethoate in safflower seed and processed fractions

Sample	Residue (mg/kg)			Concentration factor
	Dimethoate	Omethoate	Total[A]	
Whole safflower seed	0.69	0.01	0.70	NA
Meal	0.67	0.01	0.68	NA
Refined oil	<0.01	<0.01	<0.01	NA

a A value of 1.075 was used for the molecular weight ratio of omethoate to dimethoate.

The maximum total dimethoate residue (in parent equivalents) was 0.70 mg/kg in seed, 0.68 mg/kg in meal and <0.01 mg/kg in refined oil. Residues of dimethoate and omethoate do not concentrate in safflower meal or refined oil.

No. 8373; Schulz, H; Determination of the residues of dimethoate and omethoate in white cabbage and processed products following treatment with CHA 3620 (dimethoate 400 g/L EC), Germany 1999; 2 June 2000

A processing study was conducted on cabbage grown in Germany in 1999. Six applications of an EC formulation of dimethoate were carried out with 7-day intervals, with the final application 21 days before harvest. The first 5 applications were at the nominal rate of 400 g ai/ha, with the sixth at 1200 g ai/ha. The wetting agent 'Agral' was added to the application solution at 0.03%. Samples were collected at harvest (BBCH growth stage 49), 21 days after the last application. The cabbage heads were processed to salad and cooked cabbage.

Residues were extracted by maceration with ethyl acetate and cleaned up by gel permeation chromatography. Residues were quantified by gas chromatography using either a flame photometric detector in the phosphorus mode or mass selective detection. The LOQ for dimethoate and omethoate was 0.01 mg/kg. Recovery rates from fortified samples are summarised in Table 105.

Table 105: Recoveries from fortified control samples

	Dimethoate	Omethoate
No. of fortified samples	12	12
Mean recovery value (%)	90.3	89.4
Standard deviation (%)	9.0	15.1
Coefficient of variation (%)	10.0	16.9

No residues above the limit of quantification were detected in any of the untreated samples. Residues in samples from the treated plot are summarised in Table 106.

Table 106: Residues in white cabbage and processed products

Sample material	Residue (mg/kg)		Total residue (mg/kg)[a]
	Dimethoate	Omethoate	
White cabbage (RAC)	0.0514	0.0828	0.140
Outer leaves	0.187	0.495	0.719
Core, cut to salad	0.0108	<0.01	0.0158
Stalks	0.041	<0.01	0.046
Cooked white cabbage	<0.01	<0.01	<0.01
Boiled water	<0.01	<0.01	<0.01

a Calculated as parent equivalents, <0.01 set as 0.005 mg/kg for calculation purposes.

The highest total residue of 0.719 mg/kg was found in the outer leaves of the cabbage. Residues in the head, stalks and the core (cut to salad) were 0.140, 0.046 and 0.0158 mg/kg respectively. No residues above the LOQ were found in the cooked white cabbage samles and in the boiled water samples. Transfer factors for dimethoate and omethoate in the treated samples are shown in Table 107.

Table 107: Transfer factors for dimethoate and omethoate in the processed products of white cabbage

	Residue concentration of dimethoate (mg/kg)	Transfer factor
White cabbage (RAC)	0.051	1
Outer leaves	0.187	3.7
Inner and outer stalks	0.041	0.8
Core, cut to salad	0.011	0.2
Cooked white cabbage	<0.01	–
Boiled water	<0.01	–
	Residue concentration of omethoate (mg/kg)	**Transfer factor**
White cabbage (RAC)	0.083	1
Outer leaves	0.495	6
Inner and outer stalks	<0.01	–
Core, cut to salad	<0.01	–
Cooked white cabbage	<0.01	–
Boiled water	<0.01	–

No. 8374; CHA Doc. No. 490 DMT; Wilson, A; Dimethoate: residue study (at harvest and processing) with an EC formulation containing 400 g/L dimethoate applied to olives in Greece in 2000

Table 108: STUDY DESCRIPTION

Active Substance (common name)	Dimethoate	**Commercial Product name**	Perfekthion
Crop/Crop Group	Olives/assorted tropical and sub-tropical fruits – edible peel	**Producer of Commercial Product**	BASF
Responsible body for Reporting (name, address)	Huntingdon Life Sciences Ltd	**Indoor/Glasshouse/Other**	Field
Country:	Greece	**Other active substance in the formulation (common name and content)**	N/A
Content of Active Substance (g/kg or g/L)	400 g/L	**Residues Calculated as**	Dimethoate + Omethoate
Formulation (eg WP)	EC		

Table 109: STUDY RESULTS

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment			Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
CHA Doc. No. 490 DMT, Greece	Olives, Ladolia (Karies, Greece)	29/11/00 (harvest)	Research lance	0.06 kg ai/100 L	Water 1212, 1201, 1198, 1201 L/ha	0.756, 0.749, 0.747, 0.749 kg ai/ha	18/05/00	BBCH 80	Olive pulp	0	4.37	0.09	
							24/07/00			14	1.06	0.48	
							17/08/00			21	0.50	0.54	
							08/11/00			29	0.11	0.39	
										35	0.13	0.40	
										42	0.13	0.44	
									Olives		0.39	0.37	Prior to processing
									Raw oil		0.17	<0.002	
									Refined oil		<0.01	<0.002	
									Canned		0.12	0.07	0 day (sterilised)
									Canned		0.03	0.01	10 day (sterilised)
									Canned		0.02	<0.002	6 month (sterilised)
									Canned		0.36	0.33	0 day (non-sterilised)
									Canned		0.29	0.27	10 day (non-sterilised)
									Canned		0.11	0.02	6 month (non-sterilised)

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment			Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
	Olives, Ladolia (Karies, Greece)	29/11/00 (harvest)	Research lance	0.30 kg ai/100 L	Water 1208, 1205, 1198, 1201 L/ha	3.766, 3.757, 3.735, 3.744 kg ai/ha	18/05/00 24/07/00 17/08/00 08/11/00	BBCH 80	Olive pulp	0 21	23.19 4.79	0.16 1.48	
									Olives		3.01	0.92	Prior to processing
									Raw oil		1.26	<0.01	
									Refined oil		0.02	<0.002	
									Canned		0.34	0.04	0 day (sterilised)
									Canned		0.08	0.02	10 day (sterilised)
									Canned		0.20	0.02	6 month (sterilised)
									Canned		4.02	0.98	0 day (non-sterilised)
									Canned		3.22	0.77	10 day (non-sterilised)
									Canned		0.92	0.06	6 month (non-sterilised
													Controls <0.01 mg/kg except 0 DALA RAC sample which contained 0.03 mg/kg omethoate, dimethoate recoveries 70–110%, omethoate recoveries 70–103%, samples analysed within 22 weeks of harvest or 9 weeks of processing.

No. 8375; Wilson, A; Residue study (at harvest and processing) with an EC formulation containing 400 g/L dimethoate applied to olives in Greece and Spain in 2001; 6 January 2003

Table 110: STUDY DESCRIPTION

Active Substance (common name)	Dimethoate	Commercial Product name	Perfekthion
Crop/Crop Group	Olives/assorted tropical and sub-tropical fruits–edible peel	Producer of Commercial Product	BASF
Responsible body for Reporting (name, address)	Huntingdon Life Sciences Ltd	Indoor/Glasshouse/Other	Field
Country	Greece, Spain	Other active substance in the formulation (common name and content)	N/A
Content of Active Substance (g/kg or g/L)	400 g/L	Residues Calculated as	Dimethoate + omethoate
Formulation (eg WP)	EC		

Table 111: STUDY RESULTS

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment			Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
No. 8375, Greece, Spain	Olives, Megaritikes (Karia, Greece)	1971 (planting) 14/11/01 (harvest)	Research lance	0.31, 0.31, 0.31, 0.31 kg ai/100 L	Water 702, 701, 701, 700 L/ha	2.19 2.19 2.19 2.18 kg ai/ha	17/05/01 10/07/01 23/08/01 24/10/01	BBCH 86	Olive pulp	0 21	19.34 5.49	0.24 1.37	
									Olives		4.82	1.27	Prior to processing
									Raw oil		1.55	<0.002	
									Refined oil		1.27	0.03	
									Canned olives		0.60	0.03	6 month (sterilised)
									Canned brine		0.29	0.04	6 month (sterilised)
									Canned olives		1.33	0.10	6 month (non-sterilised)
									Canned brine		0.58	0.09	6 month (non-sterilised)
	Olives, Koroneikes (Koutso-podio, Greece)	1991 (planting) 30/01/02 (harvest)	Research lance	0.31, 0.31, 0.31, 0.31 kg ai/100 L	Water 1204, 1203, 1203, 1200 L/ha	3.76 3.75 3.75 3.74 kg ai/ha	16/05/01 10/07/01 23/08/01 10/01/02	BBCH 88	Olive pulp	0 20	20.98 13.50	0.02 0.46	
									Olives		9.80	0.47	Prior to processing
									Raw oil		3.22	<0.002	
									Refined oil		2.81	<0.002	
									Canned olives		0.81	<0.002	6 month (sterilised)
									Canned brine		0.39	<0.002	6 month (sterilised)
									Canned olives		3.67	0.07	6 month (non-sterilised)

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment			Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
									Canned brine		2.26	0.04	6 month (non-sterilised)
	Olives, Villalonga (Pedralba, Spain)	1998 (planting) 14/11/01 (harvest)	Research lance	0.31, 0.31, 0.31, 0.31 kg ai/100 L	Water 1206, 1199, 1228, 1221 L/ha	3.76 3.74 3.83 3.81 kg ai/ha	22/05/01 5/07/01 9/08/01 25/10/01	BBCH 85	Olive pulp	0 20	12.34 8.95	0.14 1.63	
									Olives		10.72	1.45	Prior to processing
									Raw oil		2.72	<0.002	
									Refined oil		2.68	<0.002	
									Canned olives		0.70	0.01	6 month (sterilised)
									Canned brine		0.23	0.01	6 month (sterilised)
									Canned olives		2.61	0.21	6 month (non-sterilised)
									Canned brine		1.39	0.12	6 month (non-sterilised)
													Controls from trial 1 contained up to 0.14 mg/kg dimethoate, 0.08 mg/kg omethoate; dimethoate recoveries 70–110%, omethoate recoveries 70–110%; samples stored for up to 17 weeks before analysis.

No. 8376; Wilson, A; Residue study (processed commodity) with an EC formulation containing 400 g/L dimethoate applied to wheat in the United Kingdom and Germany in 2001; 14 February 2003

Table 112: STUDY DESCRIPTION

Active Substance (common name)	Dimethoate	**Commercial Product name**	Perfekthion
Crop/Crop Group	Wheat, cereals	**Producer of Commercial Product**	BASF
Responsible body for Reporting (name, address)	Huntingdon Life Sciences Ltd	**Indoor/glasshouse/other**	Field
Country:	Germany, UK	**Other active substance in the formulation (common name and content)**	N/A
Content of Active Substance (g/kg or g/L)	400 g/L	**Residues calculated as**	Dimethoate + Omethoate
Formulation (eg WP)	EC		

Table 113: STUDY RESULTS

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment			Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
No. 8376, Germany, UK	Wheat, Aristos (Brunne, Germany)	21/09/00 (sowing) 31/07/01 (harvest)	Research boom	0.85, 0.85 kg ai/100 L	Water 201, 203 L/ha	1.78, 1.80 kg ai/ha	3/07/01 10/07/01	BBCH 77	Straw	16	2.09	0.04	Field sample
									Grain	16	0.157	0.017	Field sample
									Grain		0.005	<0.001	Before processing
									Grain		0.004	<0.0002	After cleaning
									Screenings		0.07	0.003	
									Whole-meal		0.016	<0.001	
									Middlings		0.015	<0.001	
									White flour		0.004	<0.001	
									Bran		0.076	0.005	
									Germ-rich		0.048	0.003	Wheatgerm
									Toppings		0.024	<0.001	
									Type 550 flour		0.003	<0.001	Blended white flour/toppings
									Bread		0.027	0.002	Wholemeal
	Wheat, Pegassos (Blumberg, Germany)	6/10/00 (sowing) 30/07/01 (harvest)	Research boom	0.85, 0.85 kg ai/100 L	Water 202, 199 L/ha	1.78, 1.76 kg ai/ha	4/07/01 12/07/01	BBCH 77	Straw	32	1.08	0.02	Field sample
									Grain	32	0.08	0.003	Field sample
									Grain		0.075	0.002	Before processing
									Whole-meal		0.051	0.002	
									White flour		0.01	<0.001	

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment			Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
									Bran		0.363	0.01	
									Germ-rich		0.225	0.008	Wheatgerm
									Toppings		0.094	0.002	
									Type 550 flour		0.009	<0.001	Blended white flour/toppings
									Bread		0.145	0.005	Wholemeal
	Wheat, Claire (Manning-tree, UK)	25/09/00 (sowing) 1/08/01 (harvest)	Research boom	0.85, 0.85 kg ai/100 L	Water 215, 208 L/ha	1.90, 1.84 kg ai/ha	5/07/01 16/07/01	BBCH 77/83	Straw	22	2.55	0.05	Field sample
									Grain	22	0.022	0.005	Field sample
									Grain		0.013	0.002	Before processing
									Whole-meal		0.008	<0.001	
									White flour		0.004	<0.001	
									Bran		0.052	0.007	
									Germ-rich		0.037	0.004	Wheatgerm
									Toppings		0.015	0.001	
									Type 550 flour		0.003	<0.001	Blended white flour/toppings
									Bread		0.023	0.003	Wholemeal
	Wheat, Hereward (Tendring, UK)	Oct.-00 (sowing) 14/08/01 (harvest)	Research boom	0.85, 0.85 kg ai/100 L	Water 202, 211 L/ha	1.78, 1.85 kg ai/ha	6/07/01 13/07/01	BBCH 77/83	Straw	18	1.31	0.02	Field sample
									Grain	18	0.034	0.007	Field sample
									Grain		0.018	0.003	Before processing

Report No. Location (Region)	Commodity/ Variety	Date of Sowing or Planting, Flowering, Harvest	Method of Treatment	Application Rate per Treatment	Dates of treatments or no. of treatments and last date	Growth Stage at last treatment or date	Portion Analysed	PHI (days)	Dimethoate (mg/kg)	Omethoate (mg/kg)	Remarks
							Whole-meal		0.004	<0.001	
							White flour		0.001	<0.001	
							Bran		0.025	0.004	
							Germ-rich		0.015	0.002	Wheatgerm
							Toppings		0.007	<0.001	
							Type 550 flour		<0.001	<0.001	Blended white flour/toppings
							Bread		0.013	0.001	Wholemeal
											Controls <0.001 mg/kg except germ-rich fraction from trial 3 which contained 0.002 mg/kg dimethoate; dimethoate recoveries 70–110%, omethoate recoveries 70–94%; samples stored for up to 19 weeks before analysis.

A summary of the transfer factors (mean of all trials) is shown in Table 114.

Table 114: Transfer factors for dimethoate and omethoate in wheat

Processed commodity	Mean transfer factor (dimethoate)	Mean transfer factor (omethoate)
Field grain (RAC)	1.00	1.00
Cleaned grain	0.03	0.01
Screenings	0.45	0.18
Wholemeal	0.31	0.27
Middlings	0.10	0.06
White flour	0.09	0.18
Cleaned bran	2.03	1.40
Germ-rich fraction	1.31	0.99
Toppings	0.56	0.27
Type 550 flour	0.08	0.18
Wholemeal bread	0.85	0.63

For residues <LOQ, 0.001 mg/kg has been used for calculation purposes; For residues <LOD, 0.002 mg/kg has been used for calculation purposes.

APPENDIX 5: STORAGE STABILITY

No. 8377; Williams, B B; Freezer storage stability study for dimethoate and its oxygen analog, Omethoate, in raw agricultural commodities; 22 August 1994

A study was conducted to investigate the freezer storage stability of dimethoate and omethoate in or on potato tubers, orange fruit, sorghum grain, sorghum forage and cottonseed. Samples were homogenised and fortified with either dimethoate at 1 mg/kg or omethoate at 0.5 mg/kg. Samples were stored in the dark in a freezer at temperatures between –10 and –20^{o}C. Samples were taken for analysis after nominal 0, 1, 2, 4 and 6 month intervals, except cottonseed which was analysed after 2, 4 and 6 month intervals.

Samples (except cottonseed) were extracted with acetone and filtered. Extracts were partitioned with methylene chloride and cleaned up with a charcoal/celite column. Cottonseed samples were extracted with ethyl acetate using a Soxhlet extractor and cleaned up by gel permeation chromatography. Residues were quantified by gas chromatography with a flame photometric detector in the phosphorus mode. Recoveries of dimethoate and omethoate from samples of potato tubers, orange fruit, sorghum forage, sorghum grain and cottonseed fortified at 0.01, 0.05 and 0.5 mg/kg were within acceptable limits as summarised in Table 115.

Table 115: Method validation data for dimethoate and omethoate

Matrix	Number of samples	Dimethoate			Omethoate		
		Recovery range (%)	Mean (%)	SD (%)	Recovery range (%)	Mean (%)	SD (%)
Potato tuber	13	85–98	92	4	83–130	101	14
Whole orange fruit	15	79–110	96	8	81–120	95	11
Sorghum grain	14	81–97	87	5	75–97	87	7
Sorghum forage	14	73–101	84	7	71–104	89	8
Delinted cottonseed	8	90–110	97	6	49–80	63	12

A summary of the storage stability data is given in Table 116.

Table 116: Storage stability of dimethoate and omethoate in potato, orange, sorghum grain and forage, and cottonseed

Crop	Interval (days)	Dimethoate		Omethoate	
		Fortification level (mg/kg)	Average corrected recovery (%)	Fortification level (mg/kg)	Average corrected recovery (%)
Potato	0	1.0	100	0.50	100
	39	1.0	91	0.50	93
	70	1.0	96	0.50	97
	137	1.0	90	0.50	91
	188	1.0	95	0.50	96

Crop	Interval (days)	Dimethoate		Omethoate	
		Fortification level (mg/kg)	Average corrected recovery (%)	Fortification level (mg/kg)	Average corrected recovery (%)
Orange	0	1.0	100	0.50	100
	39	1.0	94	0.50	95
	70	1.0	106	0.50	104
	137	1.0	90	0.50	99
	188	1.0	98	0.50	101
Sorghum grain	0	1.0	100	0.50	100
	34	1.0	103	0.50	78
	67	1.0	80	0.50	91
	137	1.0	96	0.50	92
	185	1.0	98	0.50	96
Sorghum forage	0	1.0	100	0.50	100
	36	1.0	90	0.50	104
	69	1.0	95	0.50	94
	139	1.0	93	0.50	89
	187	1.0	97	0.50	100
Cottonseed	82	1.0	96	0.50	87
	126	1.0	95	0.50	79
	189	1.0	89	0.50	76

No. 8378, CHA Doc. No. 455 DMT; Harper, H; Dimethoate and omethoate: storage stability in cherries at approximately -18°; 2001

Untreated cherry samples were fortified individually with either dimethoate or omethoate at 0.1 mg/kg and placed in storage at –18°C. Samples were analysed at 0 days after fortification and after 6 months of storage. Residues were extracted by homogenisation in dichloromethane. An aliquot was evaporated to dryness and resuspended in hexane. Residues were extracted into water, washed with a further aliquot of hexane and quantified by LC-MS. Results for the storage stability samples and also the procedural recoveries are shown in Table 117.

Table 117:

	Recovery of dimethoate (%)			Recovery of omethoate (%)		
	Time 0	Time 6 months	Procedural recoveries	Time 0	Time 6 months	Procedural recoveries
Cherries	104, 104	103, 106	95, 97	94, 95	92, 84	96, 93

APPENDIX 6: ANALYTICAL METHODS

No. 8351; CHA Doc. No. 457 DMT; Harper, H; Dimethoate and omethoate: validation of methodology for the determination of residues in apples, artichokes, celery, cherries , lettuce, tomatoes, wheat (grain, green plant and straw), sugar beets (tops and roots), asparagus and melons (peel and pulp); 2001

An analytical method was developed and validated for the determination of dimethoate and omethoate in apples, artichokes, celery, cherries, lettuce, tomatoes, wheat (grain, green plant, straw), sugar beet (tops and roots), asparagus and melons (peel and pulp).

The method involves maceration of samples in dichloromethane. The extract is removed after centrifuging and the solids re-extracted. An aliquot of the extract is evaporated to dryness and the residue re-suspended in hexane. The residues are extracted into water and after washing with a further aliquot of hexane are quantified by LC-MS.

The LOQ for the method is 0.01 mg/kg for dimethoate and omethoate in all matrices. The LOD is 0.002 mg/kg. The method was validated over the range of 0.01 to 1.0 mg/kg for all matrices as summarised below.

Table 118: Recovery data for dimethoate

Matrix	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Apples	84–109	99	7.5	7.6
Artichokes	86–107	96	5.9	6.2
Celery	87–110	97	7.5	7.7
Cherries	85–106	99	6.5	6.6
Lettuce	89–109	100	6.0	5.9
Tomatoes	86–108	97	6.7	6.9
Wheat (grain)	71–104	90	10.0	11.1
Wheat (green plant)	75–109	92	12.3	13.4
Wheat (straw)	73–109	92	10.4	11.3
Sugar beet (tops)	92–109	100	5.4	5.4
Sugar beet (roots)	92–110	102	5.4	5.3
Asparagus	83–110	99	8.7	8.8
Melon (peel)	81–108	94	8.5	9.1
Melon (pulp)	87–106	98	4.8	4.9

Table 119: Recovery data for omethoate

Matrix	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Apples	70–106	92	9.7	10.5
Artichokes	87–109	96	7.5	7.8
Celery	79–110	96	8.8	9.2
Cherries	70–110	90	15.2	17.0
Lettuce	73–109	95	13.4	14.1
Tomatoes	78–105	88	9.9	11.3
Wheat (grain)	81–110	100	10.2	10.2
Wheat (green plant)	70–109	89	14.0	15.7
Wheat (straw)	71–104	91	11.6	12.7
Sugar beet (tops)	82–99	90	5.7	6.4
Sugar beet (roots)	84–105	95	6.9	7.3
Asparagus	82–110	95	7.9	8.4
Melon (peel)	73–109	88	13.6	15.4
Melon (pulp)	71–102	76	8.4	11.1

No. 8352; Harper, H; Dimethoate and omethoate: validation of methodology for the determination of residues in bran, bread, flour, wheatgerm and wheat (grain); 26 November 2001

An analytical method was developed and validated for the determination of dimethoate and omethoate in bran, bread, flour, wheatgerm and wheat (grain).

The method involves maceration of the sample in dichloromethane. The extract is removed and the maceration of the solids repeated in a second aliquot of dichlorormethane. The combined extract is passed through an ENVI-Carb bond elut cartridge and evaporated to dryness. The sample is resuspended in hexane and extracted with water and the aqueous extract washed with hexane prior to quantitation by LC-MS. The LOQ for the method is 0.001 mg/kg for dimethoate and omethoate in all matrices. The LOD for the method is 0.0002 mg/kg for both matrices. The method has been validated over the range 0.001 to 0.1 mg/kg for bran, bread, flour and wheatgerm as summarised in Table 120.

Table 120: Recovery data for dimethoate

Matrix	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Bran	77–94	84	7.1	8.5
Bread	79–104	90	8.5	9.4
Flour	80–93	89	4.9	5.5
Wheatgerm	82–100	93	7.8	8.4
Wheat (grain)	89–101	97	5.0	5.2

Table 121: Recovery data for omethoate

Matrix	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Bran	76–92	84	6.1	7.3
Bread	70–87	80	7.7	9.7
Flour	79–104	90	10.0	11.2
Wheatgerm	70–93	81	9.8	12.1
Wheat (grain)	76–80	78	1.6	2.0

No. 8353; Flatt, S G; Dimethoate and omethoate: validation of the method of analysis for determination of residual concentrations in whole plant, leaves and root of sugarbeet; 4 September 1995

The purpose of this study was to validate a method for the determination of dimethoate and omethoate in whole plant, leaves and root of sugar beet.

The method involves maceration of the samples in aqueous acetone. After filtering, residues in the extract are purified by liquid-liquid partition with dichloromethane followed by further purification using activated charcoal. Quantitation of dimethoate and omethoate is by gas chromatography using a flame photometric detector. The limit of determination for both compounds in all matrices was 0.01 mg/kg. Recoveries of dimethoate and omethoate from fortified samples of whole plants, leaves and roots were generally within acceptable limits, as summarised in tables 122 and 123.

Table 122: Dimethoate recoveries

Matrix	Fortification level (mg/kg)	Recovery range (%)	Mean recovery (%)	SD (%)	CV (%)
Sugar beet, whole plant	0.01	93–112	103	9.1	8.8
	0.20	104–116	107	4.7	4.4
	5.0	95–103	101	3.1	3.1
Sugar beet leaves	0.01	94–116	107	8.2	7.7
	0.20	96–116	107	7.2	6.7
	5.0	98–106	102	2.7	2.7
Sugar beet roots	0.01	69–108	94	15.8	16.9
	0.2	88–113	102	9.6	9.4

Table 123: Omethoate recoveries

Matrix	Fortification level (mg/kg)	Recovery range (%)	Mean recovery (%)	SD (%)	CV (%)
Sugar beet, whole plant	0.01	71–116	91	14.7	16.1
	0.20	96–107	101	4	4
	5.0	76–97	90	8.4	9.4
Sugar beet leaves	0.01	79–108	94	10.3	10.9
	0.20	81–107	97	10.7	11
	5.0	86–99	94	5.2	5.6
Sugar beet roots	0.01	79–104	91	8.7	9.6
	0.2	77–104	90	10.7	11.7

No. 8354, CHA Doc. No. 454 DMT; Dimethoate and omethoate: validation of methodology for the determination of residues of dimethoate and omethoate in olives (RAC); 2001

The purpose of this study was to develop and validate an analytical method for the determination of dimethoate and omethoate in olive flesh and oil. Olive flesh samples were macerated in dichloromethane until homogenous. The extract solution was removed and the procedure repeated. Olive oil samples were simply diluted with dichloromethane. An aliquot of the combined olive flesh extracts or the diluted oil solution was evaporated to dryness and resuspended in hexane. The residues were extracted into water and washed with hexane prior to analysis by LC-MS. The LOQ for the method was 0.01 mg/kg for dimethoate and omethoate in both matrices. The LOD was 0.002 mg/kg. Validation data are summarised in tables 124 and 125.

Table 124: Dimethoate recovery data

Matrix	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Olive flesh	70–100	81	11.4	14.2
Olive oil	76–108	93	9.7	10.5

Table 125: Omethoate recovery data

Matrix	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Olive flesh	81–110	100	10.8	10.8
Olive oil	94–109	104	3.9	3.7

No. 8355; Jalali, K; Hiler, R L; Validation and radiovalidation of the analytical residue method for the determination of dimethoate and omethoate in goat and hen tissues, milk and eggs; 2 December 1998

The purpose of this study was to validate an analytical method for the determination of dimethoate and omethoate in goat and hen tissues, milk and eggs. Radiovalidation was also investigated by evaluating the recovery of incurred ^{14}C-omethoate residues in goat liver and hen egg white samples obtained from animal metabolism studies.

The method involves an initial extraction with acetone or acetonitrile followed by a protease enzyme treatment of the dried post extraction solids. The combined acetone extract and enzyme hydrolysate are partitioned with dichloromethane. The organic layer is subjected to several steps including solid phase extraction and evaporation and re-suspension in benzene. Analysis is by GC-FPD. The method's limit of quantitation is 0.001 mg/kg for milk and eggs, and 0.01 mg/kg for tissues. Recoveries of dimethoate and omethoate from fortified samples of milk, eggs and tissues were generally within acceptable limits, except for the recovery of omethoate from egg whites fortified at 0.001 mg/kg (mean recovery 64%). Recoveries are summarised in Table 126.

Table 126: Recoveries of dimethoate and omethoate from fortified tissues, milk and eggs

Sample	Fortification level (mg/kg)	Dimethoate % Recovery	Omethoate % Recovery
Milk	0.001	115±5 (118.9, 112)	100±3 (101.9, 97.6)
	0.01	102±7 (107.1, 97.2)	88.5±4 (91.1, 85.7)
	0.1	92±4 (95.1, 88.9)	90±2 (92.0, 88.8)
Liver	0.01	83±6 (87.4, 79.2)	93±17 (105.0, 81.2)
	0.05	102±16 (90.8, 113.1)	95±21 (80.3, 110.5)
	0.5	101±6 (105.3, 96.8)	92±3 (94.7, 90.2)
Kidney	0.01	120±4 (122.9, 116.6)	114±15 (124.4, 103.2)
	0.05	114±2 (115.6, 112.3)	100±4 (103.3, 97.4)
	0.5	100±10 (93.3, 107.4)	105±20 (91.2, 119.1)
Fat	0.01	116±5 (119.6, 113.1)	89±4 (91.3, 85.8)
	0.05	91±3 (88.3, 93.2)	80±3 (77.5, 81.8)
	0.5	118±2 (119.5, 117.2)	87±18 (100.0, 74.4)
Muscle	0.01	93±20 (78.7, 106.4)	92±6 (88.4, 96.2)
	0.05	101±13 (109.8, 91.9)	93±13 (102.2, 84.3)
	0.5	100±12 (107.8, 91.4)	94±5 (98.3, 90.6)
Whole eggs	0.05	76±2 (75.0, 77.6)	81±0 (81.3, 80.8)
	0.5	84±1 (85.4, 83.6)	85±9 (78.5, 91.6)
Egg whites	0.001	92±12 (100.1, 83.6)	64±5 (59.8, 67.2)
	0.01	96±10 (89.5, 103.5)	93±17 (80.9, 105.4)
	0.1	102±1 (102.8, 102.0)	86±1 (86.2, 84.8)

In order to radiovalidate the method, goat liver and hen egg white samples containing incurred ^{14}C-dimethoate residues (from the animal metabolism study) were analysed for omethoate by the GC-FPD method and also the HPLC radiochromatographic method used in the metabolism study. The results are summarised in Table 127.

Table 127: Incurred ^{14}C-omethoate residues detected in goat liver and hen egg whites

Matrix	TRR (mg/kg)	Residue analytical method expressed as mg/kg (% TRR)	Metabolism method expressed as mg/kg (% TRR)
Goat liver	0.975	0.001 (0.1%)	0.009 (0.9%)
Hen egg whites	0.133	<0.001 (<0.8%)	0.003 (2.3%)

The study suggested residue levels determined by the two methods were in general agreement, although the GC method appeared to result in lower recoveries.

Storage stability was also investigated by comparing the data collected from the original metabolism study with those from the current study (storage interval not stated). The data (Table 128) show that, while the TRR and the amount of omethoate in egg whites is relatively unchanged over time, omethoate residues in liver degrade over time, with very little remaining by the time the validation study was conducted.

Table 128: Storage stability: Comparison of residue levels in metabolism study (A) with those found in current study (B)

Sample	TRR A	TRR B	% extracted A	% extracted B	Omethoate level A (mg/kg)	Omethoate level B (mg/kg)
Liver	1.221	0.975	64.9	60.8	0.120	0.009
Egg whites	0.127	0.133	64.4	72.2	0.004	0.003

No. 8356; Anonymous; Determination of dimethoate and omethoate residues in the raw agricultural commodities of grain sorghum by gas chromatography; 6 June 1994

A method was developed for the detection of dimethoate and omethoate in sorghum grain, forage and hay samples. Residues are extracted with acetone. The residues are partitioned into methylene chloride. The extracts are cleaned up on a celite/activated charcoal column. Residues are suspended in acetone prior to analysis by gas chromatography with a flame photometric detector in the phosphorus mode. Results are calculated by interpolation from a standard curve. The LOQ for the method is 0.01 mg/kg. Recoveries were determined for samples of grain, forage and hay fortified at 0.01, 0.05 and 0.5 mg/kg as summarised below.

Table 129: Recoveries of dimethoate and omethoate from fortified sorghum samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Grain	80–90	83	4	80–90	85	4
Forage	80–90	86	4	87–100	91	5
Hay	75–100	85	8	83–100	93	7

No. 8357; Anonymous; Determination of dimethoate and omethoate residues in the raw agricultural commodities of peas by gas chromatography; 9 June 1994

A method was developed for the detection of dimethoate and omethoate in peas (peas plus pods), vine and hay samples. Residues are extracted with acetone. The residues are partitioned into methylene chloride. The extracts are cleaned up on a celite/activated charcoal column. Residues are suspended in acetone to analysis by gas chromatography with a flame photometric detector in the phosphorus mode. Results are calculated by interpolation from a standard curve. The LOQ for the method is 0.01 mg/kg. Recoveries were determined for samples of peas and pods, vines and hay fortified at 0.01, 0.05 and 0.5 mg/kg as summarised in Table 130.

Table 130: Recoveries of dimethoate and omethoate from fortified pea samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Peas + pods	90–110	99	8	91–120	104	12
Vines	79–100	88	9	83–120	98	16
Hay	70–82	75	5	74–120	83	9

No. 8358; Anonymous; Determination of dimethoate and omethoate residues in wheat grain and its processed commodities by gas chromatography, 10 June 1994

A method was developed for the detection of dimethoate and omethoate residues in wheat grain and its processed commodities. Residues are extracted from the crop samples with acetone:water and partitioned into methylene chloride. The extracts are cleaned up on a celite/activated charcoal column and suspended in acetone for analysis by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of wheat grain, bran, middlings, shorts and patent flour fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 131.

Table 131: Recoveries of dimethoate and omethoate from fortified wheat samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Grain	90–96	92	3	103–120	110	7
Bran	90–98	93	3	96–109	103	5
Middlings	80–99	93	6	91–100	97	3
Shorts	90–102	95	5	92–120	106	11
Patent flour	70–140	92	23	73–124	93	17

No. 8359; Anonymous; Determination of dimethoate and omethoate residues in corn grain and its processed commodities by gas chromatography; 28 June 1994

A method was developed for the detection of dimethoate and omethoate residues in corn grain and its processed commodities. For samples other than corn oil, residues are extracted with acetone and partitioned into methylene chloride. The extracts are cleaned up on a celite/activated charcoal column. For corn oil, residues are extracted with ethyl acetate and cleaned up by gel permeation chromatography. Residues are suspended in acetone prior to quantification by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of corn grain, grits, meal, flour, starch and oil fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 132.

Table 132: Recoveries of dimethoate and omethoate from fortified corn samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Grain	80–92	87	5	77–100	86	10
Grits	87–94	90	2	83–110	92	9
Meal	76–100	88	8	74–100	88	8
Flour	91–100	96	4	82–120	101	15
Starch	80–90	86	4	80–100	86	6
Oil	78–91	87	5	66–100	78	13

No. 8360; Anonymous; Determination of dimethoate and omethoate residues in oranges and its processed commodities by gas chromatography; 14 July 1994

A method was developed for the determination of dimethoate and omethoate residues in oranges and its processed commodities. Samples other than orange oil were extracted with acetone/water. Residues are partitioned into methylene chloride and cleaned up on a celite/activated charcoal column. Orange oil samples are diluted in cyclohexane/methylene chloride (85:15) and cleaned up by gel permeation chromatography. Residues are suspended in acetone prior to analysis by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of whole oranges, juice, dry pulp, molasses and oil samples fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 133.

Table 133: Recoveries of dimethoate and omethoate from fortified orange samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Whole oranges	95–110	101	5	82–120	100	12
Juice	102–110	107	4	90–107	99	5
Dry pulp	69–84	79	5	63–80	72	6
Molasses	70–100	87	12	66–101	90	12
Oil	80–100	93	7	65–120	79	21

No. 8361; Anonymous; Determination of dimethoate and omethoate residues in potatoes and processed commodities by gas chromatography; 10 June 1994

A method was developed for the determination of dimethoate and omethoate residues in potatoes and its processed commodities. For samples other than chips, residues are extracted with acetone and partitioned into methylene chloride. The extracts are cleaned up on a celite/activated charcoal column. For chips, residues are extracted with ethyl acetate and cleaned up by gel permeation chromatography. Residues are

suspended in acetone prior to quantification by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of tubers, granules, chips, wet peel and dry peel fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 134.

Table 134: Recoveries of dimethoate and omethoate from fortified potato samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Tubers	90–98	93	3	100–130	114	10
Granules	90–106	98	6	101–130	112	9
Chips	91–120	100	11	77–130	96	21
Wet peel	80–97	89	5	91–120	101	13
Dry peel	80–93	83	5	76–100	87	8

No. 8362; Anonymous; Determination of dimethoate and omethoate residues in tomatoes and its processed commodities by gas chromatography; 18 January 1995

A method was developed for the determination of dimethoate and omethoate residues in tomatoes and their processed commodities. Samples are extracted with acetone/water and residues partitioned into methylene chloride. Residues are cleaned up using a celite/activated charcoal column and suspended in acetone prior to quantification by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of tomatoes, paste and dry pomace fortified at 0.01, 0.05 and 0.5 mg/kg (and also 5 mg/kg for paste) are summarised below.

Table 135: Recoveries of dimethoate and omethoate from fortified tomato samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Whole tomatoes	80–96	89	7	100–116	110	5
Paste	90–110	98	7	60–107	88	18
Dry pomace	70–104	87	11	70–114	97	17

No. 8363; Anonymous; Determination of dimethoate and omethoate residues in soil by gas chromatography; 27 September 1994

A method was developed for the determination of dimethoate and omethoate residues in soil. Residues are extracted from soil with acetone/water (95:5). An aliquot of the extract is concentrated by rotary evaporation and residues partitioned into methylene chloride. Residues are cleaned up on a celite/activated charcoal column and suspended in acetone prior to quantification by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of soil fortified at 0.01, 0.05, 0.10 and 1.0 mg/kg are summarised in Table 136.

Table 136: Recoveries of dimethoate and omethoate from fortified soil samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Soil	82–110	90.9	6.7	80–100	86.0	6.2

No. 8364; Anonymous; Determination of dimethoate and omethoate residues in soil by gas chromatography; 2 September 1994

This method for the determination of dimethoate and omethoate residues in soil is the same as that summarised in the previous study (No. 8363). Recoveries of dimethoate and omethoate from samples of soil fortified at 0.01, 0.05, 0.10 and 1.0 mg/kg are summarised in Table 137.

Table 137: Recoveries of dimethoate and omethoate from fortified soil samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Soil	70–112	93.2	12.8	66–90	76.4	8.0

No. 8365; Anonymous; Determination of dimethoate and omethoate residues in the raw agricultural commodities of beans by gas chromatography; 24 January 1995

A method was developed for the determination of dimethoate and omethoate residues in beans (succulent and dry), forage and straw samples. Residues are extracted from samples with acetone and partitioned into methylene chloride. The extracts are cleaned up on a celite/activated charcoal column and suspended in acetone prior to quantification by gas chromatography with a flame photometric detector in the phosphorus mode. The LOQ for the method is 0.01 mg/kg. Recoveries of dimethoate and omethoate from samples of succulent bean, forage and straw samples fortified at 0.01, 0.05 and 0.5 mg/kg are summarised in Table 138.

Table 138: Recoveries of dimethoate and omethoate from fortified soil samples

Matrix	Dimethoate			Omethoate		
	Recovery range (%)	Mean recovery (%)	SD (%)	Recovery range (%)	Mean recovery (%)	SD (%)
Beans	90–98	92	3	105–120	111	6
Forage	80–95	89	5	94–108	100	4
Straw	86–100	92	4	80–110	96	11

CHA Doc. No. 315 DMT; Flatt, S G, Gillis, N A; Dimethoate and omethoate: validation of method of analysis for determination of residual concentrations in whole plant and tubers of potatoes; 1996

The purpose of this study was to validate a method for the determination of dimethoate and omethoate in whole plant and tubers of potatoes. The method involves maceration of samples with aqueous acetone. After filtration residues are liquid–liquid partitioned with dichloromethane followed by further purification with activated charcoal. Quantitation is by gas chromatography with a flame photometric detector. The LOQ for the method is 0.01 mg/kg for both dimethoate and omethoate.

Recoveries for the method are summarised below.

Table 139: Dimethoate recoveries

Matrix	Fortification level (mg/kg)	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Whole plant	0.01	77–131	96	18.5	19.2
	0.21	101–106	104	2.2	2.1
	5.2	94–107	100	4.7	4.7
Tubers	0.01	87–107	95	8.3	8.7
	0.21	96–112	104	7.0	6.7

Table 140: Omethoate recoveries

Matrix	Fortification level (mg/kg)	Recovery range (%)	Mean recovery (%)	Standard deviation (%)	Coefficient of variation (%)
Whole plant	0.01	80–138	100	17.2	17.1
	0.20	86–103	96	5.8	6.1
	5.1	86–101	91	6.0	6.5
Tubers	0.01	79–103	92	8.8	9.5
	0.20	80–107	94	11	11.7

CHA Doc. No. 463 DMT; Cordon, C; Dimethoate and omethoate: validation of methodology for the determination of residues of dimethoate and omethoate in citrus; 2001

The purpose of this study was to validate a method for the determination of dimethoate and omethoate in citrus whole fruit, peel and pulp. For orange commodities the sample homogenate is extracted using acetone. Residues are then partitioned into dichloromethane and cleaned up using activated charcoal and column chromatography with silica gel. Quantitation is by gas chromatography with flame photometric detection (GC-FPD). For lemon commodities the sample homogenate is also extracted with acetone. An aliquot is concentrated by evaporation and reconstituted in hexane. The residues are partitioned into water

and quantified by liquid chromatography with mass spectrometric detection (LC-MS). The LOQ for both methods is 0.01 mg/kg for dimethoate and omethoate in all matrices. Validation data for the method are summarised below.

Table 141: Dimethoate recoveries

Matrix	Fortification range (mg/kg)	Recovery range (%)	Mean recovery (%)	Coefficient of variation (%)
Orange whole fruit (RAC)	0.01–1.00	76.1–95.8	87.4	6.10
Orange peel	0.01–1.00	70.6–90.7	80.7	6.85
Orange pulp	0.01–1.00	76.4–91.4	84.4	5.14
Lemon whole fruit (RAC)	0.01–1.00	78.0–110	95.1	7.73
Lemon peel	0.01–1.00	92.0–110	102	5.65
Lemon pulp	0.01–1.00	80.0–111	100	10.2

Table 142: Omethoate recoveries

Matrix	Fortification range (mg/kg)	Recovery range (%)	Mean recovery (%)	Coefficient of variation (%)
Orange whole fruit (RAC)	0.01–1.00	77.0–99.6[a]	86.8	7.74
Orange peel	0.01–1.00	66.2–93.6	79.4	8.90
Orange pulp	0.01–1.00	67.9–96.1	82.5	10.5
Lemon whole fruit (RAC)	0.01–1.00	91.0–108	101	5.90
Lemon peel	0.01–1.00	95.9–109	103	4.90
Lemon pulp	0.01–1.00	84.0–103	92.5	5.60

a A recovery of 126% at 0.01 mg/kg was considered an outlier.

CHA Doc. No. 703 DMT; Jones, A; Development of analytical methodology to determine omethoate and dimethoate in olives, olive oil, oranges, lettuce and wheat grain; 2002

Methods were developed for the determination of omethoate and dimethoate in olive flesh, olive oil, orange whole fruit, lettuce and wheat grain. Homogenised samples of olives, oranges, lettuce and wheat grain were extracted using ethyl acetate with the addition of sodium sulphate and sodium hydrogen carbonate, and equilibration at 40^{o}C. Extracts were cleaned up by gel permeation chromatography (GPC) with determination by GC-FPD. For olives, clean up by GPC alone was evaluated as well as an additional clean involving partition between hexane and acetonitrile prior to GPC. For olive oil the method involved dissolution in hexane and partition between hexane and acetonitrile prior to GPC. Determination was again by GC-FPD.

Recovery data (single point only) for samples fortified at the LOQ (0.01 mg/kg) are summarised in tables 143 and 144 for the two different columns used in the study.

Table 143: Recoveries determined on DB-17 column

Commodity	Dimethoate recovery (%)	Omethoate Recovery (%)
Olives (GPC only)	77	70
Olives (partition + GPC)	88	72
Olive oil	88	84
Orange	90	73
Lettuce	83	77
Wheat grain	100	70

Table 144: Recoveries determined on DB-1701 column

Commodity	Dimethoate recovery (%)	Omethoate Recovery (%)
Olives (GPC only)	80	77
Olives (partition + GPC)	84	72
Olive oil	80	80
Orange	90	70
Lettuce	80	73
Wheat grain	110	80

CHA Doc. No. 577 DMT; Jones, A; Validation of analytical methodology for the determination of dimethoate and omethoate in olives, olive oil, oranges, lettuce and wheat grain; 2003

The purpose of the study was to validate a method for the determination of dimethoate and omethoate in olive flesh, olive oil, orange whole fruit, lettuce and wheat grain. The method for olives, oranges, lettuce and wheat grain involves extraction with ethyl acetate followed by clean up using gel permeation chromatography. Olive oil is extracted with acetonitrile after dissolution with hexane. Residues are quantified by GC-FPD. The LOQ for the method is 0.01 mg/kg for dimethoate and omethoate in all matrices. Validation data are summarised in tables 145 and 146.

Table 145: Dimethoate

Matrix	Fortification level (mg/kg)	Recovery range (%)	Mean recovery (%)	% RSD
Olives	0.01–0.1	79.8–90.6	85.8	3.6
Olive oil	0.01–0.1	73.8–95.0	82.5	7.8
Oranges	0.01–0.1	79.1–90.6	84.0	3.7
Lettuce	0.01–0.1	74.8–83.8	77.5	3.1
Wheat grain	0.01–0.1	66.8–83.0	73.7	6.7

Table 146: Omethoate

Matrix	Fortification level (mg/kg)	Recovery range (%)	Mean recovery (%)	% RSD
Olives	0.01–0.1	61.4–87.9	77.1	11.7
Olive oil	0.01–0.1	66.5–91.4	77.7	9.2
Oranges	0.01–0.1	69.4–89.1	76.3	8.7
Lettuce	0.01–0.1	79.4–91.4	85.0	5.4
Wheat grain	0.01–0.1	59.1–76.7	69.3	8.2

CHA Doc. No. 407 DMT; Schulz, H; Determination of the residues of dimethoate and omethoate in white cabbage (RAC and cooked) and water—validation of the method, 2000

The purpose of the study was to validate method AK/3377/CN for the determination of dimethoate in tap water, white cabbage (RAC) and white cabbage (cooked). The method involves extraction with ethyl acetate and purification of the extract by gel permeation chromatography. Quantification of dimethoate and omethoate is by gas chromatography using a flame photometric detector in the phosphorus mode or mass selective detection. The LOQ for the method was 0.01 mg/kg for both substances in the matrices tested. The validation data are summarised in tables 147 and 148.

Table 147: Dimethoate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	SD (%)	CV (%)
Tap water	0.01	84.0 (n = 5)	2.0	2.4
	5	85.0 (n = 5)	4.6	5.5
White cabbage (RAC)	0.01	81.2 (n = 5)	2.7	3.3
	5	79.2 (n = 5)	3.8	4.8
White cabbage (cooked)	0.01	73.4 (n = 5)	7.1	9.6
	5	87.0 (n = 5)	9.8	11.2

Table 148: Omethoate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	SD (%)	CV (%)
Tap water	0.01	100.8 (n = 5)	2.7	2.7
	5	90.2 (n = 5)	2.8	4.0
White cabbage (RAC)	0.01	90.2 (n =5)	3.7	4.1
	5	78.2 (n = 5)	5.9	7.6
White cabbage (cooked)	0.01	75.6 (n = 5)	6.8	9.0
	5	90.4 (n = 5)	12.5	13.8

ABBREVIATIONS FOR APPENDIXES

ai	active ingredient
AR	
BBCH	Biologische Bundesanstalt, Bundessortenamt and Chemical Industry (German scale used to identify the phenological development stages of a plant)
DALA	days after last application
EC	emulsifiable concentrate
GC	gas chromatography (to identify chemicals)
GC-MS	gas chromatography–mass spectrometry (to identify chemicals)
HPLC	high-performance liquid chromatography (to identify chemicals)
LC-MS	liquid chromatography–mass spectrometry (to identify chemicals)
LOD	Limit of detection
LOQ	Limit of Quantitation
LOR	limit of reporting
LSC	liquid scintillation counting
MRL	Maximum Residue Limit
NRS	National Residue Survey
PFBB	pentafluorobenzyl bromide (chemical used to identify metabolites of dimethoate)
RAC	raw agricultural commodity
(SAX) SPE	(Strong anion exchange) solid phase extraction- used to extract chemicals for analysis
SD	standard deviation
TLC	thin layer chromatography
TRR	total radioactive residue