OXYDEMETON-METHYL (166)

DEMETON-S-METHYL (073)

EXPLANATION

Oxydemeton-methyl (ODM) was evaluated for residues by the JMPR in 1968, 1973, 1979, 1984, 1989, and 1992. The 1992 review was a complete re-evaluation. It reviewed extensive residue data from supervised trials on all major crops and associated data on use patterns, storage stability, processing, and methods of residue analysis were reviewed and numerous MRLs were recommended. The MRLs are expressed as the sum oxydemeton-methyl, demeton-S-methyl, and demeton-S-methylsulphon, expressed as oxydemeton-methyl. The ADI was established in 1989 at 0.0003 mg/kg body weight and is for the sum of the three compounds.

Demeton-S-methyl is an insecticide. The sulfoxide of demeton-S-methyl is ODM. It currently has no MRLs.

The 1995 CCPR scheduled ODM and demeton-S-methyl for periodic review of residue aspects by the 1997 JMPR (ALINORM 95/24A). This was changed by the 1997 CCPR, which scheduled ODM and demeton-S-methyl for periodic review by the 1998 JMPR.

Bayer AG has submitted data in support of the Periodic Review which included information on crops and regions of interest to that company. The governments of Germany and The Netherlands have also submitted information.

IDENTITY

Common name (ISO): Oxydemeton-methyl

Chemical name:
IUPAC: *S*-2-ethylsulfinylethyl *O,O*-dimethyl phosphothioate
CA: *S*-[2-ethylsulfinyl)ethyl] *O,O*-dimethyl phosphothioate

CAS number: 301-12-2

EU-index number: 015-046-00-7

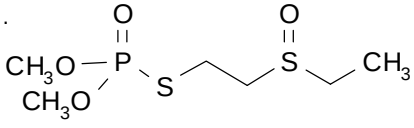
EINECS number: 206-110-7

CIPAC number: 171

Molecular formula: $C_6 H_{15} O_4 P S_2$

Synonyms: Metasystox R

Structural formula:



Molecular weight: 246.3 g/mol

Physical and chemical properties

Pure active ingredient:

Vapour pressure: 3.8×10^{-5} hPa at 20°C; 5.1×10^{-5} hPa at 25°C (Sevekow, 1980, Bayer AG Report PC884)

Boiling point: 106°C at 0.013 hPa
Melting point: < -10°C

Octanol/water partition coefficient: log P_{ow} = -0.74 at 21°C (Hellbusch, 1983)

Solubility:	water:	miscible in any ratio	
	n-hexane:	0.025 g/l	
	toluene:	>200 g/l	
	dichloromethane:	>200 g/l	
	2-propanol:	>200 g/l	
	1-octanol:	>200 g/l	
	polyethyleneglycol:	>200 g/l	
	acetone:	>200 g/l	
	dimethylformamide:	>200 g/l	
	ethyl acetate:	>200 g/l	
	acetonitrile:	>200 g/l	
		(all at 20°C)	(Krohn, 1987a,b)

Specific gravity: 1.29 g/cm^3 at 20°C (Krohn, 1986)

Hydrolysis:

Half-life, days of ODM in sterile aqueous buffer solution

pH	25°C	40°C
5	94	22
7	40	7.5
9	2.5	<1

(Pither and Puhl, 1978, 1989)

Photolysis: Half-life in pH 5 buffer in natural sunlight 137 days, in the absence of light 194 days. Photolytic processes contribute little to the decomposition (Kesterson *et al.*, 1988)

Technical material:

Purity: Oxydemeton-methyl (TGAI), batch 808306101-103, purity 89.0%.

Stability: Stable for 8 months at ambient temperature (as a 50% solution). The technical material is unstable (Bayer AG)

Formulations

Bayer AG provided a list of formulations for itsproducts. The list does not include products manufactured or distributed by other companies.

Product	Country	Formulation	Common name of ai	g ai/l
Metasystox R 50	Austria	48.5 EC	oxydemeton-methyl	50
Metasystox R 100	Sweden	100 EC	oxydemeton-methyl	100
Aphitox R 250 EC	South Africa	250 EC	oxydemeton-methyl	250
Metasystemox R	France		oxydemeton-methyl	
Metasystox 250	Ireland		oxydemeton-methyl	
Metasystox R	Austria Finland Germany Portugal Spain		oxydemeton-methyl	
Metasystox R EC 25	Morocco		oxydemeton-methyl	
Metasystox R 25	Mexico		oxydemeton-methyl	
Metasystox R 250 EC	South Africa		oxydemeton-methyl	
Enduro	France	258 EC	oxydemeton-methyl beta-cyfluthrin	250 8
Metasystox R 25 EC	Turkey	265 EC	oxydemeton-methyl	265
Ecombi	Germany	375 EC	oxydemeton-methyl parathion	200 175
Metasystox R 50 SL	Greece	50 SL	oxydemeton-methyl	50
Metasystox R spezial	Austria Germany	100 SL	oxydemeton-methyl	100
Metasystox R	Italy	188.7 SL	oxydemeton-methyl	188.7

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

The metabolism of [*ethylene*-1-^{14}C]oxydemeton-methyl was investigated with male and female Sprague-Dawley rats (Walter and Kane, 1989). The compound dissolved in deionized water was administered orally and intravenously at different dose levels to groups of 5 rats. In a pilot experiment doses of 20.0 mg/kg body weight were administered to 3 male and 2 female rats in order to measure radioactivity in the expired air.

Both oral and intravenous studies showed that <1% of the dose was present in the tissues and blood 72 hours after the final dosing. The highest concentrations, 0.15 mg/kg as ODM or 0.02% of the administered dose, were found in the blood of both female and male rats from a single oral dose of 20 mg/kg bw. Transformation to volatiles was a very minor pathway, 0.059%-0.062% of the dose. The major route of elimination was in the urine, reaching 89–105% at 72 hours after the final dose. More than 80% of the urinary radioactivity was eliminated during the first 24 hours.

Urine samples taken at 0–24 hours were combined and fractionated by reverse-phase HPLC. Isolated compounds were purified by HPLC, extraction or TLC and identified by mass spectrometry (direct chemical ionization and GC electron impact) and comparison with reference compounds.

Pooled faeces samples were extracted with methanol in a Soxhlet unit. The extracts were analysed by TLC and compared to authentic standards. Only the high dose (20 mg/kg bw) provided enough material (3% of the administered ^{14}C) for useful analysis.

The identified compounds are shown in Table 1.

Table 1. Compounds identified in urine and faeces of rats dosed orally or intravenously with ODM.

Sample	Compound	% of dose
Urine	ODM	50
	ODM sulfone [M 01]	2.2
	1-(ethylsulfinyl)-2-(methylsulfinyl)ethane ["sulfinyl" M 03]	16
	1-(ethylsulfonyl)-2-(methylsulfinyl)ethane ["sulfonyl" M 04]	16
	Demethyl ODM [M 06]	2.9
	Demethyl ODM sulfone [M 07]	2.1
	Unknown A	11
	Unknowns B–D	3.7
	Total identified	89%
Faeces	ODM	0.24
	ODM sulfone	0.21
	ODM sulfide	0.80
	Unknowns 1–3	0.62
	Unextracted	0.42
	Total identified	1.2%

The identified metabolites are consistent with a metabolic pathway that starts with hydrolysis of the thioester followed by *S*-methylation of the free thiol. Further oxidation of the sulfur results in metabolites M 03, 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane, and M 04, 1-(ethylsulfonyl)-2-(methylsulfinyl)ethane.

The metabolism of [*ethylene*-1-^{14}C]ODM in a lactating Swiss Alpine goat was studied by Merricks (1987). The treated goat (39 kg) and a control (29 kg) each received 1.5 kg of feed per day, which was completely consumed. The test goat was given one capsule containing lactose and [^{14}C]ODM diluted with natural abundance ODM to a specific activity of 22,543 dpm/µg each day after the morning milking for three consecutive days. Each capsule contained lactose and. Each capsule contained 273 mg of total ODM, corresponding to 7 mg ODM/kg body weight. The control goat received a placebo capsule each day. Milk was collected twice each day, and the pm milk of one day was added to the am milk of the following day. Urine and faeces were collected daily and stored frozen. The goats were slaughtered 2 hours after the third dosing, and liver, kidney, muscle (composite of loin, round, and flank) and fat (composite of omental, renal, and subcutaneous) were collected and stored frozen.

Tissues were homogenized, and subsamples (10 g) were mixed with water (15 ml) and centrifuged. The water extracts were purified on C-18 "Sep-pak" cartridges eluted with methanol. The water extracts contained virtually all of the radioactivity from all tissues except fat, where about 14% remained in the post-extraction solid. The C-18 clean-up recovered 83% (from fat) to 94% (from muscle) of the radioactivity in the extracts.

Milk samples were centrifuged and the supernatants again purified on C-18 "Sep-pak" cartridges. Virtually all of the radioactivity was transferred to the aqueous phase. The C-18 clean-up recovered about 80% of it.

Extracts were analysed by TLC and co-chromatographed with authentic reference standards (ODM, ODM sulfone, sulfonic acid, demethyl-sulfone). A linear analyser was used to scan the developed plates. Kidney tissue was subjected to repeated extractions and preparative TLC. All components accounting for more than 10% of the dose in the original tissue extract were isolated and subjected to mass spectrometry. References to the mass spectra were provided, but no actual spectra or data.

The results are shown in Table 2. The maximum residue in milk was found on day 2 pm, 3.83 mg/kg ODM equivalents.

Table 2. Distribution of radioactive residues in tissues and milk after oral administration of [^{14}C]ODM to a lactating goat.

Compound	% of total radioactive residue				
	Kidney (13 mg/kg)	Liver (4.2 mg/kg)	Muscle (4.0 mg/kg)	Fat (0.62 mg/kg)	Milk (3.8 mg/kg)
ODM	23.2	25.7	42.2	41.3	61.0
ODM sulfone	13.9	0.8	23.4	16.1	7.0
Unknown 1 (TLC origin; polar, 6 compounds)	16.2	34.5	7.5	11.1	2.5
Unknown 2	4.3	6.7	2.2	3.8	5.9
Unknown 3	13.3	21.4	10.0	15.2	23.6
Unknown 4	29.1	9.8	14.3	12.0	0.0
Identified, %	37.1	26.5	65.6	57.4	68.0

Marsh *et al.* (1987) dosed a group of laying hens with [*ethylene*-^{14}C]ODM, diluted to a specific activity of 8 mCi/mmol by the addition of natural abundance ODM, for three consecutive days at 6.9 mg/kg body weight. Eggs were collected each day at 7:30 and 20:00, pooled by day and refrigerated. Hens were killed within four hours of the final dosing and appropriate tissues were collected and pooled for the group. Homogenized samples of eggs and tissues were combusted and radio-assayed.

Homogenized tissue subsamples (5 g) were extracted with water or chloroform or sequentially with acetonitrile and water and the extracts were analysed by HPLC. Subsamples of eggs, fat and skin were extracted with methylene chloride/acetone (1:1) and the extracts purified by gel permeation chromatography. Extraction efficiencies before GPC were 93% for breast, 93% for thigh, 83% for liver, 93% for kidney, 70% for eggs and apparently 160% for fat. Appropriate fractions, as determined by radio-assay, were subjected to HPLC and TLC.

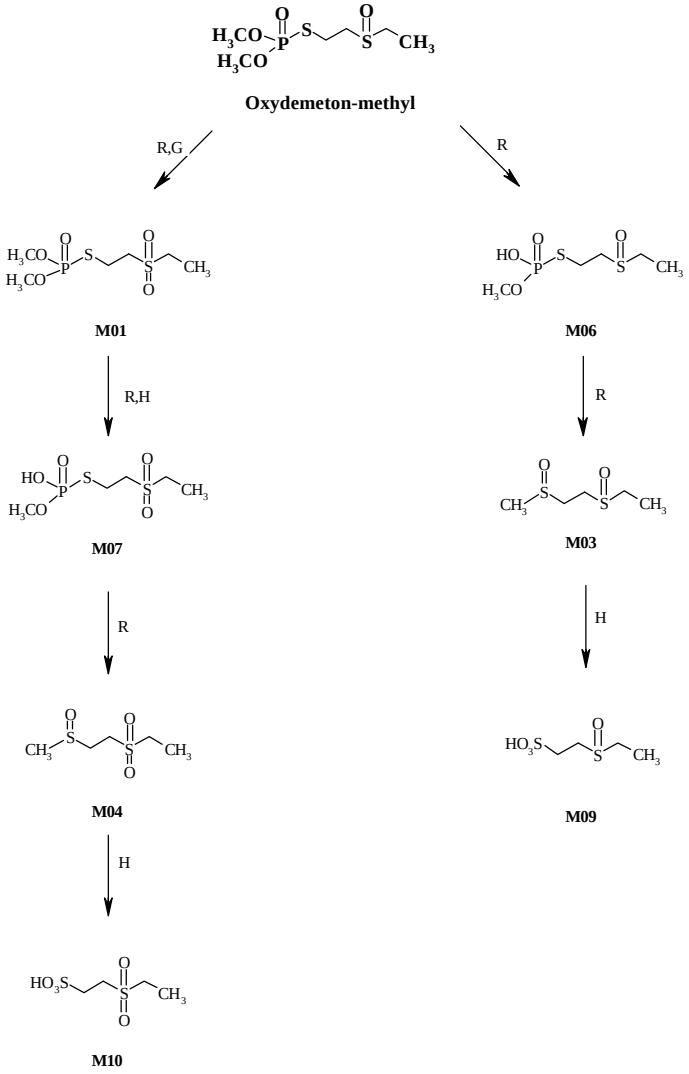
The results are shown in Table 3. Demethyl-ODM sulfone (M 07) was the major or only residue present in eggs, skin, heart, liver, breast and thigh muscle. The two sulfonic acid metabolites 2-(ethylsulfinyl)ethanesulfonic acid (M 09) and 2-(ethylsulfonyl)ethanesulfonic acid (M 10) were the main metabolites in the kidney and gizzard.

Table 3. Distribution of residues in tissues[1] and eggs after oral administration of ODM to laying hens.

Compound	% of total radioactive residue					
	Breast (0.51 mg/kg)	Thigh (0.43 mg/kg)	Liver (0.60 mg/kg)	Kidney (1.4 mg/kg)	Skin (0.41 mg/kg)	Eggs[2] (0.36 mg/kg)
ODM	3.3	n.d.	5.3	n.d.	n.d.	n.d.
M 07 (demethyl-ODM sulfone)	70.1	79.6	48.4	n.d.	100.0	100.0
M 09 + M 10 (2-(ethylsulfinyl) ethanesulfonic acid + 2-(ethylsulfonyl)ethane sulfonic acid)	26.6	n.d.	42.1	83.7	n.d.	n.d.
Unknown	0.0	20.4	4.3	16.3	0.0	0.0
Identified, %	100.0	80	96	84	100.0	100.0

[1]Fat was not successfully analysed.
[2]Day 3. Day 2 eggs = 0.20 mg/kg; day 1 eggs = 0.086 mg/kg.

Figure 1: Proposed metabolic pathways of ODM in animals (R = rats, G = goat, H = hen)



Plant metabolism

Metabolism studies were reported for ODM in sugar beets (Wagner *et al.*, 1989) and cabbages (Smyser and Halpin, 1987), demeton-S-methylsulphon in apples (Wagner *et al.*, 1984), and demeton-S-methyl in wheat (Wagner and Oehlmann, 1987).

Cabbages were treated with three foliar applications of [*ethylene*-1-^{14}C]oxydemeton-methyl at rates corresponding to 0.8 kg ai/ha each and harvested six weeks after the first application. About 97% of the radioactivity was extractable with acetone and aqueous acetone. ODM accounted for 12% of the recovered radioactivity and ODM sulfone (M 01) for 8% (TLC; MS). About 70% of the recovered radioactivity was characterized as being from acidic, very polar, water-soluble metabolites (TLC; HPLC; derivatizations). TLC analysis revealed at least 5 components.

[*Ethylene*-^{14}C]ODM (87.8 µci/mg) was applied with a microlitre syringe to three sugar beet plants. Roots and tops were sampled after 28 and 42 days, and tops also after 6 days. The samples were homogenized and extracted sequentially with chloroform, methanol and water. Most of the radioactivity was removed by the solvents (80–95% from foliage, 82–94% from roots). Extracts were analysed by TLC. Some identifications were confirmed by isolation and MS (EI and CI). The results are shown in Table 4.

Table 4. Distribution and identification of the radioactive residue from the application of [^{14}C]ODM to sugar beets.

Sample	Foliage[1] (% of ^{14}C)	Roots [1](% ^{14}C)
Surface washing	2.70	---
Organosoluble	61.40	8.10
Water-soluble	18.72	3.26
Unextractable	3.09	2.71
Total	85.91	14.07
Compound		
ODM	2.21	---
M 01 (ODM sulfone)	1.71	---
M 06 (demethyl-ODM)	14.19	3.39
M 11 (bis[2-(ethylsulfinyl)ethyl] disulfide)	11.53	1.03
M 12 (2-hydroxy-3-[(2-ethylsulfinyl)ethyl)thio]propionic acid)	13.71	1.61
M 13 (2-hydroxy-3-[(2-ethylsulfonyl)ethylthio]propionic acid)	10.09	0.61
M 14 (2-hydroxy-3-[(2-ethylsulfonyl)ethylsulfinyl]propionic acid)	10.91	2.13
Identified, %	64.34	8.68

[1] % of the sum of the radioactivity found in the root and foliage, NOT % of applied radioactivity. The total radioactivity in the foliage was 61% of the applied, and in the root 9.9% of the applied

[*Ethylene*-1-^{14}C]demeton-S-methyl was applied to spring wheat grown in a greenhouse. The test substance (241.5 µci in 2 ml benzene) was applied with a chromatography sprayer to wheat plants growing on 0.05 m^2 in a 10 l bucket. The actual radioactivity applied to the plants, excluding that on the soil, was 126.8 µci. 12 plants were harvested after 3 and 14 days, and 18 plants after 42 and 60 days. The plants were separated into kernels, chaff, straw and roots. No kernels existed at the 3-day sampling.

Straw, chaff, kernels and roots were washed with water and extracted sequentially with chloroform, acetone, methanol and water. The rinse water for straw was extracted with chloroform. The extracts were analysed by TLC. Radioactive zones located with X-ray film were scraped and determined by scintillation counting. MS was used to confirm tentative TLC identifications. The radioactivity levels, as demeton-S-methyl equivalents, were 10–17 mg/kg in the straw and 0.7–1.6 mg/kg in the kernels. In the 60-day samples, 76% of the residue was extractable from straw (13.6 mg/kg) and 24% from grain (0.72 mg/kg). The results are shown in Table 5.

Table 5: Identification of the radiolabelled residue in the straw and grain of wheat harvested 60 days after the application of [^{14}C]demeton-S-methyl (^{14}C in the plant at 60 days = 100%)

Compound	^{14}C, % of total in straw	^{14}C, % of total in grain
Demeton-S-methyl (test compound)	1.2	0.12
M 01 (ODM sulfone)	9.8	0.11
ODM	11.7	0.01
M 03 (1-(ethylsulfinyl)-2-(methylsulfinyl)ethane)	8.9	0.02
M 10 (2-(ethylsulfonyl)ethanesulfonic acid)	8.2	0.07
M 11 (bis[2-(ethylsulfinyl)ethyl] disulfide)	1.1	0.18
M 15 (*S*-2-(ethylsulfonyl)ethyl dihydrogen phosphorothioate	5.2	0.04
M 16 (1-(ethylsulfinyl)-2-(methylthio)ethane)	4.5	0.06
M 17 (2-ethylsulfinylethanol)	5.1	---
M 18 (2-ethylsulfonylethanol)	0.6	0.02
M 19 (2-ethylsulfinylethylene)	1.2	0.03
Unknown	5.8	0.07
Unextracted	23.8	3.8
Total Identified	58%	0.66%

[*Ethylene*-^{14}C]demeton-S-methylsulphon was applied to five apples on one tree of the James Grieve variety with an Eppendorf pipette (Wagner *et al.,* 1984). The exact application rate could not be ascertained from the information provided. Single apples were harvested at 0, 6, 13, 20 and 27 days after the application. The apples were rinsed with water to remove surface residues, peeled and the peel and pulp were each lyophilized and homogenized with dichloromethane. The residue was extracted sequentially with acetone, ethanol, methanol and water, and the residual solid was combusted and radio-assayed. Forty one per cent of the applied radioactivity was solvent-extractable on day 27 from both peel and pulp and 1.9% was recoverable from a surface water rinse. The extractable percentage was constant at each sampling, but the proportion in the surface rinse decreased from 13% on day 6. Extracts were analysed by TLC and tentative identifications were confirmed by MS. The results are shown in Table 6. Neither radioactive residue concentrations (mg/kg as parent compound) nor the data needed to calculate them were supplied.

Table 6. Identification of the residues from the application of [^{14}C]demeton-S-methylsulfon to apples.

Compound	Day 6 (% of applied)		Day 13 (% of applied)		Day 20 (% of applied)		Day 27 (% of applied)	
	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel
(Parent M 01)	25	35	30	21	26	25	24	24
M 07 (demethyl M 01)	2.0	4.5	2.4	1.9	1.3	3.2	2.6	2.9
M 15 (*S*-2-(ethylsulfonyl)ethyl dihydrogen phosphorothioate	2.0	1.5	1.7	1.9	0.6	3.3	1.2	3.4
Demeton-S-methyl sulfone (M 01)	0.9	3.5	0.9	3.3	1.4	3.1	0.9	0.9
2-(ethylsulfonyl)ethanesulphonic acid	1.4	0.9	1.3	1.1	3.9	1.4	3.2	2.2
1-(ethylsulfonyl)-2-(methylsulfinyl)ethane	1.1	1.5	2.5	1.3	0.7	1.9	1.6	1.3

Compound	Day 6 (% of applied)		Day 13 (% of applied)		Day 20 (% of applied)		Day 27 (% of applied)	
	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel
1-(ethylsulfonyl)-2-(methylsulfonyl)ethane	0.6	0.6	0.9	0.7	0.4	0.9	0.7	0.6
2-(ethylsulfonyl)ethanol (M 18)	1.1	2.4	6.2	3.1	0.5	2.3	2.9	0.2
Unknown	0.4	0.7	1.2	1.9	0.6	4.3	0.2	3.3
Total identified	34	50	46	34	35	41	37	39

The four metabolism studies were inadequately detailed. The data needed to verify ^{14}C concentrations and to convert counts to mg equivalents per kg substrate were not provided. The qualitative identification steps were not detailed, but the mass spectra used to confirm identifications were provided. Two of the studies were conducted with compounds closely related to ODM, but not with ODM. The studies taken together indicate the metabolic pathways shown in Figure 2.

Environmental fate in soil

Stevenson (1989) applied [*ethylene*-1-^{14}C]oxydemeton-methyl to a sandy loam soil at a rate corresponding to 3.4 kg ai/ha and incorporated it to a depth of 15 cm in Stilwell, Kansas, USA (1988–1989). The soil consisted of 56% sand, 36% silt, 8% clay, 2.6% organic matter and the pH was 5.1. The treated soil was aged under aerobic conditions for 34 days. Kale, wheat and red beets were planted in the first rotation. The second and third rotational crops were planted 184 and 351 days after treatment. The crops were harvested at normal maturity and radio-assayed.

The radioactive residues in the soil decreased from 0.5 mg/kg on the day of application to 0.07 mg/kg at harvest 351 days after treatment. No residues (<0.01 mg/kg) were detectable in the rotational leafy and root crops. Residues in rotational wheat were low (0.03 mg/kg in straw) in the first rotation, highest (0.06 mg/kg in chaff) in the second rotation and undetectable (<0.01 mg/kg) in the third rotation. Because of the rapid decline of oxydemeton-methyl in soil, very low or negligible amounts of residues were taken up by the rotational crops. The results are shown in Table 7.

Figure 2. Proposed metabolic pathways of oxydemeton-methyl, demeton-S-methyl and demeton-S-methylsulphon in plants.

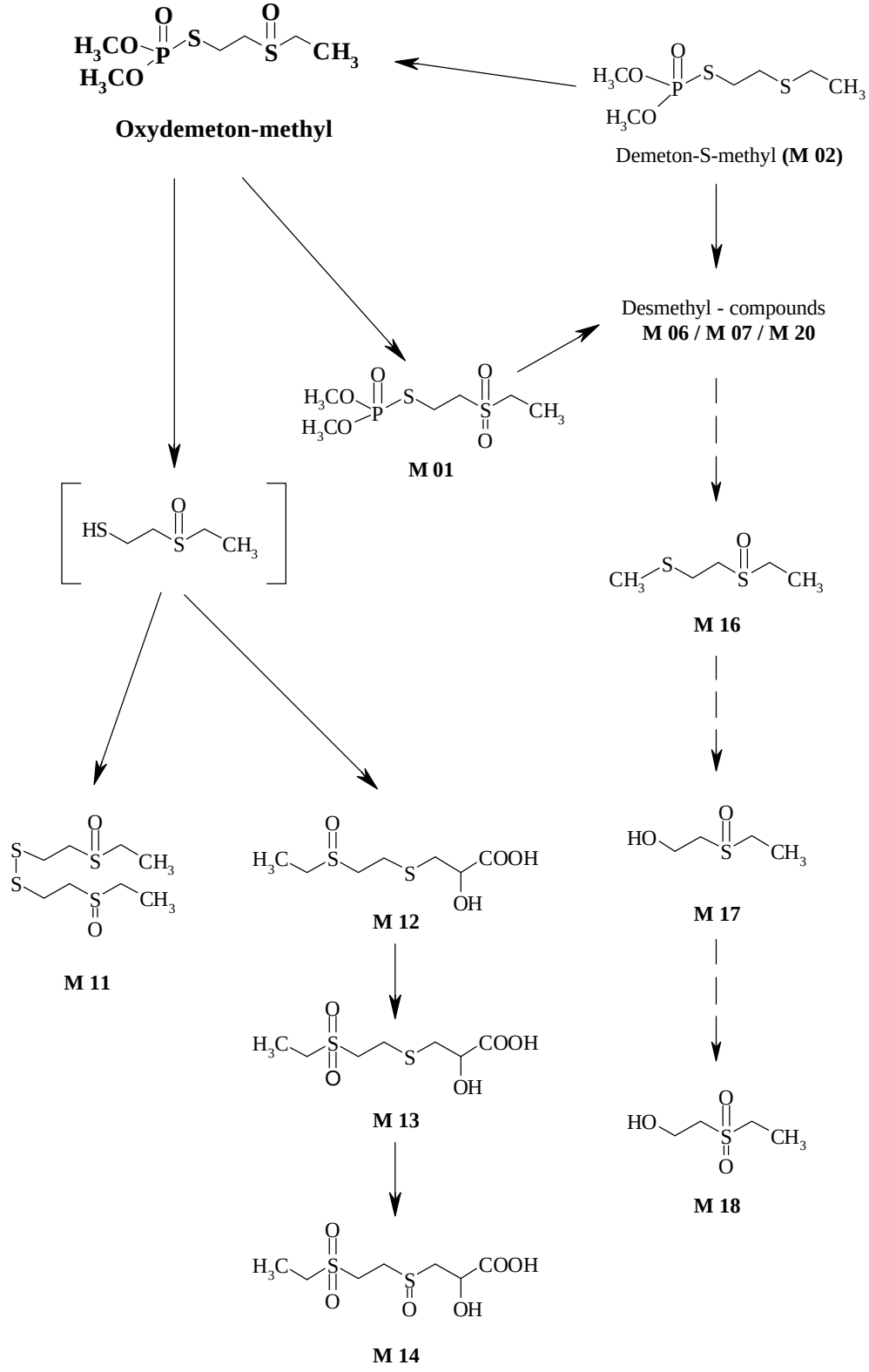


Table 7: Total radioactive residue concentrations in soil and rotational crops after application of [^{14}C]ODM to soil at 3.4 kg ai/ha.

Sample type	Planting interval, days/Sampling interval, days, after application of [^{14}C]ODM								
	First rotation (mg ai equivalent/kg)			Second rotation (mg ai equivalent/kg)				Third rotation (mg ai equivalent/kg)	
	34/	34/73	34/140	184/	184/204	184/227	184/267	305	305/351
Soil	0.14	0.01	0.01	0.10	0.08	0.08	0.08	0.07	0.07
Immature wheat (boot stage)		0.02		<0.01					
Kale		<0.01			<0.01			<0.01	
Red beet top			<0.01				<0.01		<0.01
Red beet root			<0.01				<0.01		<0.01
Wheat straw			0.03			0.02			<0.01
Wheat chaff			0.02			0.06			<0.01
Wheat grain			0.01			0.02			<0.01

As the radioactive residues in all the crop samples were very low, <0.01-0.06 mg/kg, they could not be characterized or identified. Wheat grain, chaff and straw from the first and second rotations were extracted with methanol/water and refluxed with 1 N HCl. Neither solvent nor acid extracted the radiolabel.

The photolysis of ODM on soil was studied by Jackson *et al.* (1988). [*Ethylene*-1-^{14}C]oxydemeton-methyl was applied to a sandy loam soil and irradiated under natural sunlight conditions for 30 days. The resulting half-lives of 61 days of the irradiated samples and 53 days of the dark controls indicated that degradation was not due to photolysis. At the end of the study about 76% of the applied radioactivity was due to unchanged parent compound. The products identified were demethyl-ODM sulfone (M 07, 1.9%) and the sulfonic acids M 09 (2-ethylsulfinylethanesulfonic acid) and M 10 (2-ethylsulfonylethanesulfonic acid), their sum 22.1%. The products and their proportions were the same for the irradiated and unexposed soils at each time interval. None of the unknown products accounted for >4% of the applied radioactivity

The adsorption and desorption of [*ethylene*-1-^{14}C]oxydemeton-methyl was investigated by Daly (1987, revised 1988). A batch equilibrium procedure was used to determine the K_d and K_{oc} values in four soils equilibrates at 25 $\pm$ 1ºC with an aqueous solution of the radiolabelled ODM. The mass balance was >98%. The results are shown in Table 8. The soils were sand (88% sand, 7% silt, 5% clay, 0.52% organic carbon, pH 4.3), sandy loam (56% sand, 30% silt, 14% clay, 0.58% organic carbon, pH 6.6), silt loam (17% sand, 66% silt, 17% clay, 1.53% organic carbon, pH 5.9), and clay loam (21% sand, 50% silt, 29% clay, 1.16% organic carbon, pH 6.4). The low K_{oc} values suggest that oxydemeton-methyl is not significantly bound to soil.

Table 8. Adsorption of [^{14}C]ODM on different soil types.

Soil	Adsorption	
	K_d (ml/g)	K_{oc} (ml/g)
Sand	0.09	17
Sandy loam	0.01	2
Silt loam	0.89	58
Clay loam	0.45	39

In a study of the leaching of aged ODM in a sandy clay loam soil after incubation under greenhouse conditions (Obrist and Thornton, 1978) the soil was treated with [*ethylene*-1-^{14}C]oxydemeton-methyl and aged aerobically for 30 days, by which time almost 50% of the originally applied radioactivity was lost as CO_2. After the ageing period samples were placed on the top of three soil columns (30 cm long and 4.8 cm i.d.) containing fresh soil. The rate of addition of leaching water was adjusted to equal approximately the rate of discharge. 1.25 cm of simulated rainfall was applied each day for 45 days. The results are shown in Tables 9 and 10. ODM and its degradation products showed little tendency to leach through the soil.

Table 9. Leaching of [*ethylene*-1-^{14}C]oxydemeton-methyl in a sandy clay loam soil (56% sand, 23% silt, 21.0% clay, 0.36% org. C, pH6.0) during 45 days.

	% of radioactivity recovered from column		
	I	II	III
Layer 1 (0-1.25 cm)	36.2	1.1	0.3
Layer 2 (1.25-2.5 cm)	50.6	80.7	79.4
Layer 3 (2.5-5.0 cm)	1.4	3.4	6.5
Layer 4 (5-7.5 cm)	0.7	2.2	0.9
Layer 5 (7.5-12.5 cm)	1.0	1.1	1.1
Layer 6 (12.5-17.5 cm)	0.6	0.6	0.8
Layer 7 (17.5-22.5 cm)	<0.1	<0.1	<0.1
Layer 8 (22.5-27.5 cm)	<0.1	<0.1	<0.1
Layer 9 (27.5-30.0 cm)	<0.1	<0.1	<0.1
Total in soil	90.5	89.1	89.0
Leachate	9.5	10.9	11.0
Total	100.0	100.0	100.0

Table 10. Distribution of degradation products of [*ethylene*-1-^{14}C]oxydemeton-methyl in the leachate from aged soil column II.

Compound	^{14}C, mg/kg as ODM, at days					Total
	1-9	10-18	19-27	28-36	37-45	
oxydemeton-methyl	0.13	0.24	0.09	0.03	0.02	0.51
M 01 (ODM sulfone)	0.03	0.06	0.02	trace	trace	0.27
M 09 (2-ethylsulfinylethanesulfonic acid)	0.21	0.32	0.09	trace	trace	0.62
M 10 (2-ethylsulfonylethanesulfonic acid)	0.26	0.40	0.24	0.06	trace	0.90
M 21 (2-ethylsulfinylethanethiol)	0.07	0.13	0.05	0.02	trace	0.27
Unknown	0.64	0.78	0.29	0.13	0.13	1.73
Unextractable	0.41	2.58	1.89	0.89	0.69	6.76

The degradation of oxydemeton-methyl was investigated under aerobic conditions on a sandy loam soil (Schmidt *et al.*, 1993). Soil samples were treated with [*ethylene*-1-^{14}C]oxydemeton-methyl under dark conditions at a temperature of 25 $\pm$ 1°C at a nominal concentration of 25 mg/kg corresponding to 5 kg ai/ha, 10 times the maximum recommended US rate. Samples were collected at 0, 1, 3, 7, 14 and 21 days and 1, 2, 3, 4, 6, 9 and 12 months after treatment. Extractable residues (methanol and water/methanol) decreased from 87.1% of the applied radioactivity at day 0 to 52.6% at 12 months, and unextractable increased from 0.4% on day 0 to 21% after 12 months. Volatile residues increased to 9.3% after 12 months and were shown to be CO_2. The mean recovery of ^{14}C was 93%. The main products observed by HPLC at all sampling points were the sulfonic acids M 09 and M 10 at maximum levels of 30% of the applied radioactivity after 2 months and 26% after 12 months respectively. ODM decreased to 50% of the applied amount within 3 days and to 1% within 21 days. No compound exceeded 10% of the applied ^{14}C at any time. Minor products were ODM sulfone (M 01, 6% maximum), 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane (M 03, 1% maximum), 1-(ethylsulfonyl)-2-(methylsulfinyl)ethane (M 04, 1% maximum) and 2(ethylthio)ethane sulfonic acid (M 22, 3% maximum). Volatiles accounted for 9% of the applied radioactivity.

The half-life of oxydemeton-methyl under these conditions was calculated to be 3.2 days using first order degradation kinetics. Its degradation pathways are shown in Figure 3.

The results reported in the Schmidt experiment were in good agreement with those of an earlier study (Olson *et al.*, 1989) under dark conditions at a temperature of 25 $\pm$ 0.6°C. Soil samples were treated with [*ethylene*-1-^{14}C]oxydemeton-methyl at a nominal concentration of 8.3 mg/kg, corresponding to about 1.7 kg ai/ha. Samples were collected after 0 and 6 hours, 1, 3, 7, 14 and 21 days and 1, 2 and 3 months. As in the Schmidt study, the main products observed at all sampling points were the sulfonic acids M 09 and M 10. No other product exceeded 10% of the applied ^{14}C at any time, but about 30% was recovered as carbon dioxide after 3 months. The half-life of oxydemeton-methyl was calculated to be 9.6 hours.

In a supplementary study (Kasper and Shadrick, 1993) on degradation under field conditions two sandy loam soils from two sites in California (Fresno and Chualar) were treated with [*ethylene*-1-^{14}C]oxydemeton-methyl at a rate corresponding to 1.12 kg ai/ha. Samples were analysed in triplicate immediately after application and in duplicate after 6 and 12 hours and 1, 2, 4 7, 10, 18, 29 and 60 days. At each sampling, the top 3 cm of soil was analysed for degradation products.

During the 60-day experiment, the residues of the parent compound decreased to 2.1% of the applied radioactivity in the Fresno soil and 0.3% in the Chualar soil. Residues of M 09 and M 10 reached maximum levels of 28.5% and 14.0% on day 4 in the Fresno soil. The corresponding levels in the Chualar soil were approximately 20% and 10% between 2 and 18 days.

The total radioactive residues decreased in both soils throughout the experiment with most of the radioactivity remaining in the top 3 cm layer. Residues extracted from the top soil layers decreased as the bound residues increased. The distribution of the residues as a function of time is shown in Table 11.

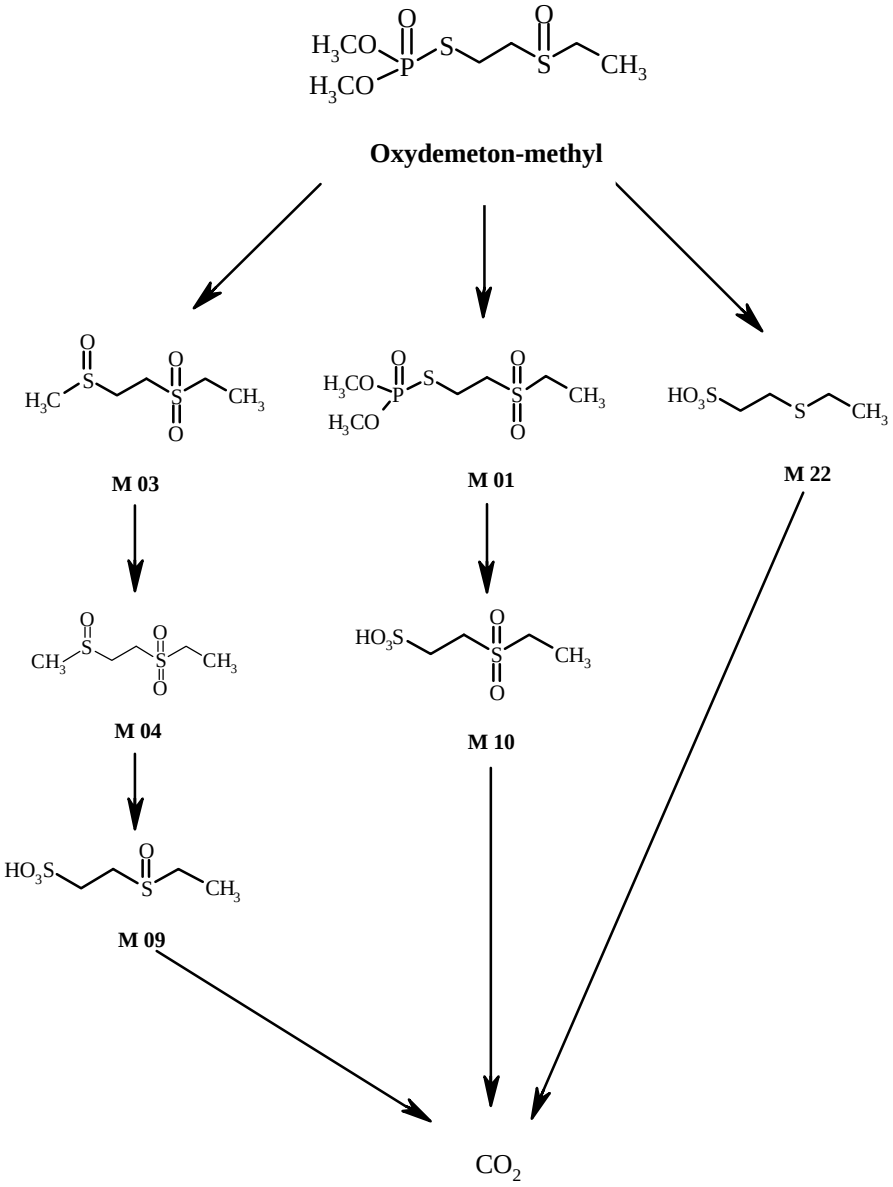
Table 11. Distribution of residues in 0-3 cm soil layers at intervals after application of [*ethylene*-1-^{14}C]oxydemeton-methyl in Fresno and Chualar, California.[1]

Elapsed time, days	^{14}C, % of applied			
	ODM	M 09	M 10	Unknowns
Fresno				
0	94.6	3.5	nd	nd
0.25	78.9	6.5	nd	4.0
0.50	84.3	6.8	nd	1.3
1	50.2	15.6	9.1	nd
2	52.8	6.6	2.1	7.5
4	11.1	28.5	14.0	6.7
7	nd	8.9	4.7	nd
10	nd	9,0	5.6	nd
18	1.4	3.6	5.2	nd
39	1.9	8.7	6.8	3.0
60	2.1	8.7	5.6	3.6
Half-life, days	10.3	5.3	4.5	
Chualar				
0	92.1	1.8	nd	nd
0.25	84.2	2.9	nd	nd
0.50	84.1	3.4	nd	1.2
1	76.0	7.4	nd	nd
2	50.6	20.2	7.0	nd
4	49.7	11.0	3.0	6.2
7	24.1	19.0	10.8	5.1
10	19.9	16.5	9.7	5.0
18	8.5	15.6	10.7	2.9
39	0.1	0.9	1.1	0.5
60	0.3	4.2	1.1	1.1
Half-life, days	6.1	6.4	8.5	

[1] Average of duplicates, except time 0 which is average of triplicates

In a study of the degradation of oxydemeton-methyl in soil under anaerobic conditions (Olson, 1989b) [*ethylene*-1-^{14}C]oxydemeton-methyl was applied to a sandy loam soil at a nominal concentration of 10.0 mg/kg, corresponding to 2 kg ai/ha. The incubation temperature was 25 $\pm$ 0.4°C. The flasks were flushed with nitrogen to maintain anaerobic conditions. Samples were taken at 0 and 10 hours and 30 and 60 days. Oxydemeton-methyl was rapidly degraded. The major degradation products were the sulfonic acids M 09 and M 10, the sum of which reached a maximum of 27% of the applied radioactivity after 10 hours. The only other identified product was M 22, 2-(ethylthio)ethanesulfonic acid, at 4% of the applied radioactivity. The sampling times were too few to calculate a half-life for ODM.

Figure 3: Proposed degradation pathways of oxydemeton-methyl in soil.



In a dissipation study under field conditions (Jacobsen, 1989; Grace and Cain, 1990) oxydemeton-methyl was applied to soil cropped with sugar beets at two sites in California (Fresno and Chualar). Two plots were treated at each site, one with a single application of 1.12 kg ai/ha and the other with six applications of 0.84 kg ai/ha. These rates represent the highest label single rate and the highest seasonal rate of 5.05 kg ai/ha/season respectively. Fifteen soil core samples were taken at each sampling.

At the multiple-application plots, core samples were taken to a depth of 15 cm directly after each of the first five applications and composited by field section to give three replicate samples per application. Cores for all the other samples were driven to a depth of 122 cm and divided into 15 cm segments. For all samples after the final or single applications the 15 cm soil segments were analysed separately and all lower segments were composited by field section to give three replicates per segment for analysis.

First-order dissipation rate constants (k) and half-life values of oxydemeton-methyl were calculated from the single-application plots. Calculations were also made from the analytical results from all the plots to indicate possible leaching of the compound and its sulfone M 01. The k values for ODM were calculated to be 0.31499 at Chualar and 0.44415 at Fresno with half-lives of 2.2 and 1.6 days respectively. At no time were any measurable residues ($\geq$0.01 mg/kg, dry weight basis) of oxydemeton-methyl or its sulfone found in soil below the 0–15 cm segments.

Environmental fate in water/sediment systems

The behaviour of [*ethylene*-1-^{14}C]oxydemeton-methyl in sterile buffer solutions pH 5, 7 and 9 at 25° and 40°C and concentrations of 5 and 50 mg/l was reported by Pither and Puhl (1978, revised 1989). The half-life of oxydemeton-methyl varied from less than one day at pH 9 and 40°C to 94 days at pH 5. Degradation was dependent on both pH and temperature. The results are given in Table12. The degradation products identified by TLC were demethyl-ODM (M 06) and 2-(ethylsulfinyl)ethamethiol (M 21). At pH 7 on day 35, M 06 and M 21 accounted for 34% and 13% of the recovered radioactivity at 25^{o}C and for 67% and 28% at 40^{o}C.

Table 12. Half-life values for the hydrolysis of [*ethylene*-1-^{14}C]ODM in sterile aqueous buffer solutions.

pH	Half-life, days	
	25°C	40°C
5	93.7	21.7
7	39.6	7.5
9	2.5	<1

Kesterson *et al.* (1985) exposed [*ethylene*-1-^{14}C]oxydemeton-methyl in sterile buffer solution at pH 5 to natural sunlight for 30 days. Duplicate irradiated and dark control samples were taken at 0, 5, 10, 15, 20 and 30 days. The half-life of ODM was 137 days for irradiated samples and 194 days for dark controls. Thus, photolytic processes account for little if any of the degradation. The major degradation products identified were demethyl-ODM sulfone (M 07; 7.4% on day 30, 2.8% in control) and the sulfonic acids M 09 and M 10 (3.7% on day 30, 8.3% in control). The recovery of the applied radioactivity was >94% from all samples.

A study of the behaviour of [*ethylene*-1-^{14}C]oxydemeton-methyl in two water/sediment systems was reported by Anderson (1987). Water and sediment were collected from on orchard drainage ditch (IJzendoorn) and from a fishpond (Lienden) in The Netherlands. Both systems were treated with 0.5 mg ai/l. Samples were taken on days 1, 7, 20 ,41 and 91.

The distribution of radioactivity between the aqueous phase and the sediment was similar in the two test systems (Table 13).

Table 13. Distribution of radioactivity from [*ethylene*-1-^{14}C]oxydemeton-methyl in water/sediment systems.

System	Sampling interval, days	^{14}C, % of applied (mean of duplicates)				
		Aqueous phase	Sediment extracts incl. HCl	Sediment unextractable	Volatile compounds	
					$^{14}CO_2$	others
IJzendoorn	1	75.8	15.2	9.6	<0.1	<0.1
	7	33.5	20.6	39.4	0.8	<0.1
	20	18.4	18.3	52.6	4.3	<0.1
	41	4.4	5.6	67.5	15.2	<0.1
	91	1.9	4.2	65.9	22.3	<0.1
Lienden	1	82.9	7.5	9.2	<0.1	<0.1
	7	45.3	11.0	35.3	0.6	<0.1
	20	32.5	8.9	45.8	3.8	<0.1
	41	22.7	6.1	48.4	11.1	<0.1
	91	20.9	4.6	44.9	26.6	<0.1

ODM was rapidly degraded in both systems. One day after application the parent compound accounted for 48.7% of the applied radioactivity in IJzendoorn and 37.6% in Lienden. After incubation for 7 days these values decreased to 11.4 and 9.7% respectively. The degradation of oxydemeton-methyl in the water/sediment systems was accompanied by the continuously increasing formation of carbon dioxide. Comparable mineralisation rates were observed in the two systems. After incubation for 91 days $^{14}CO_2$ accounted for 22% of the ^{14}C at IJzendoorn and 27% at Lienden. The identities and distribution of the components of the residue are shown in Table14 and proposed degradation pathways are shown in Figure 4.

Table 14. Distribution of ODM and its degradation products after incubation of [*ethylene*-1-^{14}C]oxydemeton-methyl in water/sediment systems at Ijzendoorn and Lienden.

Compound	Sample	% of applied ^{14}C at Ijzendoorn/Lienden after interval, days (mean of duplicates)				
		1	7	20	41	91
ODM	Surface water	46/36	11/9.3	0.2/0.7	0.4/0.4	<0.1/1.4
	Sediment	3.2/2.1	0.7/0.4	Trace/0.2	0.2/0.2	<0.1/0.1
M 01	Surface water	0.8/nd	0.3/0.6	0.2/0.5	0.4/0.3	Trace/0.7
	Sediment	nd/0.1	nd/nd	nd/0.1	nd/0.2	nd/nd
M 02	Surface water	0.7/0.4	2.6/1.3	Trace/nd	0.2/<0.1	nd/<0.1
	Sediment	3.3/0.3	0.7/0.1	0.1/nd	<0.1/nd	nd/nd
M 07	Surface water	7.2\4.9	3.1/10	3.3/11	0.3/4.5	Trace/9.0
	Sediment	0.2/nd	Nd/Trace	Nd/Nd	Nd/0.8	Nd/<0.1
M 09 + M 06	Surface water	2.3/2.4	6.5/6.5	7.3/8.6	0.8/0.2	Trace/0.5
	Sediment	0.4/0.9	1.6/0.7	0.8/0.2	0.4/0.4	0.2/0.2
M 10	Surface water	1.6/2.2	3.4/1.4	1.8/2.0	<0.1/0.3	Trace/0.9
	Sediment	1.5/trace	1.5/1.5	1.1/1.7	0.4/0.1	0.4/1.0

Compound	Sample	% of applied ^{14}C at Ijzendoorn/Lienden after interval, days (mean of duplicates)				
		1	7	20	41	91
M 11	Surface water	7.4/20	2.5/6.7	1.7/1.0	Trace/0.3	Nd/Trace
	Sediment	1.2/1.2	Nd/Nd	Nd/Nd	Nd/Nd	Nd/Nd
M 15	Surface water	Nd/1.4	Nd/Nd	Nd/Nd	Nd/<0.1	/trace
	Sediment	Nd/Nd	Nd/Nd	Nd/Nd	Nd/Nd	Nd/Nd
M 23	Surface water	1.2/4.9	1.1/3.1	Nd/0.1	Nd/<0.1	Nd/<0.1
	Sediment	Nd/Nd	Nd/Nd	Nd/Nd	Nd/Nd	Nd/Nd
Unknown	Surface water	3.7/4,7	2.1/3.5	3.1/2.2	1.2/0.5	Nd/0.5
	Sediment	Trace/0.1	1.0/0.5	2.1/0.2	0.3/0.1	0.4/0.5

M01 ODM
M02 demeton-S-methyl
M06 demethyl-ODM
M07 demethyl—ODMsulfone
M09 2-(ethylsufinylethanesulfonic acid
M10 2-(ethylsufonyl)ethanesulfonic acid
M11 bis[2-(ethylsufinyl)ethyl]disulfide
M15 bis[2-(ethylsufonyl)ethyl]disulfide
M23 2-(ethylsufinylethyl) 2-(ethylsufonylethyl disulfide
Nd = not detected

In a supplementary experiment Schmidt and Anderson (1993) applied [*ethylene*-1-^{14}C]oxydemeton-methyl to a pond sediment flooded with pond water to maintain anaerobic conditions at a nominal concentration of 2.55 µg/ml in the dark at 25 $\pm$ 1°C. Samples were collected after 0, 1, 3, 7, 14 and 21 days and 1, 2, 3, 4, 6, 9 and 12 months.

Residue levels in the water decreased from 96.1% of the applied radioactivity at day 0 to 26.1% at 12 months. The total extractable residues in the sediment changed little during the study, ranging from 3.5 to 8.5% of the applied radioactivity. Unextractable residues increased from 0.4% of the applied radioactivity at day 0 to 27% at 2 months, then decreased to 18% at 12 months. Volatile residues increased to 8.7% of the applied radioactivity at 12 months with measurable amounts of radioactivity in both the ethylene glycol and potassium hydroxide traps. The total recovery decreased from 96% at 1 day , to 81% at 1 month and 59% at 12 months.

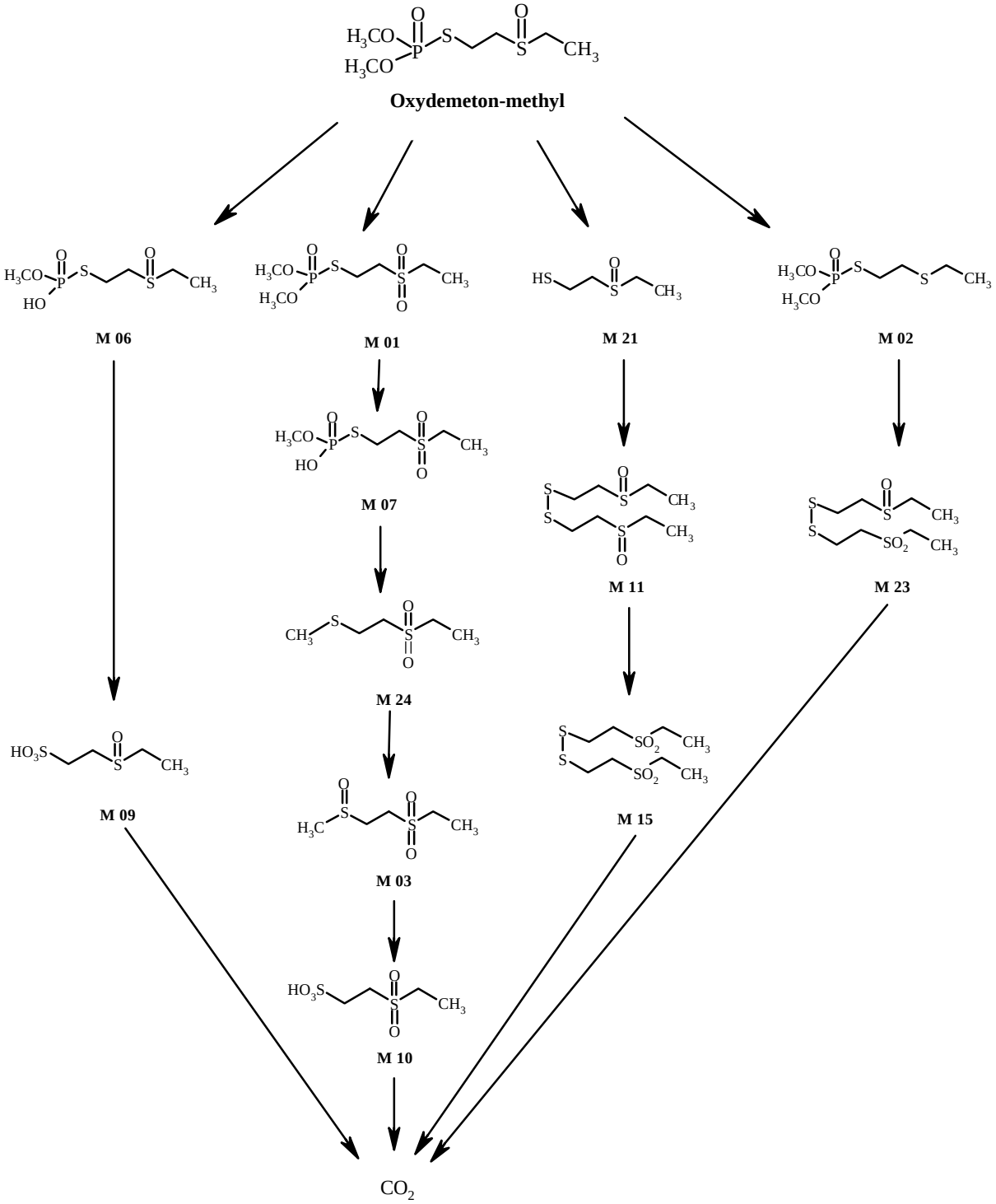
ODM was rapidly degraded. The main products were demeton-S-methyl (M 02) formed by reduction and 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane (M 03) formed by oxidation to the sulfone and cleavage of the P-S linkage. Significant levels of M 03 did not appear until about one month into the incubation. The distribution of the residues is shown in Table 15.

Table 15. Distribution of ODM and its degradation products after incubation of [*ethylene*-1-^{14}C]oxydemeton-methyl in a water/sediment system under anaerobic conditions.

Com-pound	Fraction	% of applied ^{14}C after												
		Days						Months						
		0	1	3	7	14	21	1	2	3	4	6	9	12
ODM	surface water	93.3	60.6	44.7	20.4	2.7	1.8	2.7	5.3	5.9	2.0	0.7	nd	nd
	sediment	3.4	6.7	2.0	2.6	0.2	0.2	0.1	0.2	0.4	0.4	Nd	nd	nd
	acidic extract	--	---	---	nd	nd	nd	nd	nd	nd	nd	Nd	nd	nd
M 01	surface water	nd	0.2	0.6	0.8	0.4	0.6	4.1	3.7	0.4	nd	Nd	0.8	0.4
	sediment	nd	nd	nd	nd	0.2	nd	nd	0.2	nd	nd	Nd	nd	nd
	acidic extract	---	---	---	nd	nd	nd	nd	nd	nd	nd	Nd	nd	nd
M 02	surface water	0.1	21.2	36.5	53.9	43.9	46.1	23.0	2.4	0.1	0.2	Nd	nd	nd
	sediment	0.1	nd	4.7	nd	2.7	3.3	2.5	nd	nd	nd	Nd	nd	nd
	acidic extract	---	---	---	nd	nd	0.4	0.4	0.2	nd	nd	Nd	nd	nd
M 03	surface water	0.1	nd	nd	nd	0.8	1.2	1.3	2.7	7.5	8.6	14.9	17.6	12.9
	sediment	nd	nd	nd	nd	0.2	nd	nd	0.4	1.2	0.6	1.3	0.6	0.7
	acidic extract	---	---	---	nd	0.6	0.6	nd	nd	nd	nd	0.3	0.6	Nd
M 09	surface water	nd	nd	0.4	0.6	1.6	1.8	4.2	5.1	5.9	6.9	6.1	5.7	8.8
	sediment	nd	nd	nd	0.2	0.2	nd	0.1	0.2	0.3	0.4	1.0	nd	0.3
	acidic extract	---	---	---	nd	0.2	0.2	0.1	0.2	0.1	0.4	0.4	0.2	0.8
M 10	surface water	nd	0.2	0.6	0.6	2.0	0.8	2.6	4.1	2.9	2.4	4.4	4.3	4.2
	sediment	nd	nd	nd	0.2	nd	nd	nd	nd	0.4	nd	0.3	nd	0.1
	acidic extract	---	---	---	nd	nd	nd	nd	0.2	0.1	0.4	Nd	0.2	0.4
M 22	surface water	1.7	nd	nd	nd	0.4	0.4	2.0	2.6	7.1	5.5	2.0	nd	Nd
	sediment	nd	nd	nd	0.6	0.2	nd	0.1	0.2	nd	0.2	Nd	nd	Nd
	acidic extract	---	---	---	nd	nd	nd	nd	0.2	nd	nd	0.3	nd	Nd
M 24	surface water	0.3	nd	0.2	1.8	2.0	0.6	1.2	1.0	0.7	nd	Nd	0.2	Nd
	sediment	nd	0.4	nd	nd	nd	nd	nd	nd	nd	nd	Nd	nd	Nd
	acidic extract	---	---	---	nd	0.2	nd	nd	0.2	nd	nd	Nd	nd	Nd
Un-	surface water	0.4	4.4	4.8	3.4	14.8	7.7	9.4	3.1	1.1	0.6	Nd	0.4	0.4
knowns	sediment	nd	nd	nd	3.5	2.4	1.0	1.8	2.6	0.1	0.6	Nd	2.0	1.4
	acidic extract	---	---	---	1.2	0.6	0.8	1.1	2.2	1.8	2.0	2.1	1.4	2.8

M 01 = oxydemeton-methyl sulfone
M 02 = demeton-S-methyl
M 03 = 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane
M 09 = 2-(ethylsulfinyl)ethanesulfonic acid
M 10 = 2-(ethylsulfonyl)ethanesulfonic acid
M 22 = 2-(ethylthio)ethanesulfonic acid
M 24 = 1-(ethylsulfonyl)-2-(methylthio) ethane

Figure 4. Proposed degradation pathways of oxydemeton-methyl in the aquatic environment.



METHODS OF RESIDUE ANALYSIS

Analytical methods

The Netherlands reported a multi-residue method for the determination of pesticides amenable to GLC, including ODM and demeton-S-methyl (Netherlands, 1996). ODM is oxidized to the sulfone and determined as demeton-S-methylsulphon. The same procedure is used for demeton-S-methyl. Foodstuffs are extracted and analysed without clean-up on a gas chromatograph with an ion trap detector (ITD). The limit of determination is estimated to be 0.01–0.05 mg/kg. Recoveries were demonstrated for demeton-S-methyl (various substrates, n = 10, mean recovery 79% at 0.29 mg/kg) and for demeton-S-methylsulphon (lettuce, n = 10, mean recovery = 107% at 0.29 mg/kg). The Netherlands defines the residue as the sum of ODM and demeton-S-methylsulphon, expressed as demeton-S-methyl.

Bayer AG reported both enforcement methods of the Deutsche Forshungsgesellshaft and methods used with various field trials. The enforcement method combines DFG Method S19, a multi-residue method for the extraction of numerous lipoid- and water-soluble pesticides, and DFG Method S16 (Hild and Their, 1979) a multi-residue GLC analytical method Method M012, a modification of S19 (Specht, 1990; Specht and Tillkes, 1982).

In Method S19 plant material is extracted with acetone and water is added to give an acetone/water ratio of 1/1. This is saturated with sodium chloride and partitioned with methylene chloride. The methylene chloride extract is concentrated and cleaned up by gel permeation chromatography on Bio Beads S-X3 with cyclohexane and ethyl acetate as eluants. The residue-containing fractions are concentrated, cleaned up on a silica gel minicolumn if necessary (1 g deactivated silica gel; elution with cyclohexane/ethyl acetate) and analysed by DFG method S16.

Method S16 specifies oxidation with potassium permanganate, which converts demeton-S-methyl and ODM to the sulfone. The mixture is diluted with water and extracted with methylene chloride. The solvent is replaced with acetone and the extract analysed on a gas chromatograph equipped with a flame-photometric detector. The nominal limit of quantification is 0.01 mg/kg. The only validation provided was the analysis of a single sample of sunflower seed fortified at 0.01 mg/kg, with a 99% recovery (Specht, 1990).

Bayer AG also reported its Method 00246 for the enforcement determination of ODM in plant, animal and soil samples (Thornton *et al.*, 1997). Plant samples (100 g) are homogenized and extracted with acetone (200 ml) in a blender, diluted with water and filtered. The filtrate is washed with hexane (150 ml) and extracted with chloroform (1 x 200 ml, 1 x 50 ml). Oily substrates (50 g) are extracted directly with chloroform (300 ml). The chloroform is evaporated and the residue dissolved in hexane. The hexane is partitioned with water and the water with chloroform. The final chloroform extract is evaporated to dryness.

Soil samples (50 g) are extracted in a Soxhlet apparatus for 4 hr using 300 ml of a 1/1 chloroform/methanol mixture. The extract is evaporated to dryness.

Animal tissues except fat, milk and eggs (50 g) are blended with anhydrous sodium sulfate (50 g), Hyflo Super-Cel (10 g) and acetonitrile (200 ml). The mixture is filtered and the cake is extracted with hexane (300 ml). The filtrates are combined and shaken and the acetonitrile layer is retained and evaporated. The residue is dissolved in hexane and extracted with water. The water fraction is extracted with chloroform and the chloroform extract evaporated to dryness. Fat (50 g) is extracted directly with hexane and the filter cake is extracted with acetonitrile. The acetonitrile and hexane fractions are combined and processed as above. Milk (100 g) is extracted in the same way as animal tissues, but the

sodium sulfate is omitted. Single whole eggs are blended with acetone and filtered. The cake is washed with chloroform and the combined chloroform and acetone fractions are allowed to separate. The organic layer is evaporated to dryness, taken up in hexane and extracted with acetonitrile (3 x 50 ml). The combined acetonitrile extracts are evaporated to dryness.

The evaporated residue is dissolved in acetone (2 ml) and oxidized with potassium permanganate (25 ml of 0.1 M). The reaction is allowed to proceed for 30 minutes at room temperature. The mixture is extracted with chloroform (3 x 25 ml) and the combined extracts are evaporated to dryness, dissolved in acetone and analysed on a gas chromatograph equipped with a alkali-flame detector and a 1 m packed column of 10% DC 200 and 1.5% QF-1, run isothermally at 210°C. Calibration is with external standards taken through the oxidation step, or ODM sulfone may be used as the standard. The reported recovers are shown in Table16.

Table 16: Recovery of ODM and demeton-S-methyl from various samples by Bayer Method 00246.

Sample	Fortification, mg/kg (ODM and demeton-S-methyl at same level)	ODM		Demeton-S-methyl	
		Number	Recovery, %	Number	Recovery, %
Apples	0.05-0.10	8	108±10	6	94±6.6
Grapes	0.05-0.10	3	103±5.2	3	85±2.9
Lettuce	0.05-0.50	10	93±7.8	12	99±11
Nut meat	0.05-0.10	6	100±13	5	102±9.1
Animal tissue	0.05-0.10	17	94±7.8	13	92±8.4
Animal fat	0.05-0.10	5	93±4.2	5	94±3.7
Bovine milk	0.005-0.01	6	93±6.5	6	96±13

Bayer AG reported several similar methods for the determination of ODM in field trial samples. All involve solvent extraction, clean-up of the extract, oxidation of the residue with permanganate, post-oxidation clean-up and gas chromatography. The differences are in the extraction solvents, the types of clean-up and the detector. The newer methods use flame photometric detectors are used instead of alkali FIDs. All the methods determine the combined residue of demeton-S-methyl, its sulfoxide (ODM) and its sulfone as the sulfone.

In Method I47 (Thornton and Olson, 1967, revised 1971), the plant sample (100 g) is chopped and extracted with acetone (200 ml) and water. The filtrate is mixed with skellysolve B and the acetone phase is extracted with chloroform (200 + 50 ml). The solvent is changed to acetone and the extract oxidized with permanganate. The mixture is extracted with chloroform (3 x 20 ml). The combined extracts are redissolved in acetone and analysed by GLC as in Method 00246. Calibration is by external standard taken through the oxidation step. The LOD is estimated to be 0.05 mg/kg. Recoveries at 0.1 and 0.4 mg/kg (5 replicates at each level) were 68-104% for ODM and 77–102% for ODM sulfone from lettuce, 70-104% for ODM and 81-116% for ODM sulfone from sugar beet roots, and 76-110% for ODM and 90-108% for ODM sulfone in sugar beet tops.

Method RA-133/74 (Wagner, 1974) is a modification of Method I47. Only the extraction procedure varies. The sample (100 g) is macerated with acetone (200 ml) and water (to 300 ml total volume) and an aliquot of the filtrate (150 ml) is partitioned with chloroform (5 x 100 ml). The solvent is changed to acetone and the residues oxidized with 0.5 N potassium permanganate solution (30 minutes at room temperature). Recoveries from various plant materials fortified at 0.1 mg/kg were reported to be 67-96%, but no details were provided. The nominal limit of quantification was 0.01 or 0.05 mg/kg.

Bayer Method 148 (Olson, 1967, revised 1971) was for crops with a high oil content, such as cotton seed, or dry crops such as walnuts. The sample (100 g) is blended with chloroform (300–500 ml). After filtration, the chloroform is stripped and the residue dissolved in skellysolve B (200 ml). This is extracted with water (2 x 50 ml) and the combined water extracts are partitioned with chloroform (300 ml, 2 x 100 ml). The residue is oxidized with permanganate and the final solution is analysed by gas chromatography with a flame ionization detector. The recoveries at 0.1 mg/kg were 101% for ODM and 103% for ODM sulfone from cotton seed (single samples) and 106% and 117% for ODM and 104% for ODM sulfone from walnuts. The limit of quantification is estimated to be 0.01 mg/kg.

Bayer Method 00009 and its variants are the most commonly used with field trials (Wagner and Thornton, 1977). Plant samples are macerated with acetone and filtered. The macerate is extracted 3 times with chloroform and the solvent changed to acetone. The solution is oxidized with potassium permanganate and the mixture extracted 3 times with chloroform. The organic fraction is dried with sodium sulfate, re-dissolved in acetone, and determined as ODM sulfone by gas chromatography (flame ionization detector). Recoveries are shown in Table 17.

Several variations of 00009 exist. M 003 uses methylene chloride in the initial extraction of the macerate. The limit of quantification is about 0.05 mg/kg (Blass, 1990). In M 004 the proportions of solvents are changed, and toluene is substituted for acetone as the final (GLC) solvent¸ and an FPD is used. The limit of quantification is about 0.01 mg/kg.

In modification M 002 (Ohs, 1988) the oxidation mixture is on an “Extrelut” column and the sulfone is eluted with methylene chloride. A flame photometric detector replaces the thermionic detector. M 007 (Seym, 1993) is a further modification of M 002. The methylene chloride extract from the “Extrelut” column is dissolved in cyclohexane and cleaned up on a Mega Bond silica gel cartridge (SPE), which is rinsed 3 times with cyclohexane/ethyl acetate and eluted with ethyl acetate and acetonitrile. Recoveries are shown in Table 17.

Method 00015/M 009 is used specifically for the determination of “Enduro”, a mixture of ODM and cyfluthrin. The procedure for ODM is similar to method 00009 with modification M 002 and electron capture detection. The limit of quantification is 0.04–0.05 mg/kg.

Method 00255 (Ohs, 1992) is also used for the determination of ODM and cyfluthrin. The plant samples are extracted with an acetone/water mixture and the filtered extract is partitioned with methylene chloride. Oily samples are taken up again in acetonitrile and partitioned with n-hexane. The solvent is changed to acetone and the residue oxidized with potassium permanganate. The product mixture is cleaned up on a Chem-Elut column eluted with methylene chloride. The chromatograph is equipped with

a 30 m x 0.53 mm megabore column (HP 1) and the oven is programmed from 205°C to 280°C at 20°/min. The residues re determined as in 00009/M 002. Recoveries are shown in Table 17.

Table 17. Recoveries of ODM from various crops by Method 00009 and its variants (single analyses).

Crop	Commodity	Fortification, mg/kg	Recovery, %		
			Demeton-S-methyl	ODM	ODM sulfone
Method 00009					
Apples		0.01	100	108	
		0.1	95	72	
		0.5		75	97
		1.0	100		97
Potatoes		0.01	100	99	100
		0.1	89		100
		0.5		86	98 (0.4 mg/kg)
		1.0	95		
Lettuce		0.01		96	
		0.1	90		
		0.2		78	
		0.6		85	
		1.0	91		
Grapes		0.01	95		
		0.1		82	98
		0.5		91	
		1.0			91
	wine	0.1			100
		0.2		114	
Wheat	grain	0.01	81	88	
		0.1	91		
		0.2		87	
		0.4		88	
		1.0	100		
	straw	0.1	84		
		1.0	90	96	
Sugar beet	root	0.01	100	81	
		0.05			86
		0.5		76	98

Crop	Commodity	Fortification, mg/kg	Recovery, %		
			Demeton-S-methyl	ODM	ODM sulfone
		1.0		81	
	tops	0.05			96
		0.5			100
		1.0		85	
Bush beans		0.1		72	
		0.6		67	
Strawberries		0.2		72	
		0.5		80	101
Currants		0.05		90	
		0.1			100
		0.5		98	
		1.0		90	100
Potatoes		0.01		99	100
		0.05			102
		0.1			100
		0.2			101
		0.4			98
		0.5		86	
Kohlrabi		0.1		85	
		1.0		72	
Carrots		0.01		91	100
		0.05			102
		0.1		77	
		0.4			102
Grape must		0.05		88	
		0.5			94
Peaches		0.05		80	
		0.5		94	
Plums		0.05		98	
		0.1			97
		0.5		80	
		1.0			96
Spinach		0.1		72	
		0.6		83	
Tomatoes		0.1		81	

Crop	Commodity	Fortification, mg/kg	Recovery, %		
			Demeton-S-methyl	ODM	ODM sulfone
Cabbage (Savoy)		0.5		82, 76	
Modification M 007					
Lettuce	head	0.02		91, 96	
		0.50		76, 77	
Kale		0.02		65, 75	
		0.50		71, 83	
Almonds	kernels	0.02		83, 85	
		0.50		93, 94	
Modification M 002					
Fodder beet	leaves	0.02		80	
		0.05		92	
		0.2		73	
		1.0		92	
		2.0		84	
	roots	0.02		88	
		0.2		83	
Modification M 003					
Cabbage	head	0.05		74, 76	
		1.0		77, 88	
	cooked head	0.05		75, 81	
		1.0		65, 65	
Modification M 004					
Orange	pulp	0.1		75, 80	
	peel	0.1		75, 80	
Cauliflower	head	0.1		94, 94	
Kale	head	0.1		89, 94	
Peaches	fruit	0.1		94, 94	
Cabbage (Savoy)	head	0.1		99, 103	
Sugar beet	leaves	0.01		94, 94	
	roots	0.01		94, 94	
Modification M 009					
Wheat	forage	0.05		90	
		2.0		86	
	grain	0.05		96	
		2.0		116	

Crop	Commodity	Fortification, mg/kg	Recovery, %		
			Demeton-S-methyl	ODM	ODM sulfone
	straw	0.05		77	
		2.0		93	
Barley	forage	0.04		92, 97, 90, 95	
		2.0		110	
	grain	0.04		100, 100, 108, 121	
		2.0		116	
	straw	0.04		129, 129, 77, 81	
		2.0		98	
Oats	forage	0.04		97	
		2.0		99	
	grain	0.04		97	
		2.0		116	
	straw	0.04		89	
		2.0		95	
Method 00255					
Peas	forage	0.01		85, 101	
		0.05		74, 77, 93	
	vines (green)	0.01		81, 83	
		0.1		84, 90, 98	
	seeds	0.01		78, 98, 108, 123	
		0.1		84, 90, 98	
Field pea	vines (green)	0.01		92, 101	
		0.1		81, 87	
	vines (dry)	0.04		73, 86	
	seeds	0.01		112, 115	
		0.1		91, 102	
Barley	forage	0.04		94, 96	
		0.08		93	
		0.4		83, 97	
		0.8		92	
	grain	0.04		76, 99	
		0.4		88	
	straw	0.04		74, 76, 111	
		0.4		79, 72	
Oats	forage	0.04		102, 102	

Crop	Commodity	Fortification, mg/kg	Recovery, %		
			Demeton-S-methyl	ODM	ODM sulfone
	grain	0.04		87, 96, 98	
		0.4		79, 85	
	straw	0.04		99, 102	
		0.4		77, 79, 91	
Potatoes	tubers	0.01		93, 94, 88	
		0.1		73, 86, 89	
Wheat	forage	0.04		64, 74, 84, 88, 111, 121	
s		2.0		85, 101	
	grain	0.04		93, 100, 114, 118	
		0.4		104, 106	
	straw	0.04		76, 91, 94, 96	
		0.4		74	
		2.0		94	
Sugar beet	roots	0.01		90, 92, 96	
		0.1		61, 71, 73, 75, 80, 85, 89	
	leaves	0.04		92, 93	
		0.4		65, 68, 69, 70, 73, 75, 79, 79	
		2.0	79; 94		

Stability of pesticide residues in stored analytical samples

Bayer AG reported a study of the storage stability of ODM in cabbage (Lenz and Smyser, 1989) and summary information on its stability in lettuce, papaya and maize (Morris, 1980a,b; Anon., 1985). The cabbage trial was part of a study of metabolism. Cabbages treated with [^{14}C]ODM were analysed, stored frozen at –10^{o}C, then re-analysed for ODM and ODM sulfone after 5 days and 4, 10 and 115 weeks of frozen storage. The samples were extracted with acetone (97% efficiency), partitioned into chloroform and spotted on TLC plates. Radioactive areas were defined by autoradiography, scraped and quantified by LSC. The methods used for maize, lettuce and papaya were I47 and I48.

The results are shown in Table 18.

Table 18. Stability of ODM and ODM sulfone in frozen plant samples.

Sample	Storage, days	Storage temperature, °C	ODM recovery, %	Sulfone recovery, %
Cabbage	28	-10	89[1]	133[1]
	49		108[1]	116[1]
	805		115[1]	123[1]
Maize forage (green)	14	-18 to -23	91	94
	43		101	65
	62		74	97
	157		59	91
Maize kernels	15		106	109
	43		108	111
	93		-	146
Maize cob	14		102	84
	43		69	60
Maize husk	15		85	103
	43		91	98
	93		127	98
Lettuce	51	-18 to –23	89	
	183		98 (69; 128)	
Papaya	9	-18 to –23	118[1]	
	10		131	
	13		134	

[1] Relative to the radioactive components at 5 days. No day 0 samples analysed

Definition of the residue

On the basis of the metabolism of oxydemeton-methyl (ODM) by plants and animals, and the residues determined by the available analytical methods, the residue should be defined as the sum of demeton-S-methyl, ODM and demeton-S-methylsulphon, expressed as ODM, both for compliance with the MRLs and for the estimation of dietary intake.

USE PATTERN

Bayer AG submitted labels and extensive information on GAP. The labels from Finland, France, Germany, Morocco, Sweden, Portugal and Austria were not however translated into English and the information could not be adequately verified in those cases. New and presumably unapproved labels were provided for Austria, Greece and Spain, but only the label for Spain had an adequate translation. The Netherlands stated that neither demeton-S-methyl nor ODM were approved for use. Germany submitted information on GAP. Bayer AG emphasized that the labels were for their formulations only. The information is shown in Table 19.

Table 19. Registered uses of ODM on crops.

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
Almonds	Spain	EC 250 g/l	0.25 1000	0.025	2	Individual tree spray	30	
Anise	Turkey	EC 265 g/l	0.26 600	0.044	1	Broadcast spray	21	
Apple	Austria	EC 250 g/l	0.38 1500	0.025	3	Row spraying	35	
		SL 100 g/l	0.25 1500		2	Row spraying	90	
	Finland	EC 250 g/l		0.025	2	Broadcast spray		
	France	EC 250 g/l		0.020		Broadcast spray	60	
	Germany	EC 250 g/l	0.38 1500	0.025	1	Row spray	~60	Before or directly after flowering
		SL 100 g/l	0.38 1500	0.025	3	Row spray	28	
	India	EC 250 g/l	1.8 2500	0.07	2	Individual tree spray	21	
	Ireland	EC 250 g/l		0.0075 high volume (1500 l water max /ha) 0.081 low volume (200 l water/ha)	2	Foliar	~90	Apply before and/or immediately after flowering
	Italy	SL 189 g/l	0.34 1200	0.028	1	Broadcast spray	30	
	Mexico	EC 250 g/l	0.75 l/ha 1500 l water/ha	-	1 or 2	Broadcast spray	30	
	Morocco	EC 250 g/l	0.38 1500	-	4	Broadcast spray	21	
	Peru	EC 250 g/l	0.2 800	-	2	Broadcast spray	30	
	Portugal	EC 250 g/l	0.37 1000 (calculated)	0.037	2	Broadcast spray	56	
	South Africa	EC 250 g/l	0.88 3500	0.025	1	Individual plant spray	21	
	Spain	EC 250 g/l	0.25 1000	0.025	2	Individual plant spray	30	`
	Turkey	EC 250 g/l	0.53	0.026	1	Individual tree spray	21	
Apricot	Germany	EC 250 g/l	0.38 1500	0.025	1	Row spray	28	
	Germany	SL 100 g/l	0.38 1500	0.025	3	Row spray	28	
	South Africa	EC 250 g/l	0.88 3500	0.025	1	Individual tree spray	21	
	Spain	EC 250 g/l	0.25 1000	0.025	2	Individual tree spray	30	
Banana	India	EC 250 g/l	0.25 500	0.05	1	Broadcast spray	21	
Barley	Austria	EC; SL 250 g/l;	0.12 400	0.031	1	Broadcast spray	21	

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
		100 g/l						
	Germany	EC 250 g/l 375 g/l	0.12 400	0.031	2	Broadcast spray	21	
	Ireland	EC 250 g/l	0.12 (200 l water/ha)		1	broadcast	21	
	Kenya	EC 250 g/l	0.12 800	0.016	2	Broadcast spray	-	
	Peru	EC 250 g/l	0.2 ?	-	1	-	14	
	South Africa	EC	0.12 300	0.042	2	Broadcast spray	33	
Bean	Italy	SL 189 g/l	0.17 600	0.028	1	Broadcast spray	21	
	Kenya	EC 250 g/l	0.15 800	0.019	2	Broadcast spray	-	
	Mexico	EC 250 g/l	0.25 400	0.062	1 or 2	Broadcast spray	21	
	South Africa	EC 250 g/l	500	0.02	2	Broadcast spray	14	
Bean, Field	Germany	EC 250 g/l	0.15 0.22	-	2	Broadcast spray	28	Under 50 cm uses lower rate.
	Ireland	EC 250 g/l	0.15 0.22 (200 l water/ha)	-	2	Broadcast spray	28	Lower rate for plants under 50 cm.
	Sweden	EC 250 g/l	400	-	1	Individual leaf spray	21	
Bean, French	Germany	EC 250 g/l 375 g/l	0.17–0.33	-	3	Broadcast spray	7	0.17 if <50 cm 0.25 if 50-125 cm
		SL 100 g/l	0.23	-	2	Row spray	7	
Bean, mung	India	EC 250 g/l	0.12 500	0.025	1	Row spray	14	
Beet, beta	France	EC 250 g/l	0.38 300	0.12	3	Broadcast spray	-	
Beet, fodder	Germany	EC 250 g/l 375 g/l 590 g/l	0.24 400	0.06	4	Broadcast spray	28	
Broccoli	Germany	EC 375 g/l	0.12	-	1	Broadcast spray	21	
	Mexico	EC 250 g/l	0.25 400	-	1 or 2	Broadcast spray	7	
	South Africa	EC 250 g/l	0.1 500	0.02	2	Row spray	10	
Brussels sprouts	Austria	EC 250 g/l	0.25 1000	-	1	Row spray	21	
	Germany	EC 375 g/l	0.12 or 0.19	-	2	Broadcast spray	14	Higher rate >50 cm
	Mexico	EC 250 g/l	0.25 400	-	1 or 2	Broadcast spray	7	
	South Africa	EC 250 g/l	0.1 500	0.02	2	Row spray	14	

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
Cabbage	Austria	EC 250 g/l SL 100 g/l	0.25 1000	-	1	Row spray	21	Savoy and head
	Germany	EC 375 g/l	0.12	-	2	Broadcast spray	21	Head, red, Savoy
		EC 250 g/l SL 100 g/l	0.15		1	Broadcast spray	21	Head, red, Savoy. Under 50 cm
	Ireland	EC 250 g/l	0.15		1	Broadcast spray	21	Under 50 cm
	Italy	SL 189 g/l	0.28 1000	0.028	1	Broadcast spray	-	Wild
	Mexico	EC 250 g/l	0.25 400	-	1 or 2	Broadcast spray	21	Wild
	South Africa	EC 250 g/l	0.1 500	0.02	2	Row spray	10	Wild
Cauli-flower	Austria	EC 250 g/l	0.25 1000	-	1	Row spray	21	
		SL 100 g/l	0.22 1000	-	1	Row spray	21	
	Germany	EC 375 g/l	0.12	-	1 or 2	Broadcast spray	14	
		EC 250 g/l	0.15	-	1	Broadcast spray	21	Under 50 cm
	Ireland	EC 250 g/l	0.15	-	1	Broadcast spray	21	Under 50 cm
	Mexico	EC 250 g/l	0.25 400	-	1 or 2	Broadcast spray	7	
	South Africa	EC 250 g/l	0.1 500	0.02	2	Row spray	10	
Cereals	France	EC 250 g/l	0.10			Foliar	21[1]	
Cherry	Austria	EC 48 g/l	0.36 1500	-	3	Row spray	35	Dwarf and sweet
	Spain	EC 250 g/l	0.25 1000	0.025	2	Individual tree spray	30	Sweet
Chilli	Sri Lanka	EC 250 g/l	0.25 665	0.038	6–8	Broadcast spray	21	
Citrus	India	EC 250 g/l	1.25 2500	0.05	1	Individual tree spray	21	
	Kenya	EC 250 g/l	0.25 1000	0.025	3	Broadcast spray	-	
	Portugal	EC 25 g/l	0.1 2000	0.005	1	Row spray	35	
	Portugal	EC 250 g/l	0.75 calc	0.050	2	Foliar	84	
	South Africa	EC 250 g/l	0.1 500	0.02	1 or 2	Broadcast spray	-	
Coconut	Sri Lanka	EC 250 g/l	0.6 1	60	1	Trunk inject	21	
Coffee	India	EC 250 g/l	0.62 1000	0.062	1	Individual tree spray	21	
Cotton	India	EC 250 g/l	0.25 500	0.05	2	Broadcast spray	21	

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
	Kenya	EC 250 g/l	0.38 800	0.047	2	Broadcast spray	-	
	Mexico	EC 250 g/l	0.38 400	-	1 or 2	Broadcast spray	14	
	Morocco	EC 250 g/l	0.25 1000	-	2	Row spray	21	
	Peru	EC 250 g/l	0.25 100	-	3	Broadcast spray	14	
	South Africa	EC 250 g/l	0.12 300	0.042	2	Broadcast spray	21	
	USA	EC 250 g/l	0.84		3	Broadcast spray	14	
	Turkey	EC 265 g/l	0.26 600	0.044	1	Row spray	21	
Cucum-ber	Germany	EC 250 g/l	0.3	-	3	Broadcast spray	4	0.17 if <50 cm
	Italy	SL 189 g/l	0.23 800	0.028	1	Broadcast spray	21	
	Mexico	EC 2509 g/l	0.75 400	-	1 or 2	Broadcast spray	-	
Cucur-bits	Peru	EC 250 g/l	0.2 800	-	2	Broadcast spray	14	
Currant	Austria	EC 48 g/l	0.36 1500	-	3	Row spray	35	Red
Egg plant (aubergi ne)	India	EC	0.25 l/ha 500 l water/ha	0.05	2	Broadcast spray	14	
	Mexico	EC	0.5 l/ha 400 l water/ha	-	1 or 2	Broadcast spray	7	
	South Africa	EC	0.1 l/ha 500 l water/ha	0.02	2	Row spray	14	
Endive	Germany	EC 375 g/l 250 g/l	0.12 0.15	-	2 ?	Broadcast spray	14	winter
Fodder peas	France	EC 250 g/l	0.10			Broadcast spray	-	
Garlic	India	EC 250 g/l	0.12 500	0.025	2	Broadcast spray	14	
Gourd	Mexico	EC 250 g/l	0.75 400	-	1 or 2	Broadcast spray	14	
	Peru	EC 250 g/l	0.4	-	2	-	14	
Grapes	Austria	SL 100 g/l	0.2 1000	0.02	1	Row spray	-	European
	Germany	EC 250 g/l	0.5 2000	0.025	1	Row spray	-	European. Applied from 2 leaf stage to inflorescence
	India	EC 250 g/l	0.19 500	0.038	2	Broadcast spray	21	European
	Italy	SL 189 g/l	0.23 800	0.028	1	Broadcast spray	40	European
	Mexico	EC 250 g/l	0.5 600	0.083	1 or 2	Broadcast spray	90	European
	Morocco	EC	0.02	-	1	Row spray	21	European

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
		250 g/l	800					
Grape-fruit	Mexico	EC 250 g/l	0.75 1500	-	1 or 2	Broadcast spray	7	
Ground-nut	India	EC 250 g/l	0.25 500	0.038	2	Broadcast spray	14	
	South Africa	EC 250 g/l	0.12 500	0.025	2	Broadcast spray	21	
Kale	Austria	SL 100 g/l	0.22 1000	-	1	Row spray	21	Including curly. turnip
	Germany	EC 250 g/l	0.22	-	2	Broadcast spray	14	Curly only
		SL 100 g/l	0.15	-	2	Broadcast spray	14	Including curly. Turnip
		EC 250 g/l	0.15	-	1	Broadcast spray	21	Under 50 cm
	Ireland	EC 250 g/l	0.15	-	1	Broadcast spray	21	Under 50 cm.
		EC 375 g/l	0.12	-	2	Broadcast spray	14	Turnip
Kohlrabi	Germany	EC 214 g/l	0.13 400	0.021	2	Broadcast spray	14	
		EC 250 g/l	0.15		1	Broadcast spray	21	Under 50 cm
Leek	Italy	SL 189 g/l	0.17 600	0.028	1	Broadcast spray	-	Common
Lemon	Mexico	EC 250 g/l	0.75 1500	-	1 or 2	Broadcast spray	7	
Lettuce	Austria	EC 250 g/l SL 100 g/l	0.15 600	0.025	1	Row spray	21	Cutting
	Germany	EC 375 g/l	0.12	-	2	Broadcast spray	14	Cutting, head
		EC 250 g/l	0.16	-	1	Broadcast spray	21	Cutting, head
	Ireland	EC 250 g/l	0.15 (200 l water/ha)	-	1	Broadcast spray	21	
		SL 100 g/l	0.15	-	2	Broadcast spray	14	Head
	Mexico	EC 250 g/l	0.5 400	-	1	Broadcast spray	21	
Maize	India	EC 250 g/l	-	-	-	-	14	
	Kenya	EC 250 g/l	0.12 800	0.016	2	Broadcast spray	-	
	Mexico	EC 250 g/l	0.25 400	-	1	Broadcast spray	7	
	South Africa	EC 250 g/l	0.12 300	0.042	1	Broadcast spray	21	
Mango	India	EC 250 g/l	2.5 2500	0.1	2	Individual tree spray	21	
Melon	Mexico	EC 250 g/l	0.75 400	-	1	Broadcast spray	14	
	Portugal	EC 25 g/l	0.025 1000	0.0025	1	Row spray	35	
Mustard	India	EC	0.19	0.038	1	Broadcast	14	Black

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
		250 g/l	500			spray		
Oats	Austria	EC 250 g/l	0.12 400	0.031	1	Row spray	21	
Okra	India	EC 250 g/l	0.25 500	0.05	2	Broadcast spray	7	
Oil seed	France	EC 250 g/l	0.15			Broadcast	-	
Olive	South Africa	EC 250 g/l	0.25 1000	0.025	2	Individual tree spray	90	
Onion	India	EC 250 g/l	0.19 500	0.038	2	Broadcast spray	14	
	Mexico	EC 250 g/l	0.25 400	-	1	Broadcast spray	30	
	South Africa	EC 250 g/l	0.075 1000	0.0075	2	Broadcast spray	21	
Orange	Mexico	EC 250 g/l	0.75 1500	-	1 or 2	Broadcast spray	7	
	Spain	EC 250 g/l	0.75 3000	0.025	2	Individual plant spray	30	Sweet. Mandarin.
Paprika	India	EC 250 g/l	0.25 500	0.05	2	Broadcast spray	14	
Pea	Germany	EC 375 g/l	0.18	-	2	Broadcast spray	7	Garden
	India	EC 250 g/l	0.19 500	0.038	1	Broadcast spray	14	Garden
	Italy	SL 189 g/l	0.23 800	0.0283	1	Broadcast spray	21	Outdoor and greenhouse. Garden.
	Mexico	EC 250 g/l	0.25 400	0.062	1	Broadcast spray	21	
	South Africa	EC 250 g/l	0.12 500	0.025	21	Broadcast spray	21	
	Sweden	EC 100 g/l	0.2 400	-	1	Individual leaf spray	21	Garden
Peach	Austria	EC 48 g/l	0.36 1500	-	3	Row spray	35	
	Germany	EC 250 g/l	0.38 1500	0.025	1	Broadcast spray	28	
		SL 100 g/l	0.38 1500	0.025	3	Row spray	28	
	India	EC 250 g/l	0.62 2500	0.025	1	Individual tree spray	21	
	Italy	SL 189 g/l	0.28 1000	0.028	1	Broadcast spray	30	
	Portugal	EC 25 g?l	0.05 1000	0.005	1	Row spray	35	
	South Africa	EC 250 g/l	0.88 3500	0.025	1	Individual tree spray	21	
	Turkey	EC 265 g/l	0.53 2000	0.0265	1	Individual tree spray	21	
Pears	France	EC 250 g/l		0.02		Broadcast spray	-	
	Germany	EC 250 g/l SL 100 g/l	0.38 1500	0.025	1	Broadcast spray (EC) Row spray (SL)	~60	For EC, apply before or directly after flowering
	Ireland	EC 250 g/l	0.32 calculated at	0.0075 high volume	2	Foliar	~90	Apply before and/or directly after

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
			low volume	0.081 low volume (200 l water/ha)				flowering
	Italy	SL 189 g/l	0.34 1200	0.028	1	Broadcast spray	30	
	Mexico	EC 250 g/l	0.75 1500	-	1	Broadcast spray	30	
	Portugal	EC 250 g/l	0.38 1000 (calculated)	0.038	2	Row spray	56	
	South Africa	EC 250 g/l	0.88 3500	0.025	1	Individual plant spray	21	
	Spain	EC 250 g/l	0.25 1000	0.025	2	Individual tree spray	30	
	Turkey	EC 265 g/l	0.53 2000	0.026	1	Individual tree spray	21	
Pepper, Sweet	Mexico	EC 250 g/l	0.5 400	-	1	Broadcast spray	-	
	South Africa	EC 250 g/l	0.1 500	0.02	2	Broadcast spray	14	
Pistachio	Turkey	EC 265 g/l	0.80 2000	0.040	1	Individual tree spray	21	
Plum	Austria	EC 48 g/l	0.37 1500	-	3	Row spray	35	
	Germany	EC 250 g/l SL 100 g/l	0.38 1500	0.025	1	Row spray	~60	Including Syrian plum. Before or directly after flowering.
	India	EC 250 g/l	0.62 2500	0.025	1	Individual Plant spray	21	
	Ireland	EC 250 g/l		0.022 high volume 0.19 low volume (200 l water/ha)	1	Foliar	~90	Apply before cot-split stage.
	Mexico	EC 250 g/l	0.88 1500	-	1	Broadcast spray	35	
	South Africa	EC 250 g/l	0.88 3500	0.025	1	Individual tree spray	21	
	Spain	EC 250 g/l	0.25 1000	0.025	2	Individual tree spray	30	
Pome fruit	Germany	EC 276 g/l	0.41 1500	0.027	3	Broadcast spray	28	Status of this formulation is unknown.
	Germany	EC 250 g/l	0.40	0.026	1	Foliar	60	
	France	EC 250 g/l		0.020		Foliar	60	
Potato	Austria	EC 250 g/l SL 100 g/l	0.25 400	0.0625	3	Broadcast spray	42	
	Germany	EC 375 g/l	0.3 400 600 pending	0.075	3	Broadcast spray	21 28 pending	
	India	EC	0.25	0.05	1	Broadcast	14	

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
		250 g/l	500			spray		
	Ireland	EC 250 g/l	0.15 0.22 (200 l water/ha)		2-4	Broadcast spray	28	Lower rate under 50 cm.
	Italy	SL 189 g/l	0.23 800	0.028	1	Broadcast spray	21	
	Mexico	EC 250 g/l	0.38 400	-	1	Broadcast spray	7	
	Peru	EC 250 g/l	0.3 800	-	2	Broadcast spray	21	
	South Africa	EC 250 g/l	0.25 500	0.05	4	Broadcast spray	21	
	Spain	EC 250 g/l	0.3 600	-	3	Individual plant spray	30	
Quince	France	EC 250 g/l	0.25 1000	0.025	2	Broadcast spray	-	
Radish	India	EC 250 g/l	0.19 500	0.038	1	Broadcast spray	21	
Rape	France	EC 250 g/l	0.12 400	0.031		Broadcast spray	-	
Rice	India	EC 250 g/l	0.25 500	0.05	1	Broadcast spray	14	Planted (paddy)
Rye	Austria	EC 250 g/l SL 100 g/l	0.12 400	0.031	1	Broadcast spray	21	
	Germany	EC 375 g/l	0.12 400	0.03	2	Broadcast spray	21	
	Ireland	EC 250 g/l	0.12 (200 l water/ha)		1	Broadcast spray	21	
Safflow-er	India	EC 250 g/l	0.15 500	0.03	1	Broadcast spray	14	
	Mexico	EC 250 g/l	0.25 400	0.062	1	Broadcast spray	7	
Sesame	India	EC 250 g/l	0.15 500	0.03	1	Broadcast spray	14	
Sorghu m	India	EC 250 g/l	-	-	-	-	14	Grain
	Mexico	EC 250 g/l	0.25 400	-	1	Broadcast spray	45	Grain
	South Africa	EC 250 g/l	0.12 200	0.062	2	Broadcast spray	57	Grain
Soya bean	India	EC 250 g/l	0.19 500	0.038	1	Broadcast spray	14	
Spinach	Germany	EC 375 g/l	0.12	0.022	2	Broadcast spray	14	
Stone fruit (except cherry)	Germany	EC 276 g/l	0.41 1500	0.027	2	Broadcast spray	28	
Straw-berry	Germany	EC 250 g/l	0.5 2000	0.025	1	Broadcast spray	-	
	Ireland	EC 250 g/l		0.022 high volume 0.25 low	1	Broadcast	Post harv est	

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
				volume (200 l water/ha)				
	Mexico	EC 250 g/l	0.25 400	-	1	Broadcast spray	3	
	Portugal	EC 250 g/l	0.025 1000	0.0025	1	Row spray	-	
Sugar beet	Austria	EC 250 g/l SL 100 g/l	0.2 400	-	2	Broadcast spray	28	
		EC 48 g/l	0.1 400	0.024	2	Broadcast spray	35	
	France	EC 258 g/l	0.15 400	0.038		Broadcast spray	28	
		EC 250 g/l	0.38 300	0.12	3	Broadcast spray	-	
	Germany	EC 590 g/l	0.24 400	0.06	4	Broadcast spray	28	
		EC 375 g/l	0.2 400	0.05	2	Broadcast spray	28	
		EC 250 g/l	0.38		1	Broadcast spray	28	
	Italy	SL 189 g/l	0.14 500	0.028	1	Broadcast spray	30	
	Morocco	EC 250 g/l	0.25 600	-	1	Row spray	21	
	Spain	EC 250 g/l	0.38 600	-	3	?	30	
	Sweden	EC 100 g/l	0.2 400	-	1	Individual leaf spray	21	
Sun-flower	France	EC 258 g/l	0.1 400	0.025	1	Broadcast spray	-	
	France	EC 250 g/l	0.10			Broadcast spray	-	
Tea	South Africa	EC 250 g/l	0.12 500	0.025	1	Individual plant spray	21	
Tomato	Germany	EC 250 g/l	0.33 1200	0.028	3	Broadcast spray		0.17 if <50 cm
	India	EC 250 g/l	0.25 500	0.05	2	Broadcast spray	14	
	Italy	SL 189 g/l	0.23 800	0.028	1	Broadcast spray	21	Outdoors and green-house
	Portugal	EC 25 g/l	0.025 1000	0.0025	1	Row spray	35	
	South Africa	EC 250 g/l	0.1 500	0.02	2	Row spray	14	
Triticale	France	EC 258 g/l	0.1 400	0.025	3	Broadcast spray	-	See wheat
Turmeric	India	EC 250 g/l	0.25 500	0.05	2	Broadcast spray	14	
Walnut	Mexico	EC 250 g/l	0.88 1500	-	1	Broadcast spray	30	
Water-melon	Peru	EC 250 g/l	0.4	-	2	-	14	
Wheat	Austria	SL 100 g/l EC	0.12 400	0.031	1	Broadcast spray	21	

Crop	Country	Form	Application				PHI, days	Comments
			Rate, kg ai/ha & l water/ha	Spray conc., kg ai/hl	No.	Method		
		250 g/l						
	Germany	EC 375 g/l	0.12 400	0.03	2	Broadcast spray	21	
		EC 250 g/l	0.14 400	0.03	1	Broadcast spray	21	
	India	EC 250g/l	0.12 500	0.025	1	Broadcast spray		Soft
	Ireland	EC 250 g/l	0.12 (200 l water/ha)		1	Broadcast spray	21	Includes triticale
	Italy	SL 189 g/l	0.17 600	0.028	1	Broadcast spray	30	Soft
	Kenya	EC 250 g/l	0.12 800	0.016	2	Broadcast spray	-	Soft
	Peru	EC 250 g/l	0.2 600	-	2	Broadcast spray	14	Soft
	Portugal	EC 250 g/l	0.12		1	Broadcast spray	21	Winter
	South Africa	EC 250 g/l	0.12 300	0.042	2	Broadcast spray	48	soft

[1] or unspecified

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of the supervised residue trials are shown in Tables 20–30. The trials were reported in sufficient detail, including acceptable information on sample storage and analysis, unless otherwise noted.

The trials are reviewed in the sequence of the Codex Alimentarius Classification of Foods and Feeds.

Citrus fruits

Trials were reported on the spray application of ODM to oranges and lemons in Spain, Italy and Portugal (Seym, 1996; Seym and Heinemann, 1996). GAP for oranges in Spain is 2 x 0.75 kg ai/ha, 30-day PHI (90 days proposed), and for citrus fruit in Portugal 2 x 0.050 kg ai/hl (0.75 kg ai/ha), 84-day PHI. GAP for Italy was not available and is assumed to be that of Portugal.

Table 20. Residues of ODM + ODM sulfone following application of ODM EC formulation to lemons and oranges in Spain, Italy and Portugal.

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg[1]	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment, (days), Growth stage at final application			
Orange						
Pobla Llarga, Spain 1994	0.75	3000	2 15 early fruit setting	0 21 35 64 pulp 92 64 peel 92	0.33 0.03 0.01 <0.01 <u><0.01</u> 0.01 <u>0.01</u>	Method 00009/ M 004 Recovery 83–101% at 0.01 and 0.1 mg/kg. Up to 11 months from sampling to extraction
Vinaroz, Spain 1994	0.75	3000	2 15 30% final size	0 21 35 62 pulp 90 62 peel 90	0.83 0.04 0.01 <u><0.01</u> <0.01 <u><0.01</u> <0.01	
Pobla Llarga, Spain 1994	0.75	3000	2 19 early fruit setting	0 92 pulp 92 peel	0.22 <u><0.01</u> <u>0.01</u>	
Riudoms, Spain 1995	0.75	3000	2 30 start of ripening	0 89 pulp 89 peel	0.26 <u>0.02</u> <u>0.13</u>	Method 00009/ M 007. Recovery 71–098% at 0.01 and 0.1 mg/kg from pulp and 79–108% from peel (n = 10)
Chamusca, Portugal 1995	0.75	3000	2 31 start of ripening	0 18 35 pulp 35 peel 62 pulp 62 peel	0.28 0.10 0.03 0.21 <u>0.02</u> <u>0.09</u>	
Raval de Jesus, Spain 1995	0.75	3000	2 28 start of ripening	0 90 pulp 90 peel	0.26 <u>0.04</u> <u>0.13</u>	
Sta. Barbara, Spain 1995	0.75	3000	2 29 Begin of ripening	0 90 pulp 90 peel	0.17 <u>0.01</u> <u>0.06</u>	
Lemons						
Alhama de Murcia, Spain 1995	0.75	3000	2 29 Bloom to fruit growing	0 fruit 21 fruit 35 pulp 35 peel 63 pulp 63 peel 90 pulp 90 peel	0.56 0.03 <0.01 0.02 <0.01 <0.01 <u><0.01</u> <u><0.01</u>	Method 00009/ M 007. Recovery 68–79% at 0.01 and 0.1 mg/kg from pulp and 75–83% from peel (n = 4).
Avola, Italy 1995	0.75	2500 3000	2 30 20% fruit size	0 fruit 90 pulp 90 peel	0.95 <u><0.01</u> <0.01	

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg[1]	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment, (days), Growth stage at final application			
Siracusa, Italy 1995	0.75	2500 3000	2 30 bloom to fruit growing	0 fruit 89 pulp 89 peel	0.86 <0.01 <0.01	
Orihuela, Spain 1995	0.75	3000	2 30 40% fruit size	0 fruit 21 fruit 35 pulp 35 peel 63 pulp 63 peel 90 pulp 90 peel	0.39 0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	

[1]In whole fruit unless otherwise stated

Pome fruits

Supervised field trials on apples and pears in various European countries were reported (Seym, 1993, 1996a,b; Anon., 1991). GAP for apples and pears in France is 0.020 kg ai/hl, 60-day PHI, and in Germany 1 x 0.026 kg ai/hl (0.40 kg ai/ha calculated for 1500 l water/ha), applied before or directly after flowering, with an estimated PHI of 60 days. GAP for pears in Portugal, applicable to Spain and Italy, is 0.025 kg ai/hl with a 56-day PHI. GAP for Belgium was not reported and was assumed to be that of Germany. The conditions and results of the field trials are shown in Table 21.

Table 21. Residues of ODM + ODM sulfone following application of ODM EC formulation to apples and pears in France, Belgium and Germany.

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment, (days), Growth stage at final application			
Apples						
Versuchsgut Hofchen (NE Cologne), Germany, 1995	0.25 0.38	1000 1500	2 41 20 mm fruit	90	<0.01	Method 0009/ M 0007. Recovery 65–89% at 0.01 and 0.1 mg/kg, n = 8. Samples stored 3 or 5 months frozen before analysis.
Versuchsgut Laacherhof (S Dusseldorf), Germany 1995	0.25 0.38	1000 1500	2 49 20 mm fruit	90	<0.01	
St. Etienne du Gres, France 1995	0.25 0.38	1000 1500	2 25 2nd fruit fall	30 45 60 75 92	0.08 0.05 0.03 0.01 0.01	Method 00009/ M 007
Pernes les Fontaines, France 1995	0.25 0.38	1000 1500	2 26 2nd fruit fall	30 92	0.10 <0.01	Method 00009/ M 003

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment, (days), Growth stage at final application			
Melkwezer, Belgium 1991	0.25	300	1 end of flowering	94 108 122	<0.04 <0.04 <0.04	Method 0009/ M 003
Ezemaal, Belgium 1991	0.25	300	1 after flowering	100–128	<0.01	Method 00009/ M 003
St. Paterne Racan, France, 1991	0.25	1000	1 50 mm fruit	29, 58	<0.04	Method 00009/ M 003
Hofchen, Germany 1990	0.38	1500	1 5 cm fruit	0 14 21 29 29 sauce 29 juice	0.26 0.15 0.12 0.09 <0.05 0.08	Method 00009/ M 003
Laacherhof, Germany 1990	0.38	1500	1 advanced ripening	0 14 20 28 35 28 sauce 28 juice	0.33 0.43 0.41 0.39 0.26 0.13 0.38	Method 00009/ M 003
Pears						
Versuchsgut Hofchen (NE Cologne), Germany 1995	0.25 0.38	1000 1500	2 56 20 mm fruit	44 77 105	<0.01 <0.01 <0.01	Method 00009/ M 0007. Recovery 59-115% at 0.01 and 0.1 mg/kg, n =14 (mean 84% ± 19%).
Versuchsgut Laacherhof (S Dusseldorf) , Germany 1995	0.25 0.38	1000 1500	2 58 2nd fruit fall	44 120	<0.01 <0.01	
Pogio Renatico, Italy 1995	0.25 0.38	1000 1500	2 4 10-20 mm fruit	45 60 75 90 115	<0.01 <0.01 <0.01 <0.01 <0.01	Method 00009/ M 007. Recovery 63-115% at 0.01 and 0.1 mg/kg, n = 14.
Dugliolo, Italy 1995	0.25 0.38	1000	2 8	45, 86	<0.01	
Ezemaal, Belgium 1991	0.25	300	1 end of flowering	109–137	<0.04	Method 00009/ M 003. Samples stored frozen up to 6 months before analysis
Rivarennes, France 1991	0.25	1000	1 fruit development	0 14-52	0.21 <0.04	Method 00009/ M 003. Samples stored frozen for 5 months before analysis.

Stone fruits

Supervised field trials on plums in Germany were reported (Seym, 1996). GAP for stone fruit in Germany is 1 x 0.026 kg ai/ha (0.38 kg ai/ha calculated), with application before or directly after flowering. This gives a PHI of about 60 days. The results of the trials are shown in Table 22.

Table 22. Residues of ODM + ODM sulfone following application of ODM EC formulation to plums in Germany, 1995.

Location	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment, (days), Growth stage at final application			
Plums						
Hofchen Burscheid, Germany	0.38	1500	1 fruit fall after flowering	61 107	0.040 0.011	Method 00009/ M 007. Recovery 66-114% at 0.01 and 0.1 mg/kg, n = 9. Samples stored frozen for 7 months before analysis.
Laacherhof Monheim, Germany	0.38	1500	1 sepals falling	60 118	0.014 <0.01	
Obstbaubetrieb Tackheide Tonisvorst, Germany	0.38	1500	1 sepals falling	60 120	0.044 <0.01	
Obsthof Schick Freinsheim, Germany	0.38	1500	1 sepals falling	60 120	0.028 <0.01	

Berries and other small fruits

Supervised field trials on grapes in Germany and Italy were reported (Seym, 1993a,b). GAP for use on grapes in Germany is 1 x 0.025 kg ai/hl, applied for 2 leaves to inflorescence development, PHI not specified, and in Italy 1 x 0.23 kg ai/ha, 40-day PHI. The results of the trials are shown in Table 23. The report was a summary only and did not include details of sampling, sample storage or analysis.

Table 23. Residues of ODM + ODM sulfone following application of ODM EC formulation to grapes in Germany and Italy.

Location/ Year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Grapes						
Bickensohl, Germany 1991	0.2	800	1	115	<0.04	Method 00009/ M 003. Samples stored about 4 months before analysis.
Albig, Germany 1991	0.2	800	1	94	<0.04	
Diesesfeld, Germany 1991	0.2	800	1	109	<0.04	
Ravenna, Italy 1991	0.28	1000	1 fruit setting 70%	0 40 60 40 must 40 wine	0.37 <0.04 <0.04 <0.04 <0.04	Method 00009/ M 003. Two trials were on different varieties. Samples stored at –20°C for about 4-6 months before analysis.
Ravenna, Italy 1991	0.28	1000	1 fruit setting 30%	0 40 60 40 must 40 wine	0.30 0.06 <0.04 <0.04 <0.04	

Brassica vegetables

Supervised field trials were carried out in Germany on cabbage (Anon., 1973-1986; Schmidt, 1992), curly kale (Seym and Heinemann, 1996) and kohlrabi (Anon., 1974-1980). The results are given in Table 24. The reports of the cabbage and kohlrabi trials were summaries only. For example details of the sampling, sample storage and analysis including validation and chromatograms were not provided. The report of the curly kale trials was complete. Although 21 trials on cabbages were reported, 5 of the 9 trials in 1976 were apparently replicates of others, so only 4 results from that year were evaluated separately. GAP for cabbage, kale and kohlrabi in Germany is 1 x 0.15 kg ai/ha at a plant height under 50 cm and a PHI of 21 days.

Table 24. Residues of ODM + ODM sulfone in or on brassica vegetables after foliar application of ODM EC formulation. Germany.

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Cabbage						
Monheim Laacherhof, 1976	0.15	600	2	0 7-14	0.08 <0.01	Method 00009
Monheim, Laacherhof, 1976	0.15	600	2	0 7-14	0.21 <0.01	Method 00009
Monheim, Laacherhof, 1976	0.15	600	2	0 7 14	0.21 0.01 <0.01	Method 00009
Monheim Laacherhof, 1979	0.15	600	2 flower diameter 10 cm	0–28	<0.03	Method 00009
Klein-Niedesheim, 1979	0.15	600	2 start of head development	0–28	<0.05	Method 00009
Worms-Heppenheim, 1986	0.15	600	2 start of head development	0 3 7	0.4 0.18 <0.01	Method 00009
Monheim, Laacherhof, 1986	0.15	600	2 start of flower development	0 3 7 14	0.15 0.06 0.04 <0.01	Method 00009
Monheim, Laacherhof, 1976	0.12	600	2	0–21	<0.06	Method 00009
Monheim, Laacherhof, 1976	0.12	600	2	0–21	<0.06	Method 00009
Monheim, Laacherhof, 1976	0.12	600	2	0–21	<0.06	Method 00009
Burscheid, 1978	0.18	600	2 head formation	0 3 7 14 21 28	0.75 0.27 0.31 0.25 0.02 0.02	Method 00009
Klein-niedersheim, 1978	0.18	600	2 head formation	0 4–28	1.2 <0.05	Method 00009
Monheim, Laacherhof, 1973	0.12	600	2	22	<0.01	Method 00009. Trials with 3 varieties.
Klein Niedesheim, 1986	0.12	600	3 start of head	0 3-21	1 <0.1	Method 00009

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
			development			
Monheim Laacherhof, 1986	0.12	600	3 flower formation	0 3-21	0.1 <0.1	Method 00009
Monheim, Laacherhof, 1979	0.15	600	2 start of head development	0 7-28	0.7 <0.04	Method 00009
Burscheid, 1990	0.15	300	1 33% final head size	0 7 14 14 cooked head 14 cooking water	0.70 0.13 <0.05 <0.05 <0.05	Method 00009
Monheim, Laacherhof, 1990	0.15	600	1 66% final head size	0 7 14 14 cooked head 14 cooking water	0.40 <0.05 <0.05 <0.05 <0.05	<Method 00009
Leichlingen, 1976	0.12	600	2	0 4 7 14-21	0.31 0.05 0.11 <0.03	Method 00009
Langenfeld-Reusrath, 1976	0.12	600	2	0 4 14-21	0.72 0.11 <0.03	Method 00009
Leichlingen, 1976	0.12	600	2	0 4-21	0.06 <0.03	Method 00009
Kale						
Klein- Niedesheim, Germany 1994	0.15	600	1 plant development	0 5 6 14-28	2.7 0.28 0.13 <0.01	Method 00009. Samples stored 6.5 months before analysis. Recovery 74-86%, mean 80%, fortified at 0.01 and 0.1 mg/kg n = 5, for all kale trials
Worms-Heppenheim, 1994	0.15	600	1 plant development	0 5 7 14-28	2.6 0.11 0.05 <0.01	Method 00009. Samples stored 7 mos. before analysis. See first kale entry for recovery.
Burscheid Hofchen, 1994	0.15	600	1 plant development	0 plant (aerial) 5 7 14 21 leaf 21 leaf cooked 21 leaf cooking water	1.2 0.17 0.12 0.02 <0.01 <0.01 <0.01	Method 00009. Samples stored 7 months before analysis
Monheim Laacherhof, 1994	0.15	600	1 plant development	0 plant (aerial) 4	3.2 0.89	Method 00009. Samples stored 8 months before

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
				7 14 21 leaf 21 leaf cooked 21 leaf cooking water	0.42 0.01 <0.01 <u>≤0.01</u> <0.01	analysis.
Kohlrabi						
Monheim, Laacherhof	0.15	600	2 walnut size	0 leaf 7 14 21 0–28 root	3.59 0.62 0.22 <0.01 <u>≤0.01</u>	Method 00009
Klein Niedesheim, 1979	0.15	600	2 50% final diameter	0 leaf 7–28 0–28 root	0.48 <0.06 <u>≤0.06</u>	Method 00009
Dicksander-koog, 1975	0.15	300	3	0 root 14-35 root	<0.01; 0.02; 0.02 <u>≤0.01</u>	Method RA133/74. Three trials
Monheim, Laacherhof, 1974	0.12	600	2 mature	0 7 14 21 28	0.06 0.05 0.05 <u>0.03</u> 0.01	Method I11 (unknown)

<u>Leafy Vegetables</u>

Supervised field trials were reported from Germany on lettuce (Anon., 1975a,b; Seym and Nusslein, 1995). Only the 1995 report was detailed, the 1975 reports being only summaries. GAP for the use of ODM on lettuce in Germany is 1 x 0.16 kg ai/ha of an EC formulation, PHI 21 days. The results are given in Table 25. There were only three independent trials, with triplicate plots at Monheim in 1975 and duplicates at Monheim and Burscheid in 1993.

Table 25. Residues of ODM + ODM sulfone in or on lettuce after foliar application of ODM EC formulation in Germany

Location, year	Application			PHI, days	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Monheim, Laacherhof 1975	0.12	600	2 before harvest	0 8 14 21-28	2.5 0.04 0.04 <0.01	Method I 47 modified
Monheim, Laacherhof 1975	0.12	600	2	0 8 14 21-28	4.91 0.21 0.05 <0.01 <0.01	Method I47 modified
Monheim, Laacherhof 1975	0.15	600	2	0 8 15 21 28	5.29,5.32,5.21 0.76,0.77,0.79 0.29,0.16,0.17 0.02,0.02,0.03 0.01,0.01,0.01	Method I 47 modified. Results represent trials with 3 varieties
Burscheid Hofchen 1993	0.15	600	1 80% final size	0 7 10 14	1.5 0.39 0.18 0.13	Method 00009, M 07. Samples stored frozen 5–6 months before analysis. Head were fortified at 0.02, 0.20 and 0.50 mg/kg with ODM. Recovery ranged from 71–96%, mean 88%.
Monheim Laacherhof 1993	0.15	600	1 80% final size	0 7 10 14	2.5 0.72 0.31 0.20	
Burscheid Hofchen 1993	0.15	600	1 80% final size	0 7 10 14	1.1 0.29 0.09 0.04	
Monheim Laacherhof 1993	0.15	600	1 harvest	0 7 10 14	0.55 0.09 0.09 <0.02	

Pulses

Supervised field trials were conducted in Germany and France on field peas (Anon., 1991; Seym, 1996)., and in Germany on field beans (Anon., 1975, 1979). The results are shown in Table 26. GAP for beans in Germany is 2 x 0.15 kg ai/ha (under 50 cm), 0.25 kg ai/ha (over 50 cm), with a 28-day PHI. GAP for peas was not reported for Germany or France for the 250 kg ai/l EC formulation. GAP for Italy is 1 x 0.23 kg ai/ha, 21-day PHI.

Table 26. Residues of ODM + ODM sulfone in or on dried field peas and field beans after foliar application of ODM EC formulation.

Location, year	Application			PHI, days, Sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Field peas						
Worms-Heppenheim, Germany 1995	0.1	600	2 pods 10% length	0 pea with pod 28 seed, dry 28 straw	0.22 <0.01 <0.02	Method 00009, M 007. ODM recoveries at 0.01 and 0.1 mg/kg, 67, 71, 89, 76, 79% from pod with seed, 69, 77, 87, 76, 78% from seed. ODM recoveries at 0.01, 0.02, 0.1 mg/kg from straw 67, 75, 85, 70, 82%
Burscheid Hofchen, Germany 1995	0.1	600	2 pods typical size	0 pea with pod 13 seed, dry 13 straw 28 straw	<0.01 <0.01 <0.02 0.15	
Monheim Laacherhof, Germany 1995	0.1	600	2 10% pods ripe	0 pea with pod 28 seeds, dry 28 straw	0.19 <0.01 <0.02	
Prey, France 1991	0.1	280	2 10 pod development	0 hull, green 11 hull, green 23 hull, dry 23 seed	0.33 0.02 <0.04 <0.01	Method 00255. Summary translation only.
Les Essarts, Bretagnolles, France 1991	0.1	280	2 10 pod development	0 hull, green 11 hull, green 23 hull, dry 23 seed	0.19 <0.01 <0.04 <0.01	Method 00255. ODM recoveries 90% at 0.01 mg/kg. Summary translation only.
Field beans						
Monheim, Laacherhof, Germany 1975	0.15	600	3 19 26	0 bean 7 14 21 28 0 pod 7 14 21 28 0 straw 7 14-28	<0.01 0.02 0.05 <0.01 <0.01 0.3 0.04 0.02 0.0-4 <0.01 2.9 0.1 <0.01	Method I47 modified. Samples analysed within 1 month
Monheim, Laacherhof, Germany 1975	0.15	600	3 19 26	0 bean 7 14 21 28 0 pod 7 14 21 28 0 straw 7 14 21 28	0.05 0.02 <0.01 <0.01 <0.01 0.42 0.08 0.04 0.04 <0.01 13. 0.35 0.06 <0.01 <0.01	Method I 47 modified. Samples stored up to 2 months before analysis.

Location, year	Application			PHI, days, Sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Monheim, Laacherhof, Germany 1975	0.15	600	3 19 26	0 bean 7 14 21 28 0 pod 7-28 0 straw 76 14 21 28	0.05 0.05 0.05 0.05 0.05 0.04 <0.01 1.8 0.1 0.04 0.04 0.04	Method 00009. Samples stored up to 2 months before analysis.
Monheim Laacherhof, Germany 1979	0.22	900	3 14 14	0-28 bean 0 pod 7-28 pod 0 straw 7 straw 14-28 straw	0.05 0.04 <0.01 1.8 0.1 0.04	Method 00009. Samples stored 6 months before analysis.
Klein niedesheim, Germany 1979	0.22	900	3 14 14 start of pod development	0 bean 7 14 21 28 0 pod 7-28 0 straw 7 14 21 28	<0.02 <0.02 <0.04 <0.04 <0.04 0.06 <0.04 2.1 0.76 0.24 0.04 <0.03	Method 00009. Samples stored 8 months before analysis.

Root and tuber vegetables

The Netherlands provided summary information on a field trial with potatoes in 1971 (Olthof and Meyer, 1973), and Bayer AG reported supervised field trials in Germany, France and the UK (Anon., 1977; Seym, 1993a,b, 1996; Heinemann and Seym, 1998; Schmidt, 1992).

Bayer AG also reported supervised field trials on sugar beet (Anon., 1973, 1991; Ohs, 1993; Schmidt, 1989, 1992).

GAP for the use of ODM on potatoes in Germany and Austria is 3 x 0.3 kg ai/ha with a 21-day PHI (28-day pending) and 3 x 0.25 kg ai/ha with a 42-day PHI respectively. No information was supplied for the UK or France. GAP for Ireland is 0.15 kg ai/ha (under 50 cm), 0.22 kg ai/ha (over 50 cm), 200 l water/ha, 28-day PHI. GAP for sugar beet (fodder beet) in Germany and France is 4 x 0.24 kg ai/ha and 1 x 0.15 kg ai/ha respectively, with a 28-day PHI. No information was supplied for the UK or The Netherlands.

The results are shown in Table 27. Two of the German trials in 1993 were replicates of others and therefore not included in the evaluation.

Table 27. Residues of ODM + ODM sulfone in or on root and tuber vegetables after foliar application of ODM EC formulation.

Location/Year	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application	PHI, day, Sample	ODM + ODM sulfone, mg/kg	Comments
Potato						
Klein Niedersheim, Germany 1993	0.33 0.28 0.22	600	3 10 10 tubers 30%	0–63	≤0.02	Method 00009, M 003. Samples stored 6-8 months before analysis. ODM recoveries at 0.02 mg/kg 49-93%, n=11; at 0.2 mg/kg 44-64%, n=7; at 0.5 mg/kg 67, 68%. Example chromatograms provided.
Burscheid Hofchen, Germany 1993	0.33 0.28 0.22	600	3 13 37 tubers 70%	0–63	≤0.02	
Worms Heppenheim, Germany 1993	0.33 0.28 0.22	600	3 10 11 tubers 30%	0–63	≤0.02	
Monheim Laacherhof, Germany 1993	0.33 0.28 0.22	600	3 12 15 tubers 70%	0–63	≤0.02	
Burscheid Hofchen, Germany 1993	0.33 0.28 0.22	600	3 13 37 tubers 70%	0–63	<0.02	
Monheim Laacherhof, Germany 1993	0.33 0.28 0.22	600	3 12 15 tubers 70%	0–63	<0.02	
Haltern Hullern, Germany 1991	0.3	300	2 6 pre-ripening	0–42	≤0.02	Method 00009, M 003. Samples stored frozen for up to 8 months before analysis.
Burscheid, Germany 1991	0.3	300	2	0–42	≤0.02	Method 00009, M 003
Burscheid, Germany 1990	0.3 0.25 0.2	300	3 15 68	0 foliage 14–21 tuber 14 cooked tuber 14 peel 14 cooking water	3.8 ≤0.05 <0.05 <0.05 <0.05	Method 00009, M 003. Summary translation.
Monheim Laacherhof, Germany 1990	0.3	300	3	0 foliage 14-21 tuber 14 peel 14 cooked tuber 14 cooking water	11 ≤0.05 <0.05 <0.05 <0.05	Method 00009, M 003
Monheim Laacherhof, Germany 1977	0.3 0.24 0.2	600	3 12 14	6-36 tuber	≤0.1	Method 00009. Limited summary only.
Dohnson, Germany 1977	0.3 0.24 0.2	400	3 10 11-14	30-76 tuber	≤0.1	Method 00009. Limited summary only.

Location/Year	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application	PHI, day, Sample	ODM + ODM sulfone, mg/kg	Comments
Elm Farm Development Station, UK 1991	0.24	200	2 15 flowering	0–20	<0.02	Method 00009, M 003. Samples stored frozen for 6 months before analysis.
Deal, Kent, UK 1991	0.26 0.24	200	2 13 late flowering	0–21	<0.02	
Burscheid Hofchen, Germany 1996	0.25	300	3 8 8 tuber 70%	0-28	<0.02	Method 00255. Samples stored frozen 7-10 months before analysis. Recovery of sulfone at 0.1 mg/kg 77%.
Monheim Laacherhof, Germany 1996	0.25	300	3 8 8 tuber 100%	0-27 tuber	<0.01	
Thurston, UK 1996	0.25	400	3 9 7 tuber 60%	0-28 tuber	<0.01	
Criquebeuf-sur-Seine, France 1996	0.25	400	3 8 8 tuber 30%	0-28 tuber	<0.01	
Udenhout, The Netherlands 1971	0.25	500	1	35 tuber	<0.01	Similar to Method 00009
Blaaksedijk, The Netherlands 1971	0.25	500	1	31 tuber	<0.01	Similar to Method 00009
Sugar beet						
Sibiville, France 1991	0.38	280	2 15 before maturity	0 leaf 29 0-61 root	1.90 <0.04 <0.04	Method 00009, M 003. Samples stored 6 months before analysis.
Domart en Ponthieu, France 1991	0.38	280	2 15 before maturity	0 leaf 29-61 0-61 root	0.80 <0.04 <0.04	Method 00009, M 003. Samples stored 6 months before analysis.
Monheim Laacherhof, Germany 1973	0.2	600	2 25	21 root 21 leaf	<0.01, <0.01, <0.01 <0.01, 0.01, 0.03. avg 0.02	Method I47. 3 variants. Information incomplete.
Monheim Laacherhof, Germany 1991	0.2	300	4 12 7 9 root 80%	0-35 root 0 leaf 7 leaf 14 leaf 28-35 leaf	<0.01 3.0 0.38 0.08 <0.04	Method 00255 See fodder beet Germany 1991 for recoveries.
Albig, Germany 1991	0.2	300	4 9 10 14 root 80%	0-35 root 0 leaf 7 leaf 14 leaf 28-35 leaf	<0.01 5.7 1.7 0.54 <0.04	

Location/Year	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application	PHI, day, Sample	ODM + ODM sulfone, mg/kg	Comments
Burscheid Hofchen, Germany 1990	0.19	300	4 13 14 18 4-6 wk after crop covering (51)	0-35 root 0 leaf 7 leaf 14 leaf 28-35 leaf	<0.04 1.9 0.66 0.09 <0.04	Method 00255, M 009. About 9 months frozen storage before analysis. Concurrent recoveries 94, 88%, at 0.04 mg/kg demeton-S-methylsulphon
Monheim Laacherhof, Germany 1990	0.19	300	4 13 14 18 near maturity	0-35 root 0 leaf 7 leaf 14-35 leaf	<0.04 3.2 0.31 <0.04	
Klein Niedesheim, Germany 1990	0.19	300	4 13 14 18 harvest size	0-35 root 0 leaf 7 leaf 14 leaf 28-35 leaf	<0.04 2.9 0.07 0.05 <0.04	
Worms Hepppen-heim , Germany 1990	0.19	300	4 13 14 18 harvest size	0-35 root 0 leaf 7 leaf 14-35 leaf	<0.04 6.3 0.05 <0.04	
Burscheid, Germany 1988	4.2	600	3 14 15 6-8 week after crop cover	0 root 30 root 0 leaf 30 leaf 30 chips 30 mud 30 thin juice 30 thick juice 30 molasses 30 white sugar 30 refined sugar	0.40 <0.04 9.4 0.05 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04	Method 00009, M 002. Chromatograms included. Processing described. ODM recoveries at 0.04 and 1.0 mg/kg 60-105% from root, 78-105% from leaves, 75-102% from pulp, 92-99% from juice, 87-99% from molasses, 93-94% from sugar.
Burscheid, Germany 1988	0.83	600	3 14 15 6-8 weeks after crop cover	0 root 30 root 0 leaf 30 leaf	0.06 <0.04 3.7 <0.04	
Fodder beet (animal feed)						
Monheim, Germany 1991	0.19	300	4 12 7 9 root 80%	0-35 root 0 leaf 7 leaf 14 leaf 28-35 leaf	<0.01 2.9 0.72 0.13 <0.04	Method 00255. ODM recovery from leaf 92, 93% at 0.04 mg/kg; from root 92, 90, 96% at 0.01 mg/kg.
Kothel, Germany 1991	0.19	300	4 10 11 11 harvest (100%)	0-35 root 0 leaf 7 leaf 14-35 leaf	<0.04 4.1 0.69 <0.04	Method 00255. Root residues corrected for mean recovery of 66%.

Cereal grains, fodders and straws

Supervised trials were reported for the use of ODM on wheat, oats and barley in Europe (Schmidt, 1992a,b; Seym, 1993a,b). GAP for wheat and barley is 1 x 0.14 kg ai/ha, 21-day PHI, in Germany and 1 x 0.12 kg ai/ha, 21-day PHI in Austria. GAP for cereals in France is 3 x 0.1 kg ai/ha, PHI 21 days or not

specified. No GAP was reported for the UK, but GAP for Ireland is 0.12 kg ai/ha, 21-day PHI. The field trial conditions and results are shown in Table 28.

Table 28. Residues of ODM + ODM sulfone in or on cereals after foliar application of ODM EC formulation.

Location/Year	Application			PHI, days, Sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Wheat						
Borstorf, Germany 1990	0.12	300	1 start of ripening (80)	0 forage 7 14 29 grain 35 29 straw 35 29 wholemeal flour 29 white flour 29 wholemeal bread 29 white bread	2.3 0.36 0.17 <0.05 <0.05 <0.10 <0.10 <0.05 <0.05 <0.05 <0.05	Method 00009, M 003. Samples stored at –20ºC for 8–9 months before analysis.
Neuberg/ Rudigheim, Germany 1990	0.12	300	1 watery ripe (71)	0 forage 7 14 ear 14 straw 21 35 21 grain 35 21 wholemeal flour 21 white flour 21 wholemeal bread 21 white bread	2.4 2.2 1.1 1.2 1.2 0.75 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	
Monheim, Germany 1991	0.12	300	2 54 dough stage	0 forage 7–28 grain 7 straw 14–28 straw	3.6 <0.04 0.14 <0.04	Method 00255. Method 00015 for straw. ODM recovery at 0.04 and 2.0 mg/kg for forage, 64-101%, n=6; for grain 104-118%, n=4; at 0.04, 0.4, 2.0 mg/kg for straw, 74-94%, n=4.
Worms Heppenheim, Germany 1991	0.12	300	2 19 end of flowering	0 forage 7 14 21–28 grain 21 straw 28	2.6 0.37 0.17 <0.04 0.12 0.14	
Monheim Laacherhof, Germany 1990	0.12	300	2 14 end of heading	0 forage 5 14 21-51 straw 51 grain 21 ear	0.19 0.10 <0.05 <0.05 <0.05 <0.05	Method 00015, M 009.
Worms Heppenheim, Germany 1990	0.12	300	2 47 grain development	0 forage 7 14 21-28 grain 21 straw 28 straw	3.2 0.99 1.9 <0.05 1.3 0.82	Method 00015, M 009. Samples stored frozen for 8 months before analysis.

Location/Year	Application			PHI, days, Sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Datteln, Germany 1991	0.12	300	1 grain development	0 forage 7 14 21 ear 21 straw 28 28 grain	1.8 0.69 0.27 0.09 <u>0.18</u> 0.06 <u><0.04</u>	Method 00009, M 003. Samples stored frozen for 6 months before analysis.
Burscheid, Germany 1991	0.12	300	1 milky ripe	0 forage 7 15 21 straw 29 21 ear 29 grain	2.2 0.25 0.05 <u><0.04</u> <0.04 <0.04 <0.04	Method 00009, M 003. Samples stored frozen for 6 months before analysis.
Elm Farm Development Station, UK 1991	0.12	200	1 soft dough	0 forage 14 28 grain 28 straw	1.4 0.06 <u><0.04</u> <u><0.04</u>	Method 00009, M 003
Barley						
Burscheid, Germany 1990	0.12	300	2 18 end of heading	0 forage 7–14 21 ear 21 straw 45 straw 45 grain	3.6 <0.04 <0.04 <0.04 0.05 <0.04	Method 00015, M 009. Samples stored frozen for 10 months before analysis.
Burscheid, Germany 1991	0.12	300	2 42 milky ripe	0 forage 7 15 21–28 grain 21–28 straw	2.4 0.09 <0.04 <u><0.04</u> <u><0.04</u>	Method 00255. ODM recoveries at 0.04 and 0.4 mg/kg, n=4, 83-97% for forage; 99, 76, 88% for grain; 111, 74, 79% for straw.
Datteln, Germany 1991	0.12	300	1 grain development (caryopsis watery)	0 forage 7 14 21 ear 21 straw 28 28 grain	0.98 0.18 <0.04 <0.04 <u>0.06</u> 0.05 <u><0.04</u>	Method 00009, M 003. Samples stored frozen for 8 months before analysis.
Elm Farm Development Station, UK 1991	0.13	212	1 start of ripening	0 forage 14 27 grain 27 straw	1.1 0.06 <u><0.05</u> <u><0.05</u>	Method 00009, M 003
Oats						
Heppenheim, Germany 1990	0.12	300	2 44 caryopsis watery	0 forage 7 14 21 grain 28 21 straw 28	2.8 0.30 0.14 0.05 <0.04 0.11 0.04	Method 00009, M 003

Location/Year	Application			PHI, days, Sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Worms-Heppenheim, Germany 1991	0.12	300	2 21 end of flowering	0 forage 7 14 21 grain 28 21 straw 28	0.14 0.10 0.10 <0.01 <0.01 0.14 0.21	Method 00255. ODM recoveries at 0.04 and 0.4 mg/kg 94, 102, 102% in forage; 87, 98, 79% in grain; 102, 99, 77% in straw

Tree nuts

Supervised field trials were reported on almonds in Spain, where GAP is 2 x 0.25 kg ai/ha with a 30-day PHI (Seym, 1996; Seym and Nusslein, 1995). The results are shown in Table 29.

Table 29. Residues of ODM + ODM sulfone in or on almonds after foliar application of ODM EC formulation in Spain

Location, year	Application			PHI, days, sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Jorba-Sant Genis 1995 Variety Ferraduel.	0.25	800	2 31 7-8 cm fruit	27 fruit 42 61 nut with shell 75 91 91 kernel	0.35 0.19 <0.01 <0.01 <0.01 <0.01	Method 00009, M 007. Samples stored frozen about 5 months before analysis.
Jorba-Sant Genis 1995 Variety Ferragnes.	0.25	800	2 29 7-8 cm fruit[1]	29 fruit 44 63 nut with shell 77 93 93 kernel	0.28 0.18 0.02 0.02 0.01 <0.01	Method 00009, M 007. Samples stored frozen about 5 months before analysis.
Les Gunyoles, Spain 1995	0.25	800	2 31 8-9 cm fruit	33 fruit 46 55 77 nut with shell 82 82 kernel	1.1 0.85 0.58 0.05 <0.01 <0.01	Method 00009, M 007. Samples stored frozen about 5 months before analysis
Eis Garidells, Spain 1995	0.25	800	2 31 8-9 cm fruit	33 fruit 46 55 77 nut with shell 82 82 kernel	0.55 0.31 0.12 0.04 0.01 <0.01	Method 00009, M 007. Samples stored frozen about 5 months before analysis. Recoveries of ODM at 0.01 and 0.1 mg/kg from kernels 72-86%, n = 5; from fruit 64-89%, n=7.

Location, year	Application			PHI, days, sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Les Gunyoles, Spain 1993	0.18	710	2 28 70% final size (77)	90 nut	<0.02	Method 00009, M 007 ODM recoveries from nut fortified at 0.02 and 0.2 mg/kg, 70-99%, n=5.
Castellri de la Marca, Spain 1993	0.19	710	2 32 70% final size (77)	87 nut	<0.02	

[1]Immature nut with shell

Oilseed

Bayer AG reported supervised field trial for rape (Anon., 1988, 1989, 1990, 1991, 1996a,b,c,d; DeMonte, 1996, Hanlon, 1996; Leslie, 1989; Seym and Nusslein, 1995).

GAP in France for the use of ODM on rape (oil seed) is 1 x 0.12 kg ai/ha, PHI not specified, and on sunflower 1X 0.1 kg ai/ha, PHI not specified. GAP for ODM on cotton in Brazil and Australia was not reported, but GAP for Peru is 3 x 0.25 kg ai/ha and for the USA is 3 x 0.84 kg ai/ha, both with PHIs of 14 days.

Table 30. Residues of ODM + ODM sulfone in or on oilseed crops after foliar application of ODM EC formulation.

Location, year	Application			PHI, days, sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Rape						
St. Valery en Caux, France 1991	0.12	280	1 pod development	0 forage 79 seed 79 oil 79 pomace	1.5 <0.05 <0.05 <0.05	Method 00009, M 003. Samples stored 11-13 months before analysis.
Cany Barville, France 1991	0.12	280	1 pod development	0 forage 79 seed 79 oil 79 pomace	1.1 <0.05 <0.05 <0.05	Method 00009, M 003. Samples analysed within 2 months
Jouaville, France 1991	0.12	300	1	0 forage 63 seed 63 oil 63 pomace	1.4 <0.05 <0.05 <0.05	Method 00009, M 003. Samples stored 5-12 months before analysis.
Sunflower						
Laudun, France 1993	0.12	280	1 inflorescence	0 forage 29 head 83 seed	2.9; 2.4 <0.04; <0.04 <0.01; <0.01	Method 00255. Duplicate plots. ODM recoveries at 0.04-0.4 mg/kg 67-97% from forage (n=6), 74-82% (n=4) from heads; at 0.01 and 0.1 mg/kg 72-108% (n=4) from seed.

Location, year	Application			PHI, days, sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Drache, France 1989	0.1	300	1 6 leaf stage	87 seed	≤0.01	Method 00086, M 012. Limited summary translation.
Maille, France 1989	0.1	300	1 10 leaf stage	87 seed	≤0.01	Method 00086, M 012. Sample stored 9 months before analysis.
Cotton						
Narrabri, NSW, Australia 1993	0.075	110	1	0-21 seed	<0.01	Method 00246 ODM recoveries (not concurrent) from seed at 0.01-0.11 mg/kg 64-89% (n=4). Sulfone recovery from seed at 0.01-0.05 mg/kg 77, 85, 86%.
Narrabri, NSW, Australia 1993	0.15	110	1	0-21 seed	<0.01	
Narrabri, NSW, Australia 1995	0.075	140	1 late boll fill	0-21 seed	<0.01	Method 00246. Duplicate plots. Average recoveries for ODM (0.01-0.06 mg/kg) 82%.
Narrabri, NSW, Australia 1995	0.15	140	1 late boll fill	0-21 seed	<0.01	
Boggabilla via Goondiwindi, NSW, Australia 1996	0.075	110	2 12 6 days before defoliation	7-21 seed 7 trash 14 trash 21 trash	<0.01 0.68 0.07 0.12	Method 00246 ODM recoveries at 0.01-0.02 mg/kg from seed 74-85%, n=3; at 0.14 and 2.8 mg/kg from trash, 93 and 109%. Sulfone recoveries at 0.02 mg/kg from seed, 85%; at 0.10 and 0.60 mg/kg from trash 104 and 100%.
Boggabilla via Goondiwindi, NSW, Australia 1996	0.15	110	2 12 6 days before defoliation	7-21 seed 7 trash 14 trash 21 trash	<0.01 3.5 0.45 0.33	
Tipton via Dalby, QL, Australia 1996	0.075	77	2 9 15 days before defoliation	7-20 seed 7 trash 14 trash 20 trash	<0.01 1.5 1.6 0.86	Method 00246. Recoveries as Boggabilla, 1996.
Tipton via Dalby, QL, Australia 1996	0.15	77	2 9 15 days before defoliation	7-20 seed 7 trash 14 trash 20 trash	<0.01 4.4 2.2 0.62	
Bellata, NSW, Australia 1994	0.075	110	1 late boll fill	0-21 seed	<0.01	Method 00246. Recoveries as Narrabri 1993.
Bellata, NSW, Australia 1994	0.15	110	1 late boll fill	0-21 seed	<0.01	
Rolandia, Brazil 1988	0.2	300	4 18, 13, 14 bolls opening	0 boll 4 seed 7 seed 10 seed 14 seed	2.6 0.05 0.04 0.04 0.03	Method 00009. Samples stored 5-6 months before analysis.
Rolandia, Brazil 1988	0.4	300	4 18 13 14 bolls opening	14 seed	0.05	Method 00009. Sample stored 5 months before analysis.

Location, year	Application			PHI, days, sample	ODM + ODM sulfone, mg/kg	Comments
	Rate, kg ai/ha	Spray, l/ha	No., Retreatment (days), Growth stage at final application			
Municipio de Terenos, Brazil 1989	0.2	300	4 22 15 24 maturity	0 boll 4 seed 7 seed 11 seed 15 seed	1.8 0.04 0.04 0.03 0.02	Method 00009. Samples stored 9 months before analysis.
Municipio de Terenos, Brazil 1989	0.4	300	4 22 13 24 maturity	15 seed	0.03	Method 00009. Samples stored 9 months before analysis.
Lubbock, Texas, US 1988	0.56	190	2 open bolls	7 seed 14 21	0.03 0.01 0.02	Method 00246
Yuma, Arizona, US 1988	0.56	190	2 mature	7 seed 14	0.04 0.01	Method 00246
Calipatria, California, USA 1988	0.56	58	2 open bolls	7 seed 14	0.03 0.01	Method 00246
Benoit, Mississippi, USA 1988	0.56	61	2 mature	0 seed 4 seed 14-21 seed	0.07 0.02 <0.01	Method 00246
Benoit, Mississippi, USA 1988	0.56	47	2 mature	0 seed 4 14 21	<0.01 0.02 0.02 <0.01	Method 00246
Adams Gardens, Texas, US 1988	0.56	200	2 open bolls	0 seed 7-21 seed	0.38 <0.01	Method 00246
Adams Gardens, Texas, US 1988	2.8	200	2 open bolls	14 seed 14 hull 14 meal 14 oil 14 soapstock	0.11 <0.02 0.06 <0.02 <0.02	Method 00246

Animal feeding studies

Residues of ODM and ODM sulfone were determined in the milk and tissues of lactating dairy cows fed a diet containing ODM (Smyser and Halpin, 1987). Nine cows were divided into three groups of three each and a tenth cow was used as a control. The treated animals were fed ODM by bolus for 28 consecutive days at levels equivalent to 10, 30 or 100 ppm in the diet, or 0.3, 0.9 or 3.0 mg/kg bw. The high-dose group was removed from the study because of organophosphorus poisoning. The feed concentrations are only approximate as the actual feed consumption was not measured; it was assumed to be 3% of the body weight, or about 15 kg feed, per day. Milk was collected twice daily and the samples within each group were mixed. A 500 ml sample from each group was frozen. Within 14 hours of the last dose the cows were slaughtered and appropriate tissues were homogenized and stored frozen. The combined residue of ODM and ODM sulfone in the milk at all intervals and in liver, kidney, muscle and fat was <0.01 mg/kg. Details of the study were not provided, e.g. no analytical method was described or referenced.

A feeding study was reported on poultry (Lee and Wood, 1987). Thirty laying hens were divided into groups of 10 and fed rations containing 0.65, 1.95, or 6.50 ppm ODM for 28 days. Ten birds were

maintained as a control. Water and feed were provided *ad lib* throughout the study. The actual feed consumption was not determined. Eggs were collected each morning, stored in a refrigerator, and pooled within groups by day for analysis. The hens were asphyxiated at the end of the 28 days and liver, kidney, heart, gizzard, composite thigh and breast muscle and composite fat were taken from each hen. The samples were stored frozen, and analysed within five weeks of collection for ODM plus ODM sulfone by a method equivalent to Method 00246. The combined residue was reported as <0.01 mg/kg in all samples. Only tissues from the 6.50 ppm feed group were analysed. The recoveries from control samples fortified at 0.1 mg/kg with ODM sulfone were muscle 68%, liver 68%, gizzard 106%, skin 88%, fat 80%, heart 106%, kidney 98%, eggs 82, 85, 79, 93, 80 and 80%, and chicken feed 87, 104, 96 and 106%.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information.

In processing

In a processing study on apples in Germany in 1990 (Schmidt, 1992) trees (Cox's Orange) were sprayed twice at 0.38 kg ai/ha, with about 60 days between applications. The apples were harvested 29 days after treatment and processed into juice and sauce. Details of the process were provided in German. Samples were analysed by Method 00009/ M 003. The fruit contained 0.086–0.097 mg/kg, the sauce <0.05 mg/kg, and the juice 0.071–0.091 mg/kg sulfone equivalents. In a separate trial, Delicious apple trees were sprayed twice at 0.38 kg ai/ha with an ODM EC formulation, with about 5 months between treatments. Apples were harvested 28 days after the final treatment and processed into juice and sauce. Residues on the apples were 0.335 to 0.438 mg/kg, in the juice 0.315 to 0.447 mg/kg and in the sauce 0.124 to 0.139 mg/kg. The processing factors from the two trials were about 1 for juice and 0.5 for sauce.

In a cotton seed processing study in Texas in 1988 (Leslie, 1989) cotton was treated twice at 2.8 kg ai/ha (190 l/ha) or about 3 times the US GAP rate (3 x 0.84 kg ai/ha, 14-day PHI). The cotton bolls were harvested 14 days after the second application and immediately ginned. The ginned seed was treated by a simulated industrial process within 4 months. The seed was hulled and the kernels extracted with solvent. The process was not described in detail. The raw and processed commodities were analysed by Method 00246. Several modifications were made to the method, including the use of a 13 m x 0.32 mm i.d. capillary column (DB-5) and the addition of a silica gel column clean-up after chloroform extraction of the oxidized mixture. The longest frozen storage period before analysis was 290 days. The results are shown in Table 31. Acceptable recoveries were claimed for control analyses at 0.02 mg/kg, but no details were provided.

Table 31. Effects of processing cotton seed on ODM residues.

Sample	Residue, mg/kg	Processing factor	Control analyses	
			Fortification, mg/kg	Recovery, %
Delinted cotton seed	0.11		0.05 0.50	100 114
Hulls	<0.02	0.2	-	-
Meal	0.06	0.6	0.10	81; 80
Crude oil	<0.02	0.2	0.10	100
Refined oil	<0.02	0.2	0.10	107
Soapstock	<0.02	0.2	0.10	95

Limited information on processing was included with reports of supervised field trials on sugar beet in Germany, grapes in Italy (must and wine), curly kale in Germany (cooked leaves and cooking water), potatoes in Germany (peel, cooked potatoes, cooking water), rape in France (pomace, crude oil, refined oil) and wheat in Germany (flour and bread). In all cases both the raw agricultural commodities and the processed products lacked quantifiable residues, so processing factors could not be estimated.

Residues in the edible portion of food commodities

The analyses of pulp and peel in the supervised field trials on oranges in Spain and Portugal (1995) indicated a mean transfer factor from whole fruit to pulp of 0.4, range 0.33 to 0.6.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The Netherlands submitted the summary information on the monitoring of foods in commerce for the period 1994–1996 shown in Table 32.

Table 32. Residues of demeton-S-methylsulphon in food in commerce in The Netherland1994-1996.[1]

Commodity	No. analysed	No. <0.05 mg/kg	No. with residues <MRL	No. with residues >MRL	Mean, mg/kg	MRL, mg/kg
Cherries	252	251		1	0.05	0.5
Onions	97	97			<0.05	0.5
Peppers	1525	1524	1		<0.05	0.5
Cauliflower	348	346	2		<0.05	0.5
Leafy cabbage	99	99			<0.05	0.5
Head cabbage	62	62			<0.05	0.5
Head lettuce	471	469		2	0.07	0.5
Endive	1137	1136	1		<0.05	0.5
Beans (fresh; with pod)	617	616	1		<0.05	0.5
Other arable products	699	698		1	<0.05	0.05

[1] The method determines all compounds oxidized to demeton-S-methylsulphon

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported by Bayer AG.

Country/ Commodity	**MRL mg/kg**
Argentina	
Alfalfa forage	0.2
Carrot	0.2
Cereals	0.2
Citrus fruit	0.5
Cotton seed	0.1
Melon	0.5
Other vegetables	0.5
Pome fruit	0.7
Potato	0.2
Stone fruit	0.7
Sweet potato	0.2

Austria

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Beet, sugar	0.4
Cereals	0.2
Fruit	0.4
Other plant commodities	0.05
Potato	0.2
Vegetables except carrots	0.4

Belgium

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Cereals	0.2
Fruit	0.4
Other plant commodities	0 [1]
Vegetables except carrots	0.4

Canada

Asparagus	0.10 [2]
Barley	0.10 [2]
Bean	0.10 [2]
Beet, sugar	0.10 [2]
Cabbage	0.10 [2]
Cranberry	0.10 [2]
Cucumber	0.10 [2]
Gourd	0.10 [2]
Maize/Corn	0.10 [2]
Melon	0.10 [2]
Oats	0.10 [2]
Pea, Garden	0.10 [2]
Potato	0.10 [2]
Pumpkin	0.10 [2]
Strawberry	0.10 [2]
Turnip, edible	0.10 [2]
Wheat	0.10 [2]

Denmark

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Berries and small fruit	0.5
Brussels sprouts	0.5
Cabbage	0.5
Carrot	0.2
Cauliflower	0.5
Citrus fruit	0.5
Fruiting vegetables	0.5
Leafy vegetables	0.5
Legume vegetables	0.5
Pome fruit	0.5
Potato	0.2
Root vegetables exc. carrots	0.5
Stem vegetables	0.5
Stone fruit	0.5
Tropical fruit	0.5

European Community

Demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, singly or combined

Carrot	0 [3]

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methylsulphon

Other plant commodities	0.4

Finland

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Carrot	0.05
Fruit	0.4
Other vegetables	0.4
Potato	0.05

France

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Almond	0.4
Apple	0.4
Barley	0.05
Beets (Beta vulgaris)	0.4
Buckwheat, common	0.05
Chestnut	0.4
Fig	0.4
Hazel nut	0.4
Nuts	0.4
Oats	0.05
Other fruit	0.4
Pea	0.02 T
Pea preserve	0.02 T
Plum	0.4
Rape	0.05 T
Rye	0.05
Triticale	0.05
Vegetables except carrots	0.4
Wheat	0.05

Germany

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, Expressed as demeton-s-methyl

Beet, Sugar	0.1
Cereals	0.2
Currant, Black	2
Currant, Red	2
Grape	2
Other fruit	0.5
Other plant commodities	0.05
Peach	1
Plum	1
Pome fruit	1
Potato	0.2
Vegetables except carrots	0.5

Greece

Carrot	0 [4]
Other plant commodities	0.4

Israel

Sum of demeton-S-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Aubergine	0.5
Bean	0.5
Beet, sugar	0.1
Brassica vegetables	0.1
Citrus fruit	0.5
Cotton seed	0.1
Cucurbits	0.5
Forage crops	0.5
Grape	0.2
Pepper, sweet	0.2
Pome fruit	0.2

Potato	0.2
Squash, summer	0.5
Stone fruit	0.2
Tomato	0.5

Italy

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methylsulphon

Beet, sugar	0.1
Carrot	0[4]
Fruit	0.4
Other vegetables	0.4
Potato	0.1
Tobacco	0.1
Wheat	0.1

Luxembourg

Oxydemeton-methyl, demeton-S-methyl and demeton-S-methylsulfon, separately calculated or summed up to demeton-S-methyl

Carrot	0.05
Cereals	0.2
Fruit	0.4
Vegetables except carrots	0.4

Mexico

Alfalfa	5
Apple	1
Aubergine	1
Bean green	0.5
Broccoli	1
Brussels sprouts	1
Cabbage	1
Cauliflower	1
Cotton	0.1
Cucumber	1
Grape	0.1
Grapefruit	1
Lemon	1
Lettuce	2
Maize/Corn fodder	0.5
Maize/Corn grain	0.5
Melon	0.3
Onion	05
Orange	1
Pea, garden	0.3
Pea, Chick-	0.3
Pear	1
Pepper, sweet	0.75
Plum	1
Potato	0.1
Pumpkin	1
Quince	1
Safflower	1
Sorghum fodder	0.75
Sorghum grain	0.75
Strawberry	2
Walnut	0.3
Zucchini	1.0

Netherlands (Same information also reported by government of The Netherlands)

Sum of oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methylsulphon

Carrot	0.05 [4]
Cereals	0.1

Fruit	0.5
Other plant commodities	0 [1]
Other vegetables	0.5
Potato	0 [4]

Portugal

Sum of oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methylsulphon

Carrot	0.05
Fruit	0.4
Other plant commodities	0.4
Vegetables	0.4

South Africa

Sum of oxydemeton-methyl and its sulfone, expressed as oxydemeton-methyl

Apple	0.4
Apple	0.4 E
Apricot	0.4
Apricot	0.4 E
Aubergine	0.2
Bean	0.2
Citrus fruit	0.5
Cotton	0.1
Cruciferae	0.2
Cucurbits	0.4
Maize/Corn green	0.2
Nectarine	0.4
Nectarine	0.4 E
Olive	0.1
Onion	0.1
Pea, garden	0.2
Peach	0.4
Peach	0.4 E
Peanut	0.2
Plum	0.4
Plum	0.4 E
Potato	0.2
Rooibos tea	0.1
Sorghum grain	0.02
Tomato	0.2

Spain

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methylsulphon

Apple	1
Beet, sugar	0.1
Berries and small fruit	0.4
Brassica vegetables	0.4
Bulb vegetables	0.4
Cacao	0.05
Carrot	0.05
Cereals	0.05
Citrus fruit	0.4
Coffee	0.05
Cola	0.05
Forage crops and straw	0.05
Fruit and vegetables, dried	0.05
Fruiting vegetables	0.4
Herbs	0.4
Hops	1
Leafy vegetables	0.4
Legume vegetables	0.05
Mushroom	0.4
Nectarine	1

Nuts	0.4
Oil plants seed	0.05
Other pome fruit	0.4
Other root vegetables	0.4
Other stone fruit	0.4
Peach	1.0
Potato	0.05
Pulses	0.05
Spices	0.05
Stem vegetables	0.4
Sugarcane	0.05
Tea	0.05
Tobacco	0.05
Trop. and subtrop. fruit	0.4

Sri Lanka

Aubergine	0.2
Bean	0.2
Bean, mung	0.2
Cowpea	0.2
Other vegetables	0.2
Pepper, Cayenne-	1
Soya	0.2

Sweden

Sum of demeton-S-methyl, oxydemeton-methyl and demeton-S-methylsulphon, expressed as demeton-S-methyl

Cereals	0.2 [5]
Cereals flakes	0.1 [5]
Cereals flour	0.1 [5]
Cereals grain, hulled	0.1 [5]
Fruit	0.5
Potato	0.05 [5]
Vegetables	0.5

Switzerland

Single or combined with demeton-S-methylsulphon and oxydemeton-methyl, expressed as demeton-S-methylsulphon

Beet, sugar	0.4
Fruit	0.4
Vegetables except carrots	0.4

USA

Oxydemeton-methyl and cholinesterase-inhibiting metabolites

Alfalfa	5
Alfalfa chaff	11
Alfalfa hay	11
Alfalfa seed	11
Apple	1
Apricot	0.5 R Idaho
Aubergine	1
Bean, Kidney	0.5
Bean, Kidney forage	2
Bean, Lima	0.5
Bean, Lima forage	2
Beet, sugar	0.3
Beet, sugar top or leaves	0.5
Blackberry	2
Broccoli	1
Brussels sprouts	1
Cabbage	1
Capsicum (Peppers/Chilli)	0.75
Cattle fat	0.01
Cattle meat	0.01

Cattle meat by-products	0.01
Cauliflower	1
Clover green	5
Clover hay	11
Clover seed	11
Corn, sweet corn-on-the-cob	0.5
Corn, sweet fresh	0.5
Cotton seed	0.1
Cucumber	1
Goat fat	0.01
Goat meat	0.01
Goat meat by-products	0.01
Grape	0.1
Grapefruit	1
Hazel nut	0.05
Horse fat	0.01
Horse meat	0.01
Horse meat by-products	0.01
Lemon	1
Lettuce, head	2
Maize/Corn fodder	3
Maize/Corn forage	3
Maize/Corn fresh	0.5
Maize/Corn grain	0.5
Melon	0.3
Milk	0.01
Mint, pepper hay	12.5
Onion dry bulb	0.05
Orange	1
Pea, garden	0.3
Pea, garden forage	2
Pea, garden hay	8
Pear	0.3
Pig fat	0.01
Pig meat	0.01
Pig meat by-products	0.01
Plum	1
Potato	0.1
Pumpkin	0.3
Raspberry, American red	2
Safflower	1
Sheep fat	0.01
Sheep meat	0.01
Sheep meat by-products	0.01
Sorghum forage	2
Sorghum grain	0.75
Sorghum milled fractions except flour	2 F
Squash, summer	1
Squash, winter	0.3
Strawberry	2
Turnip, edible	0.3
Turnip, edible top or leaves	2
Walnut	0.3

[1] Below the limit of determination (0.05 mg/kg)
[2] Negligible residue tolerance
[3] Lower limit of analytical determination
[4] Below the limit of determination
[5] Level at or about the limit of determination

T = temporary
R = regional tolerance
E = export tolerance
F = food-additive tolerance

APPRAISAL

Oxydemeton-methyl (ODM), *S*-2-ethylsulfinylethyl *O,O*-dimethyl phosphorothioate, is a systemic and contact insecticide and is a metabolite (sulfoxide) of the insecticide demeton-S-methyl, *S*-2-ethylthioethyl *O,O*-dimethyl phosphorothioate. The 1992 JMPR carried out a complete re-evaluation of oxydemeton-methyl. The residue is currently defined as the sum of oxydemeton-methyl, demeton-S-methyl, and demeton-S-methylsulphon, expressed as oxydemeton-methyl.

The present evaluation is part of the Periodic Review Programme of the CCPR.

Extensive data and information were submitted for oxydemeton-methyl. No information was provided on demeton-S-methyl, except on its metabolism by wheat.

Animal metabolism

Metabolism studies were submitted on rats, poultry, and goats. Rats treated orally or intravenously with a single dose of [*ethylene*-^{14}C]ODM (20 mg/kg bw) eliminated at least 89% of the dose in the urine within 72 hours. In a composite 0–24 hour urine sample ODM accounted for about 50% of the administered radioactivity. The identified metabolites were 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane and 1-(ethylsulfonyl)-2-(methylsulfinyl)ethane, each 16%, demethyl-ODM and demethyl-ODM sulfone, each 2–3%.

Lactating goats were dosed orally with [*ethylene*-^{14}C]ODM for three consecutive days at a rate of 7 mg ODM per kg body weight. Urine and milk were collected daily, the goats were slaughtered 2 hours after the final dose, and tissues were collected. Water extracted virtually all of the radioactive residues from all samples except fat, from which about 85% was extracted. Milk was centrifuged, and the supernatant was eluted from C-18 solid phase extraction cartridges. The eluate contained about 80% of the original ^{14}C in the milk. The total radioactive residue (TRR) concentrations, expressed as ODM, were kidneys 13 mg/kg, liver 4.2 mg/kg, muscle 4.0 mg/kg, fat 0.62 mg/kg, and milk 3.8 mg/kg. The only compounds identified were ODM and its sulfone, with the parent constituting 23–61% of the TRR and the sulfone 0.8–23%.

Laying hens dosed with [*ethylene*-^{14}C]ODM at 6.9 mg/kg body weight for three consecutive days were killed within four hours of the final dosing, and appropriate tissues were collected and analysed. The total radioactive residues as ODM were breast 0.51 mg/kg, thigh 0.43 mg/kg, liver 0.60 mg/kg, kidney 1.4 mg/kg, skin 0.41 mg/kg, and eggs (day 3) 0.36 mg/kg. A mixture of acetone and methylene chloride extracted >80% of the TRR from all samples except eggs (70% extraction). Demethyl-ODM sulfone was the main compound in the breast (70% of the ^{14}C), thigh (80%), liver (48%), skin (100%) and eggs (100%). The main residue in the kidneys was a mixture of 2-(ethylsulfinyl)ethanesulfonic acid and 2-(ethylsulfonyl)ethanesulfonic acid (84%). These compounds also constituted a high proportion (42%) of the residue in the liver.

The results of the three metabolism studies indicate oxidation to the sulfone and cleavage of the P-S linkage. The Meeting concluded that the animal metabolism studies were adequate.

Plant metabolism

The metabolism of [*ethylene*-^{14}C]ODM in cabbage and sugar beet, of [*ethylene*-^{14}C]demeton-S-methyl in wheat , and of demeton-S-methylsulphon in apples were reported. The radioactive residues in cabbage were characterized only as highly polar and water-soluble. The radioactive residues in the foliage and roots of sugar beet were extracted and some of the major metabolites were identified by MS. ODM was a

minor component on the foliage (2% of the TRR). The main metabolites were demethyl-ODM 14%, bis(2-ethylsulfinylethyl) disulfide 12%, 2-hydroxy-3-(2-ethylsulfinylethylthio)propionic acid 14%, 2-hydroxy-3-(2-ethylsulfonylethylthio)propionic acid 10%, and 2-hydroxy-3-(2-ethylsulfonylethylsulfinyl)propionic acid 11%. All except demethyl-ODM involve cleavage of the P-S bond.

The metabolism of [*ethylene*-^{14}C]demeton-*S*-methyl by wheat grown in a greenhouse again revealed cleavage of the P-S bond with the formation of sulfonic acids, disulfides, and ethanol derivatives: 2-(ethylsulfonyl)ethanesulfonic acid 8% of the TRR, 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane 9%, 1-(ethylsulfinyl)-2-(methythio)ethane 4%, 2-ethylsulfinylethanol, 5%. ODM and ODM sulfone constituted 20% of the radioactive residue.

The metabolism of [*ethylene*-^{14}C]-demeton-S-methylsulphon by apples produced compounds formed by P-S bond cleavage, as well as demethyl- and didemethyl-demeton-S-methylsulphon (5-6% of the TRR). At day 27, demeton-S-methylsulphon (the test material) accounted for 24% of the applied radioactivity in both peel and pulp. The main metabolites were *S*-2-ethylsulfonylethyl *O*-methyl *O*-hydrogen phosphorothioate (demethylated parent) 3% in both pulp and peel, *S*-2-ethylsulfonylethyl *O*-*O*-dihydrogen phosphorothioate (didemethylated parent), 1% in pulp and 3% in peel, 2-ethylsulfonylethanesulfonic acid 3%, and 2-ethylsulfonylethanol 3% in pulp. The total of the identified compounds at 27 days accounted for less than 40% of the applied radioactivity.

The Meeting concluded that the plant metabolism studies were marginally acceptable. The details and raw data of the studies were not provided and therefore some results, particularly the quantitative results, could not be verified. Isolated metabolites were positively identified by mass spectrometry and comparison with authentic standards. The four studies indicate a common mechanism involving oxidation to the sulfone, formation of the demethyl and didemethyl derivatives, and cleavage of the P-S linkage.

Environmental fate

The Meeting received reports of studies on uptake by rotational crops, dissipation under field conditions, photolysis on soil, adsorption and desorption on four types of soil, leaching from clay, aerobic and anaerobic degradation on soil, and aerobic and anaerobic degradation in water/sediment systems.

The degradation of [*ethylene*-^{14}C]ODM on sandy loam soil was studied over a 12-month period. Volatile compounds accounted for only 9% of the applied radioactivity after 12 months. The major products were 2-ethylsulfinylethanesulfonic acid and 2-ethylsulfonylethanesulfonic acid, 20–30% of the applied radioactivity. The half-life of ODM, assuming first order kinetics, was 3.2 days. A similar degradation of ODM was observed under anaerobic conditions, but the data were not adequate to calculate a half-life. The Meeting concluded that ODM is degraded at a moderate rate under aerobic conditions in soil, with formation of the sulfonic acid after cleavage of the P-S bond.

For the rotational crop study, kale, wheat, and beetroot were planted in soil that had been treated with [*ethylene*-^{14}C]ODM at 3.4 kg ai/ha in Kansas, USA. The crops were planted at intervals of about 30, 180, and 300 days after treatment of the soil and harvested at normal maturity. Beet roots, beet tops and kale contained no residues after any planting interval. Immature wheat (boot stage) showed radioactivity equivalent to 0.02 mg/kg ODM from the 34-day planting. Wheat grain, straw and chaff contained low levels of radioactivity, 0.01–0.06 mg/kg, from the 34-day and 184-day plantings. These levels represent the sum of ODM and its metabolites, so ODM and demeton-S-methylsulphon are not expected to be quantifiable in crops planted at the intervals studied. The Meeting concluded that inadvertent residues in rotational crops were not a significant source of dietary exposure and need not be considered in estimating maximum residue levels.

[*Ethylene*-^{14}C]ODM applied to sandy loam soil was exposed to natural sunlight for 30 days. The rate of degradation was no more than in a dark control sample. A similar result was found for ODM in sterile buffer solution at pH 5 exposed to natural sunlight for 30 days. The Meeting concluded that photolysis was not a significant degradation pathway for ODM.

The adsorption and desorption of ODM was measured by a batch equilibrium procedure in four types of soil. ODM was not bound substantially to any of the soils.

The leaching of [*ethylene*-^{14}C]ODM through a sandy clay loam soil was studied. Aerobic ageing of the soil for 30 days before leaching caused a 50% loss of radioactivity as CO_2. Water (1.25 cm of simulated rainfall) was applied each day for 45 days to the aged soil. Over 80% of the radioactivity remained in the top 2.5 cm of the soil. A soil dissipation study under field conditions in California, USA, showed a half-life of ODM of about 2 days, with no tendency to migrate below 15 cm. The Meeting concluded that ODM is not leached through soil.

The degradation of [*ethylene*-^{14}C]ODM was studied in sterile buffer solution at 25ºC. The half-life decreased dramatically with increasing pH, from 94 days at pH 5 to 2.5 days at pH 9. The identified products were demethyl-ODM and 2-ethylsulfinylethanethiol (34 and 13% respectively after 35 days at pH 7).

Water and sediment from an orchard drainage ditch and from a fish pond were treated in separate experiments with [*ethylene*-^{14}C]ODM at 0.5 mg ai/l. Samples were taken on days 1, 7, 20, 41, and 91. Volatile compounds accounted for about 25% of the applied radioactivity after 91 days. After 7 days, ODM accounted for about 10% of the applied ^{14}C in the waters and <1% in the sediments. The main products were demethyl-ODM, 2-ethylsulfinylethanesulfonic acid, 2-ethylsulfonylethanesulfonic acid, demethyl-ODM sulfone, and bis(2-ethylsulfinylethyl) disulfide. ODM sulfone accounted for <1% of the applied ^{14}C at all intervals in all fractions.

A water/sediment degradation study was also conducted under anaerobic conditions for a period of 12 months. [*Ethylene*-^{14}C]ODM represented only 3% of the applied radioactivity within 14 days. The main products were 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane (18% of the applied ^{14}C in the surface water), 2-ethylsulfinylethanesulfonic acid (9% in the surface water), and 2-ethylsulfonylethanesulfonic acid (4% in the surface water).

The Meeting concluded that ODM is degraded rapidly in soil and in water via cleavage of the P-S bond.

Methods of residue analysis

Numerous similar methods were presented for enforcement and data collection. All determine the combined residue of demeton-S-methyl, oxydemeton methyl, and demeton-S-methylsulphon. The analyte measured is the sulfone. The crop sample is extracted with solvent, typically acetone/water, the extract is cleaned up (optionally) by gel permeation chromatography (GPC), oxidized with permanganate, cleaned up if necessary by solid-phase extraction (SPE), and analysed on a gas chromatograph equipped with a flame photometric detector. The oxidation step converts demeton-S-methyl and ODM to the sulfone. Extensive recovery data were presented. Typical lower limits of determination are 0.01 and 0.05 mg/kg as ODM.

The Meeting concluded that adequate methods exist for the determination of demeton-S-methyl and ODM in plant and animal commodities.

Stability of residues in stored analytical samples

Storage stability studies were reported for cabbage, maize forage, maize kernels, maize husks, lettuce, and papaya. ODM and demeton-S-methylsulphon were stable on cabbage stored frozen for 800 days. Demeton-S-methylsulphon was stable on maize forage stored frozen for 160 days, but ODM showed significant loss after 60 days. ODM was stable on lettuce and papaya stored frozen for 50 days and 13 days respectively. (The Meeting did not accept the apparent recovery from lettuce of 98% after 180 days as valid because it was the mean of 69% and 128%).

The Meeting concluded that the data were generally inadequate and contradictory, and that additional studies were highly desirable.

Definition of the residue

The residue is currently defined as the sum of oxydemeton-methyl, demeton-S-methyl and demeton-S-methylsulphon, expressed as oxydemeton-methyl. The analytical methods determine the combined residue as the sulfone. The pesticide demeton-S-methyl is metabolized to its sulfoxide, ODM. The Meeting confirmed the current definition, both for compliance with MRLs and for the estimation of dietary intake.

Residues resulting from supervised trials

Supervised field trials were reported for oranges, lemons, apples, pears, plums, grapes, cabbage, kale, kohlrabi, lettuce, peas, beans, potatoes, sugar beet, wheat, oats, barley, almonds, rape, sunflower, and cotton seed.

The Meeting was informed that instruction labels were being revised in Europe for some crops to include conditions aimed at yielding lower residues, such as reduced application rates and extended PHIs. Some labels may be awaiting approval by national governments. The Meeting based its recommendations on the available labels and did not consider pending or possible changes.

Oranges and lemons. Supervised field trials were conducted on oranges in Spain (6 trials, GAP for oranges 2 x 0.75 kg ai/ha, 30-day PHI) and Portugal (1 trial; GAP for citrus fruit 2 x 0.050 kg ai/hl (calculated 0.75 kg ai/ha), 84-day PHI). All the trials complied with GAP for Portugal and two of them with GAP for Spain, but the peel and pulp were analysed separately and their relative weights were not reported. The higher residues in the peel were used to estimate maximum residue levels, and the residues in the pulp to estimate STMRs. Two field trials on lemons in Spain and two in Italy complied with Portuguese GAP, and the Spanish trials also with Spanish GAP for oranges. The 11 trials on oranges and lemons may be combined for the estimation of maximum residue levels and STMRs to give rank orders of residues in the pulp of <0.01 (7), 0.01, 0.02 (2) and 0.04 mg/kg, and in the peel of <0.01 (5), 0.01 (2), 0.06, 0.09 and 0.13 (2) mg/kg. The Meeting estimated maximum residue levels of 0.2 mg/kg and STMRs of 0.01 mg/kg for oranges and lemons.

Apples and pears. Supervised field trials on apples in Germany (4 trials, GAP 1 x 0.025 kg ai/hl (0.40 kg ai/ha, 1500 l water/ha) before or directly after flowering, PHI about 60 days), Belgium (2 trials, no GAP reported), and France (3 trials, GAP 0.020 kg ai/hl, 60-day PHI) were reported. The trials in Belgium were at the higher rate of 0.083 kg ai/hl and did not comply with French or German GAP, and in two of the German trials application was apparently at a later growth stage. Two trials in Germany and three in France were according to GAP.

Field trials were also reported on pears: 2 in Germany, same GAP as apple; 1 in France, same GAP as apple, 2 in Italy according to Portuguese GAP (0.038 kg ai/hl, 56-day PHI) and 1 in Belgium, GAP not reported. All the trials were according to GAP except the Belgian trial with a longer PHI (100 days) than the 60 days of German GAP.

The five apple and five pear trials may be evaluated together: GAP for the two fruits is very similar and the residues are in one population. The residues in rank order were <0.01 (7), 0.03 and <0.04 (2) mg/kg. The Meeting estimated maximum residue levels of 0.05 mg/kg and STMRs of 0.01 mg/kg for apples and pears.

Plums. Four supervised field trials in Germany were according to GAP (1 x 0.025 kg ai./hl, before or directly after flowering, PHI about 60 days). The residues were 0.040, 0.014, 0.044 and 0.028 mg/kg). The Meeting concluded that 4 trials were insufficient for the estimation of a maximum residue level or an STMR and recommended withdrawal of the draft MRL.

Grapes. Supervised field trials were reported from Italy (2 trials, GAP 1 x 0.23 kg ai/ha, 40-day PHI) and Germany (3 trials, GAP 1 x 0.025 kg ai/hl applied from 2 leaves to developed inflorescences, BBCH Code 57). The residues were <0.04 (4) and 0.06 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.04 mg/kg.

Cabbages (head). Sixteen supervised field trials in Germany were according to GAP (1 x 0.15 kg ai/ha (height under 50 cm), 21-day PHI). The residues in rank order were <0.01 (6), 0.02, <0.03 (3), <0.04, <0.05 (4) and <0.06 mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.03 mg/kg for head cabbages, and recommended withdrawal of the draft MRL for Savoy cabbage.

Kale. Four trials in Germany were according to GAP (the same as for cabbages). All the residues were <0.01 mg/kg. The Meeting considered the four trials an adequate database for kale and estimated a maximum residue level of 0.01* mg/kg and an STMR of 0.01 mg/kg.

Kohlrabi. Four supervised field trials in Germany complied with GAP for kohlrabi (the same as for cabbages). The residues were <0.01 (2), 0.03 and <0.06 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.02 mg/kg .

Lettuce. Seven reported trials in Germany comprised one trial in triplicate (same location, time, and application) which complied with GAP (1 x 0.16 kg ai/ha, 21-day PHI) and two duplicate trials in which the PHIs were more than 33% below the GAP PHI and which displayed quantifiable residues. The Meeting concluded that one trial was not adequate for the estimation of a maximum residue level or STMR and recommended the withdrawal of the draft MRL for leaf lettuce.

Field peas and beans (dry). Three of five trials on field beans in Germany accorded with GAP (2 x 0.15 kg ai/ha (height under 50 cm) or 0.25 kg ai/ha (over 50 cm), 28-day PHI). The other two trials were apparently replicates of one of these. Three trials on field peas were conducted in Germany (GAP only for garden peas) and two in France (no GAP). GAP for Italy is 1 x 0.23 kg ai/ha, 21-day PHI, but the trials were at less than half this rate. All residues in the shelled dry peas were <0.01 mg/kg. The Meeting decided that the three trials on field beans, supplemented by the 5 trials on field peas whose rates and PHIs approximated GAP for field beans, provided an adequate data base for the estimation of a maximum residue level and STMR for beans but not peas. The residues in rank order were <0.01 (5), <0.04, 0.05 and 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.01 mg/kg for common bean (dry), and recommended withdrawal of the draft MRLs for beans (dry) and peas (dry).

Potatoes. Supervised trials were reported from Germany (14 trials, GAP 3 x 0.3 kg ai/ha, 21-day PHI), France (1 trial; no GAP reported), the UK (3 trials, no GAP reported), and The Netherlands (2 trials, no GAP reported). GAP for Ireland is 2-4 x 0.15 kg ai/ha (height under 50 cm) or 0.22 kg ai/ha (over 50 cm), 200 l water/ha, PHI 28 days. All the trials approximated German or Irish GAP but two German trials were discounted because they were replicates and not independent trials. In the 18 independent trials the residues in rank order were <0.01 (7), <0.02 (9) and <0.05 (2) mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.02 mg/kg.

Sugar beet (root). Supervised field trials were reported from Germany (9 trials + 2 on fodder beet, GAP 4 x 0.24 kg ai/ha, 28-day PHI) and France (2 trials, GAP 0.15 kg ai/ha, 28-day PHI). The two trials in France and two of the German trials were at excessive application rates. Seven trials on sugar beet and the two trials with fodder beat in Germany complied with GAP for sugar beet. The residues in rank order in the sugar and fodder beets were <0.01 (4) and <0.04 (5) mg/kg. The residues in the four trials at excessive rates were all <0.04 mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.04 mg/kg.

Sugar beet (tops). The residues in the leaves of the sugar and fodder beets treated according to GAP were 0.02 and <0.04 (8) mg/kg. The residues from the two German trials at excessive rates were also <0.04 mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.04 mg/kg for sugar beet leaves or tops.

Wheat, barley and rye. Supervised field trials on wheat were reported from Germany (8 trials, GAP 2 x 0.12 kg ai/ha, 21-day PHI) and the UK (1 trial; no GAP reported; Irish GAP for wheat, rye and barley: 0.12 kg ai/ha, 200 l water/ha, 21-day PHI). Six trials in Germany were according to maximum GAP conditions and complied with Irish GAP. Three trials on barley were reported from Germany and one from the UK. Two German trials and the UK trial (according to Irish GAP) were at the maximum GAP conditions and gave residues of <0.04 (2) and <0.05 mg/kg in the grain. Two supervised field trials on oats (grain residues <0.01 and 0.05 mg/kg) were submitted from Germany, but no current GAP was available.

A total of 10 trials on wheat and barley complied with GAP. The results may be combined for the evaluation of these crops and rye. The residues in the grain in rank order were <0.04 (6), <0.05 (2) and 0.05 mg/kg. The Meeting estimated maximum residue levels of 0.05* mg/kg and STMRs of 0.04 mg/kg for wheat, barley and rye.

Wheat, barley and rye straw and fodder. The 10 trials according to GAP for wheat and barley described above yielded residues in or on the straw of <0.04 (4), <0.05, 0.06, 0.12, 0.18, 1.2 and 1.3 mg/kg. The Meeting estimated maximum residue levels of 2 mg/kg and STMRs of 0.06 mg/kg for the fodders and straws of wheat, barley and rye.

Almonds. In six supervised field trials on almonds in Spain (GAP 2 x 0.25 kg ai/ha, 30-day PHI) all the PHIs were 90 days. The Meeting concluded that there were no results from trials according to GAP and that no maximum residue level or STMR could be estimated.

Rape seed. Three supervised field trials in France (GAP 0.12 kg ai/ha, unspecified PHI) yielded residues in the seed of <0.05 mg/kg after application at 0.12 kg ai/ha and a 63- or 79-day PHI. The Meeting concluded that 3 trials were insufficient to estimate a maximum residue level or STMR.

Sunflower seed. Three trials in France (GAP 1 x 0.1 kg ai/ha, PHI unspecified) gave residues in the seeds of <0.01 mg/kg after a foliar treatment at 0.1 kg ai/ha and an 83–87-day PHI. The Meeting concluded that 3 field trials were inadequate for the estimation of a maximum residue level or STMR.

Cotton seed. Ten trials in Australia (no GAP reported), 4 in Brazil (Peruvian GAP 3 x 0.25 kg ai/ha, 14-day PHI), and 7 in the USA (GAP 3 x 0.84 kg ai/ha, 14-day PHI) were reported. Two of the trials in Brazil with residues of 0.03 and 0.02 mg/kg complied with GAP and six US trials approximated GAP. The status of the remaining trials could not be ascertained from the information provided. The residues in rank order were <0.01 (2), 0.01 (3), 0.02 and 0.03 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg.

Feeding studies

Animal feeding studies on lactating dairy cows and laying hens were reported. The cows received daily doses of ODM for 28 days at levels equivalent to 10, 30, or 100 ppm in the feed. The combined residue of ODM and ODM sulfone was <0.01 mg/kg in all milk samples and in the liver, kidneys, muscle and fat. The demonstrated lower limit of quantification was 0.01 mg/kg.

Three groups of ten hens each were fed ODM in the daily diet for 28 days at rates of 0.65, 1.95, or 6.5 ppm in the feed. The combined residue was <0.01 in all egg samples and in the tissues of the 6.5 ppm group. Tissues from the other groups were not analysed.

The potential animal feed items containing ODM and the relevant maximum residue levels are as follows: potato culls and processed waste (potatoes 0.05 mg/kg), sugar beet tops, pulp, and molasses (sugar beet 0.05 mg/kg), wheat and barley grain (0.05 mg/kg) and straw (2 mg/kg), cotton seed and cotton seed oil, meal and hulls (cotton seed 0.05 mg/kg). A dietary burden for dairy cattle can be calculated as follows: barley straw (2 mg/kg/0.89 dry matter) x 60% of the diet + cotton seed (0.05 mg/kg/0.88 dry matter) x 20% of the diet + sugar beet tops (0.05 mg/kg/0.23 dry matter) x 20% of the diet = 1.5 ppm. The dietary burden for poultry would be cotton seed meal (0.05 mg/kg/0.89 dry matter x 20% of the diet) + grain (0.05 mg/kg/0.88 dry matter x 50% of the diet) = 0.04 ppm. The cattle and hen feeding studies at maximum rates represent approximately 66 times (100/1.5) and 160 times (6.5/0.04) these levels respectively, and no residues were found. The Meeting concluded that quantifiable residues of demeton-S-methyl, ODM, and/or demeton-S-methylsulphon in commodities of animal origin (meat, milk, poultry and eggs) are unlikely and that MRLs could be set at the practical limits of quantification of 0.05* mg/kg for all commodities except milk and at 0.01* mg/kg for milk. The Meeting recommended these levels as MRLs, estimated STMRs of 0 mg/kg for all the commodities, and recommended withdrawal of the draft MRL for derived milk products.

Processing studies

Processing studies on apples and cotton seed were reported. The processing factor for apple juice was 1 and for sauce 0.5. The processing factor for refined cotton seed oil was 0.2, for meal 0.6, and for hulls 0.2. Limited information on processing was provided for sugar beet, kale, potatoes, rape, and wheat, but in all cases the raw agricultural commodity lacked quantifiable residues.

RECOMMENDATIONS

On the basis of data from supervised trials the Meeting estimated the maximum residue levels and STMRs listed below. The maximum residue levels are recommended for use as MRLs.

All recommendations are based on the use of oxydemeton-methyl. No data were supplied for the use of demeton-S-methyl or demeton-S-methylsulphon. The residues will be the sum of oxydemeton-methyl and demeton-S-methylsulphon. Any inadvertent demeton-S-methyl would also be included.

Definition of the residue for compliance with MRLs and for the estimation dietary intake: sum of demeton-S-methyl, oxydemeton-methyl, and demeton-S-methylsulphon, expressed as oxydemeton-methyl.

Commodity		Recommendations, mg/kg		
CCN	Name	MRL		STMR
		New	Previous	
AL 1020	Alfalfa fodder	W	5	
FP 0226	Apple	0.05	1	0.01
JF 0226	Apple juice	-	-	0.005
	Apple sauce			0.01
GC 0640	Barley	0.05*	0.2	0.04
AS 0640	Barley straw and fodder, dry	2	-	0.055
VD 0071	Beans (dry)	W	0.01*	
VB 0400	Broccoli	W	1	
VB 0402	Brussels sprouts	W	1	
VB 0403	Cabbage, Savoy	W	0.01*	
VB 0041	Cabbages, Head	0.05*	1	0.03
MF 0812	Cattle fat	0.05*	0.05*	0
VB 0404	Cauliflower	W	0.01*	
FS 0013	Cherries	W	1	
AL 1031	Clover hay or fodder	W	5	
VD 0526	Common bean (dry)	0.1	-	0.01
VP 0526	Common bean (pods and/or immature seeds)	W	0.2	
SO 0691	Cotton seed	0.05	0.05	0.01
OR 0691	Cotton seed oil, edible	-	-	0.002
	Cotton seed meal			0.006
	Cotton seed, hulls			0.002
VC 0424	Cucumber	W	0.5	
LD 0106	Derived milk products	W	0.05	
VO 0440	Egg plant	W	0.2	
PE 0112	Eggs	0.05*	0.05*	0
VP 0528	Garden pea (young pods)	W	0.1	
FC 0203	Grapefruit	W	0.1	
FB 0269	Grapes	0.1	0.5	0.04
VL 0480	Kale	0.01*	0.05	0.01
VB 0405	Kohlrabi	0.05	0.01*	0.02
FC 0204	Lemon	0.2	1	0.01
VL 0483	Lettuce, Leaf	W	2	
VP 0534	Lima bean (young pods and/or immature beans)	W	0.2	
GC 0645	Maize	W	0.2	
AS 0645	Maize fodder	W	5	
FC 0206	Mandarin	W	0.5	
MM 0097	Meat of cattle, pigs and sheep	0.05*	0.05*	0
ML 0106	Milks	0.01*	0.01*	0
HH 0738	Mints	W	20	
GC 0647	Oats	W	0.2	
VA 0385	Onion, Bulb	W	0.05	
FC 0004	Oranges, Sweet, Sour	0.2	0.5	0.01
FS 0247	Peach	W	1	
FP 0230	Pear	0.05	0.5	0.01
VD 0072	Peas (dry)	W	0.01*	
VO 0051	Peppers	W	1	
MF 0818	Pig fat	0.05*	0.05*	0
FS 0014	Plums (including Prunes)	W	0.5	

Commodity		Recommendations, mg/kg		
CCN	Name	MRL		STMR
		New	Previous	
VR 0589	Potato	0.05*	0.2	0.02
PF 0111	Poultry fats	0.05*	0.05*	0
PM 0110	Poultry meat	0.05*	0.05*	0
VC 0429	Pumpkins	W	0.1*	
GC 0650	Rye	0.05*	-	0.04
AS 0650	Rye straw and fodder, dry	2	-	0.055
SO 0699	Safflower seed	W	1	
MF 0822	Sheep fat	0.05*	0.05*	0
GC 0651	Sorghum	W	0.5	
AF 0651	Sorghum forage (green)	W	1	
AS 0651	Sorghum straw and fodder, dry	W	3	
VC 0431	Squash, Summer	W	0.1*	
FB 0275	Strawberry	W	0.5	
VR 0596	Sugar beet	0.05*	0.05*	0.04
AV 0596	Sugar beet leaves or tops	0.05*	0.5	0.04
VO 0447	Sweet corn (corn-on-the-cob)	W	0.05	
VO 1275	Sweet corn (kernels)	W	0.05	
VO 0448	Tomato	W	0.5	
TN 0085	Tree nuts	W	0.05*	
VR 0506	Turnip, Garden	W	0.1*	
AV 0506	Turnip leaves or tops	W	5 fresh wt	
VC 0432	Watermelon	W	0.2	
GC 0654	Wheat	0.05*	0.2	0.04
AS 0654	Wheat straw and fodder, dry	2	-	0.055
VC 0433	Winter squash	W	0.1*	

FURTHER WORK OR INFORMATION

Desirable

Data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton-methyl are highly desirable. The available information was not representative of the various crop groups, did not cover extended storage intervals, and suggested variable storage stability.

DIETARY RISK ASSESSMENT

STMRs have been estimated for oxydemeton-methyl in 30 commodities, of which five are processed commodities and seven are products of animal origin. Where consumption data were available, these STMRs were used in the estimate of dietary risk. No MRLs were used.

The International Estimated Daily Intakes for the five GEMS/Food regional diets ranged from 10 to 90% of the ADI. The Meeting concluded that the intake of residues of oxydemeton-methyl resulting from its uses that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Anderson, C., December 13, 1987. Degradation characteristics of oxydemeton-methyl (Metasystox®) in water/sediment systems. Unpublished report no. PF 2915. Bayer AG.

Anon. 1973. Residue trials with E 605 Combi in cauliflower in Germany. Bayer report nos. 0327-73, 0328-73, 0329-73. Unpublished.

Anon. 1973. Residue trials with E 605 Combi in sugar beet in Germany. Bayer report nos. 0318-73, 0319-73, 0320-73. Unpublished.

Anon. 1974. Residue trials with E 605 Combi in kohlrabi in Germany. Bayer report no. 4720-74. Unpublished.

Anon. 1975. Residue trials with E 605 Combi in lettuce in Germany. Bayer report nos. 4700-75, 4701-75. Unpublished.

Anon. 1975. Residue trials with Metasystox R in field beans in Germany. Bayer report nos. 4809-75, 4810-75, 4811-75. Unpublished.

Anon. 1975. Residue trials with Metasystox R in kohlrabi in Germany. Bayer report nos. 4833-75, 4834-75, 4835-75. Unpublished.

Anon. 1975. Residue trials with Metasystox R in lettuce in Germany. Bayer report nos. 4800-75, 4801-75, 4802-75. Unpublished.

Anon. 1976. Residue trials with E 605 Combi in cauliflower in Germany. Bayer report nos. 4710-76, 4711-76, 4712-76. Unpublished.

Anon. 1976. Residue trials with E 605 Combi in white cabbage in Germany. Bayer report nos. 4716-76, 4717-76, 4718-76. Unpublished.

Anon. 1976. Residue trials with Metasystox R 250 EC in cauliflower in Germany. Bayer report nos. 4810-76, 4811-76, 4812-76. Unpublished.

Anon. 1977. Residue trials with E 605 Combi in potatoes in Germany. Bayer report nos. 4700/77, 4702/77. Unpublished.

Anon. 1978. Residue trials with E 605 Combi in cauliflower in Germany. Bayer report nos. 4718/6118-78, 4719/6119-78. Unpublished.

Anon. 1979. Residue trials with Metasystox R in cauliflower in Germany. Bayer report nos. 4805-79, 4806-79. Unpublished.

Anon. 1979. Residue trials with Metasystox R in field beans in Germany. Bayer report nos. 4819-79, 4820-79. Unpublished.

Anon. 1979. Residue trials with Metasystox R in kohlrabi in Germany . Bayer report no. 4804-79. Unpublished.

Anon. 1979. Residue trials with Metasystox R in white cabbage in Germany. Bayer report no. 4803-79. Unpublished.

Anon. 1980. Residue trials with Metasystox R in kohlrabi in Germany. Bayer report no. 4800-80. Unpublished.

Anon. 1985. Effect of Frozen Storage at 0 to -10 Degrees Fahrenheit on Residues. Report no. 87249. Mobay Corp. Unpublished.

Anon. 1986. Residue trials with E 605 Combi in cauliflower in Germany. Bayer report nos. 6102-86, 6103-86. Unpublished.

Anon. 1986. Residue trials with Metasystox R in cauliflower in Germany. Bayer report nos. 4808-86, 4809-86. Unpublished.

Anon. 1988. Residue trials with Metasystox R 250 EC in cotton in Brasilia. Report nos. BRA-1003-88-A and BRA-1003-88-B. Bayer do Brasil S.A. Unpublished.

Anon. 1989. Residue trials with FCR 4545 & R 2170 258 EC in sunflower in France. Bayer report nos. 0097-89, 0098-89. Unpublished.

Anon. 1990. Residue trials with Metasystox R 250 EC in cotton in Brasilia. Report nos. BRA-GOSHI89-1-A. and BRA-GOSHI89-1-B, Bayer do Brasil S.A. Unpublished.

Anon. 1991. Residue trials with FCR 4545 & R 2170 258 EC in field pea in France. Bayer report no. RA-2051/91 (incl. 0448-91, 0449-91). Unpublished.

Anon. 1991. Residue trials with FCR 4545 & R 2170 258 EC in winter rape in France. Bayer report nos. 0450-91, 0451-91, 0453-91 (RA-2052/91). Unpublished.

Anon. 1991. Residue trials with FCR 4545 & R 2170 258 EC in winter rape in France. Bayer report nos. 0450-91, 0451-91, 0453-91 (RA-2052/91). Unpublished.

Anon. 1991. Residue trials with Metasystox R 250 EC in apples and pears in France. Bayer report nos. 0528-91, 0529-91, 0568-91 (RA-2107/91). Unpublished.

Anon. 1991. Residue trials with Metasystox R in sugar beet in France. Bayer report nos. 0531-91, 0532-91 (RA-2111/91). Unpublished.

Anon. 1996. Residues of Oxydemeton-Methyl in Cotton seed. Report no. KGW 047/96 (33/92). Bayer Australia Ltd. Unpublished.

Anon. 1996. Residues of Oxydemeton-Methyl in Cotton seed. Report no. KGW 103/96 (46/93). Bayer Australia Ltd. Unpublished.

Anon. 1996. Residues of Oxydemeton-Methyl in Cotton seed. Report no. KGW 157/96 (29/94). Bayer Australia Ltd. Unpublished.

Blaß, W. 1990. Modification to method 00009. Method no. 00009/M 002. Bayer AG, Germany. Unpublished.

Blaß, W. 1991. Modification of/supplement to method. 00015. Method no. 00015/M 009 (RA-95/91). Bayer AG, Germany. Unpublished.

Burger, K. 1988. Modification to method 00009. Method no. 00009/M 004. Bayer AG, Germany. Unpublished.

Burger, K. 1990. Modification to method 00009. Method no. 00009/M 003. Bayer AG, Germany. Unpublished.

Daly, D., October 27, 1987; revised October 18, 1988. Soil Adsorption/Desorption with ^{14}C-Metasystox-R. Unpublished report no. MR 95099. Mobay Corp.

De Monte, A. J. 1996. Determination of Oxydemeton-Methyl residues in cotton seed and cotton trash after two applications of Metasystox R 250 EC to CS 50 cotton. Report no. ADM 038/96. Bayer Australia Ltd. Unpublished.

Grace, T.J. and Cain, K.S., March 14, 1990. Dissipation of Oxydemeton-methyl in California Soils. Mobay Report no. MR 99837. Unpublished.

Hanlon, E. M. 1996. Determination of Oxydemeton-Methyl residues in cotton seed and cotton trash after two applications of Metasystox R 250 EC to CS 189+cotton. Report no. EMH 404/96. Bayer Australia Ltd. Unpublished.

Heinemann, O., Seym, M. 1998. Determination of residues of Enduro 258 EC in or on potato following spray application in the field in Germany, Great Britain and France. Bayer report no. RA-2147/96 (incl. 0661-96, 0662-96, 0663-96, 0664-96). Unpublished.

Hellbusch, May 19, 1983. Partition coefficient of Oxydemeton-methyl (Metasystox R, E 2170) at 21 °C. Unpublished report no. PC890. Bayer AG.

Hicks, S.C. February 22, 1990. ®Metasystox-R Insecticide-California Soil Dissipation Study. Mobay Report no. MR 100065.

Hild, J. and Thier, H.P. 1979. Organophosphor-Pestizide mit Thioäthergruppen (Organophosphorous Pesticides with Thioether Groups). DFG analytical multiresidue method S 16 (Bayer nos. 00085 and I 311), Deutsche Forschungsgesellschaft, VCH Verlagsgesellschaft; 5th edition, 1979.

Jackson, S.B.; Kesterson, A. and Lawrence, L.J., July 12, 1988. Soil Surface Photolysis of [^{14}C]Metasystox-R in Natural Sunlight . Unpublished report no. MR 95085. Mobay Corp.

Jacobsen, K., October 9, 1989. Oxydemeton-Methyl Field Dissipation For Terrestrial Use Single and Multiple Application Plot at Chualar, California Site. Unpublished report no. MR 100060. Mobay Corp. (field parts to MR 99837).

Kasper, A.M. and Shadrick, B.A., May 27, 1993. Abbreviated Soil Dissipation of ^{14}C-Oxydemeton-metyl on California Soils . Unpublished report no. MR 105156. Miles Inc.

Kesterson, A.; Marsh, D.; Lawrence, B. and Lawrence, L.J., July 1, 1988. Solution Photolysis of [^{14}C]Metasystox-R in Natural Sunlight. Unpublished report no. MR 98015. Mobay Corp.

Krohn, J., November 28, 1986. Unpublished report no. PC883. Bayer AG.

Krohn, J., March 11, 1987. Organic Solubility of E 2170. Unpublished report no. PC889. Bayer AG.

Krohn, J., May 22, 1987. Water solubuility of Oxydemeton-methyl (Metasystox R, E 2170) at 20°C. Unpublished report no. PC888. Bayer AG.

Krohn, J., June 4, 1987. Calculation of the Henry law constant of Oxydemeton-methyl. Unpublished report no. PC 885 Bayer AG.

Lee, S.G.K. and Wood, S.E. September 24, 1987. Residues in Tissues and Eggs of Chicken Fed ®Metasystox-R. Unpublished report no. MR 94886. Mobay Corp.

Lenz, C. A. and Smyser, B. P. 1989. Storage Stability of Metasystox-R in Frozen Cabbage. Report no. 98553. Mobay Corp. Unpublished.

Leslie, W. L. 1989. Metasystox R-Magnitude of the Residue on Cotton. Mobay report no. 978499. Unpublished.

Leslie, W. L. 1989. Metasystox-R-Magnitude of the Residues on Cotton Processed Products. Report no. 99210. Mobay Corp. Unpublished.

Marsh, J.D.; Iden, B.H. and Lawrence, L.J., October 28, 1987. Quantitative Characterization of Residues in Tissues and Eggs of Laying Hens Treated Orally for Three Consecutive Days with ®Metasystox-R-^{14}C . Unpublished report no. MR 94955. Mobay Corp.

Merricks, D.L., October 28, 1987. ^{14}C-Metasystox-R Goat Metabolism Study. Unpublished report no. MR 94956. Mobay Corp.

Mix, K.H. and Berg, G., March 24, 1988.Thermal Stability of the Agrochemical Active Ingredient Oxydemeton-methyl. Unpublished report no. PC891. Bayer AG.

Morris, R. A. 1980. The Effect of Frozen Storage at 0 to -10 °F on Metasystox R Residues in Corn. Report no. 67490. Mobay Corp. Unpublished.

Morris, R. A. 1980. The Effect of Frozen Storage at 0 to -10 °F on Metasystox R Residues in Lettuce. Report no. 68693. Mobay Corp. Unpublished.

Obrist, J.J. and Thornton, J.S., February 3, 1978. Leaching Characteristics of Aged ®Metasystox-R Soil Residues. Unpublished report no. MR 63057. Mobay Corp.

Ohs, P. 1992. Method for the Gas Chromatographic Determination of Residues of the Insecticidal Compounds Beta-Cyfluthrin and Cyfluthrin in Plant Materials and their Processed Products by Online LC-GC-Coupling. Additional Determination of Fenitrothion and Oxydemeton-methyl. Method no. 00255, report no. RA-321/91. Bayer AG, Germany. Unpublished.

Ohs, P. 1993. Determination of residues of FCR 4545 & R 2170 258 EC in or on fodder beet and sugar beet under actual use conditions in the Federal Republic of Germany. Bayer report no. RA-2054/91 (incl. 0085-91, 0086-91, 0087-91, 0088-91). Unpublished.

Olson, G.L. *et al*., December 22, 1989. Aerobic Metabolism of [^{14}C]Metasystox-R® in Sandy Loam Soil . Unpublished report no. MR 99735. Mobay Corp.

Olson, G.L. *et al*., December 22, 1989. Anaerobic Metabolism of [^{14}C]Metasystox-R® in Sandy Loam Soil . Unpublished report no. MR 99736. Mobay Corp.

Olson, T. J. 1967 (revised 1971). Determination of Meta-Systox-R residues in Cotton seed and Walnuts by Thermionic Emission Gas Chromatography. Chemagro (Baychem) report no. 21590, Bayer method no. I 48 Unpublished.

Pither, K.M. and Puhl, R.J., September 6, 1978; revised November 29, 1989. Stability of ™Metasystox-R in Sterile Aqueous Buffer Solutions . Unpublished report no. MR 66500. Mobay Corp.

Placke, F.J., March 16, 1988. Dissociation constant of Oxydemeton-methyl. Bayer report no. PC 886. Unpublished.

Schmidt, B. 1989. Metasystox R-Magnitude of the Residue on Unprocessed Raw Sugar Beet and Sugar Beet Processed Products. Mobay report no. 99781 (incl. 0625-88, 0626-88). Unpublished.

Schmidt, B. 1989. Metasystox R-Magnitude of the Residue on Unprocessed Raw Sugar Beet and Sugar Beet Processed Products. Mobay report no. 99781 (incl. 0625-88, 0626-88). Unpublished.

Schmidt, B. 1992. Summarized presentation and assessment of the residue behaviour of Metasystox R in cereal growing in spring wheat. Bayer report no. PF-3709 (incl. 0628-90, 0629-90). Unpublished.

Schmidt, B. 1992. Synopsis and Evaluation of the Residue Behaviour of Metasystox R in Vegetables (White Cabbage). Bayer report no. PF-3705 (incl. 0630-90, 0631-90). Unpublished.

Schmidt, B. 1992. Test on the residue behaviour of FCR 4545 & R 2170 in sugar beets. Bayer report no. PF-3744 (incl. 0528-90, 0529-90, 0530-90, 0531-90). Unpublished.

Schmidt, B. 1992. Tests on the residue behaviour of FCR 4545 & R 2170 in cereals. Bayer report no. PF-3743 (incl. 0532-90, 0533-90, 0534-90). Unpublished.

Schmidt, B. 1992. Zusammenfassende Darstellung und Bewertung zum Rückstandsverhalten von Metasystox R im Kartoffelanbau. Bayer report no. PF-3707 (incl. 0624-90, 0625-90). Unpublished.

Schmidt, B. 1992. Zusammenfassende Darstellung und Bewertung zum Rückstandsverhalten von Metasystox R im Kernobstanbau in Apfel. Bayer report no. PF-3708 (incl. 0626-90, 0627-90). Unpublished.

Schmidt, J.M. and Anderson, T.J., August 25, 1993. Anaerobic Aquatic Metabolism of ^{14}C-Oxydemeton-methyl. Unpublished report no. MR 105039. Miles Inc.

Schmidt, J.M.; Anderson, T.J. and Dyer, D.G. June 24, 1993. Aerobic Soil Metabolism of ^{14}C-Oxydemeton-methyl. Unpublished report no. MR 105038. Miles Inc.

Sevekow, May 21, 1980. Determination of vapour pressure of Oxydemeton-methyl. Unpublished report no. PC884. Bayer AG.

Seym, M. 1993. Determination of residues of FCR 4545 & R 2170 258 EC in or on common oat, spring barley and spring wheat under actual use conditions in the Federal Republic of Germany. Bayer report no. RA-2055/91 (incl. 0090-91, 0091-91, 0092-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on grape under actual use conditions in Italy. Bayer report no. RA-2112/91 (incl. 0533-91, 0534-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on apple and pear under actual use conditions in Belgium. Bayer report no. RA-2106/91 (incl. 0509-91, 0510-91, 0511-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on grape under actual use conditions in Germany. Bayer report no. RA-2109/91 (incl. 0558-91, 0559-91, 0560-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on winter barley and winter wheat under actual use conditions in Germany. Bayer report no. RA-2110/91 (incl. 0555-91, 0556-91, 0557-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on winter barley and winter wheat under actual use conditions in Great Britain. Bayer report no. RA-2114/91 (incl. 0501-91, 0502-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on potato under actual use conditions in Germany. Bayer report no. RA-2108/91 (incl. 0553-91, 0554-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on potato under actual use conditions in Great Britain. Bayer report no. RA-2113/91 (incl. 0503-91, 0505-91). Unpublished.

Seym, M. 1993. Determination of residues of Metasystox R in or on grape under actual use conditions in Italy. Bayer report no. RA-2112/91 (incl. 0533-91, 0534-91). Unpublished.

Seym, M. 1993. Modification to method 00009. Method no. 00009/M 007 (RA-752/93). Bayer AG, Germany. Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC following spray application on orange in Spain. Bayer report no. RA-2099/94 (incl. 0386-94, 0387-94, 0662-94). Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC following spray application on almond in Spain. Bayer report no. RA-2022/95 (incl. 0358-95, 0448-95, 0449-95, 0450-95). Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC in or on apple and pear following spray application in Germany. Bayer report no. RA-2018/95 (incl. 0126-95, 0127-95, 0129-95, 0130-95). Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC in plum following spray application in Germany. Bayer report no. RA-2020/95 (incl. 0132-95, 0443-95, 0444-95, 0445-95). Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC on apple and pear in France and Italy. Bayer report no. RA-2019/95 (incl. 0348-95, 0349-95, 0350-95, 0351-95). Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC on potato following spray application in Germany. Bayer report no. RA-2090/93 (incl. 0065-93, 0186-93, 0187-93, 0188-93, 0189-93, 0190-93). Unpublished.

Seym, M. 1996. Determination of residues of Metasystox R 250 EC on field pea following spray application in Germany. Bayer report no. RA-2021/95 (incl. 0347-95, 0446-95, 0447-95). Unpublished.

Seym, M. and Heinemann, O. 1996. Determination of residues of Metasystox R 250 EC on lemon and orange following spray application in Spain, Italy and Portugal. Bayer report no. RA-2017/95 (incl. 0352-95, 0354-95, 0356-95, 0357-95, 0439-95, 0440-95, 0441-95, 0442-95). Unpublished.

Seym, M. and Heinemann, O. 1996. Determination of residues of Metasystox R 250 EC on curly kale following spray application in Germany. Bayer report no. RA-2005/94 (incl. 0020-94, 0021-94, 0023-94, 0024-94). Unpublished.

Seym, M. and Nüsslein, F. 1995. Determination of residues of Enduro 258 EC in or on sunflower under actual use conditions in France. Bayer report no. RA-2042/93 (incl. 0370-93, 0371-93). Unpublished.

Seym, M. and Nüsslein, F. 1995. Determination of residues of Metasystox R 250 EC in or on almond under actual use conditions in Spain. Bayer report no. RA-2091/93 (incl. 0427-93, 0429-93). Unpublished.

Seym, M. and Nüsslein, F. 1995. Determination of residues of Metasystox R 250 EC in or on lettuce under actual use conditions in Germany . Bayer report no. RA-2093/93 (incl. 0064-93, 0191-93, 0192-93, 0193-93). Unpublished.

Smyser, B.P. and Halpin, R.E. October 19, 1987. Residues in Tissues and milk of Dairy Cows Fed ®Metasystox-R. Unpublished report no. MR 94916. Mobay Corp.

Smyser, B.P. and Halpin, R.E. October 2, 1987. Metabolism of Metasystox-R in Cabbage.Unpublished report no. MR 95012. Mobay Corp.

Specht, W. 1990. Modification to method 00086. Method no. 00086/M 012. Bayer AG, Germany. Unpublished.

Specht, W. and Thier, H.-P. 1989. Organochlor- und Organophosphor-Verbindungen sowie stickstoffhaltige und andere Pflanzenschutzmittel (Organochlorine, Organophosphorous, Nitrogen-Containing and Other Pesticides). DFG analytical multiresidue method S 19 (Bayer nos. 00086 and I 409), Deutsche Forschungsgesellschaft, VCH Verlagsgesellschaft; 10th edition, 1989.

Specht, W. and Tillkes, M. 1982. DFG Aufbereitungsverfahren (Processing Procedure) XII 6. Deutsche Forschungsgesellschaft, VCH Verlagsgesellschaft; 6th edition, 1982.

Stevenson, T.L. September 29, 1989. Residues of [^{14}C]Metasystox® in Rotational Crops. Unpublished report no. MR 99709. Mobay Corp.

Thornton, J. S. and Olson, T. J. 1967. Determination of Meta-Systox-R Residues in Lettuce and Sugar Beets by Thermionic Emission Gas Chromatography. Method no. I 47 (Chemagro report no. 21000). Unpublished.

Thornton, J. S., Olson, T. J. and Wagner, K. 1977. Determination of Residues of Metasystox R and Metabolite in Plant and Animal Tissues and Soil. Method no. 00246 (I 219). J.Agri. Food Chem. **25**, 573-576.

Wagner, 1974. Modification to Report no. 21000. Method no. RA-133/74. Bayer AG, Germany. Unpublished.

Wagner, K. and Oehlmann, L. August 24, 1987. Metabolism of Demeton-S-methyl in Wheat. Unpublished report no. PF-2701. Bayer AG.

Wagner, K. and Thornton, J. S. 1977. Method for the gas-chromatographic determination of Metasystox (i) and Metasystox R residues in plants, soil and water. Method no. 00009, Pflanzenschutz-Nachrichten Bayer 30/1977, 1, page 1-17.

Wagner, K., Bornatsch, W. and Brauner, A. January 16, 1989. Studies on the Metabolism of Demeton-S-methylsulfoxied in Sugar Beets. Unpublished report no. PF 3109. Bayer AG.

Wagner, K. *et al.*, November 11, 1984. Metabolism of Demeton-S-methyl-sulfone in Apples. Unpublished report no. PF-2425. Bayer AG.

Walter, B.A. and Kane, V. November 17, 1989. Disposition and Metabolism of [^{14}C]Metasystox-R in Male and Female Sprague-Dawley Rats. Unpublished report no. MR 99123. Mobay Corp