OXYDEMETON-METHYL (166)

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EXPLANATION

Oxydemeton-methyl was evaluated for residues by the 1998 JMPR within the CCPR Periodic Review Programme, for residues by the 1999 Meeting and toxicology by the 2002 Meeting.

At the 31st (1999) Session of the CCPR, the JMPR was asked to clarify whether demeton-S-methyl and demeton-S-methylsulphon should remain in the definition of the residue of oxydemeton-methyl since it was believed that registration of these compounds would not be retained in the future.

At the 32nd Session of the CCPR, the Committee withdrew the draft MRLs for several commodities because there was no GAP for them, advanced the new proposed draft MRLs to Step 5 and returned other draft MRLs to Step 6 due to intake concerns, requesting more detailed information on support for oxydemeton-methyl. The Committee questioned the definition of the residue recommended by the 1999 JMPR and stated that as demeton-S-methyl was no longer supported and there was no GAP, it should not be included. However it was pointed out that demeton-S-methyl could not be distinguished from oxydemeton-methyl in analysis and could be generated from this compound during analytical processes. The Committee decided that the note "The residue definition and MRLs are based on the use of oxydemeton-methyl only" should be added to the residue definition.

At the 33rd Session the Committee noted the written comment from the EC expressing a general reservation (lack of an acute RfD) and specific reservations on MRLs for grapes, lemon and oranges, sweet, sour (acute risk) and decided to return the draft MRLs to Step 6.

At the 35th Session the Committee decided to return all MRLs to Step 6 pending the reporting of short-term intake calculations by the JMPR and the submission of data by the manufacturer to the 2004 JMPR for a review of the definition of the residue.

The present Meeting received new data on physical and chemical properties (partially updated information), analytical methods, fate of residues in processing, plant metabolism (apple), residue data (apples, pears, grapes, cabbage, Brussels sprouts, cauliflower, field peas, potatoes, sugar beet, fodder beet, wheat, barley, rape and sunflower), and information on GAP and on national MRLs.

IDENTITY

Physical and chemical properties (only new information is listed)

Pure active ingredient:

Physical state, colour	pale yellow liquid (Krohn, 1999)
Odour	slight mercaptan (Krohn, 1999)
Vapour pressure	3.9×10^{-5} hPa at 20°C (Krohn, 1999) 5.2×10^{-5} hPa at 25°C (Krohn, 1999)

Solubility in organic solvents, g/l, at 20°C (Krohn, 2002)

n-hexane	>0.1
toluene	>250

dichloromethane	>250
2-propanol	>250
1-octanol	>250
polyethylenoglycol + ethanol	>250
acetone	>250
dimethylformamide	>250

Relative density 1.31 (Krohn, 1999)

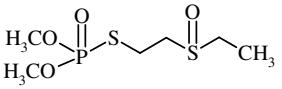
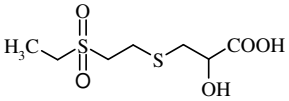
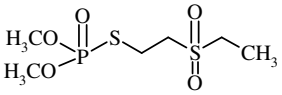
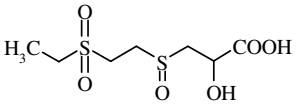
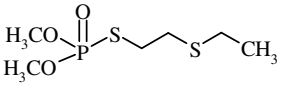
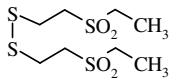
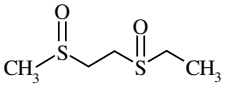
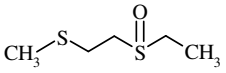
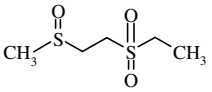
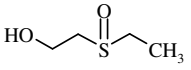
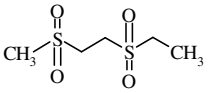
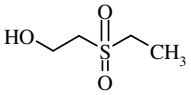
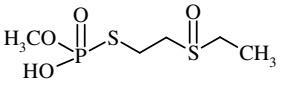
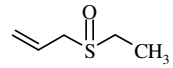
Technical formulation (purity 53.6 %)

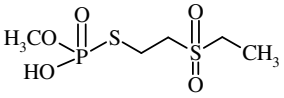
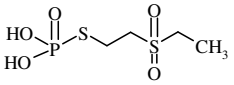
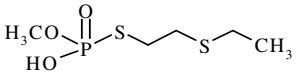
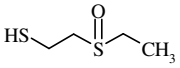
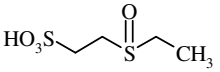
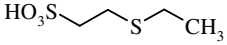
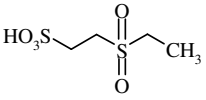
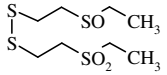
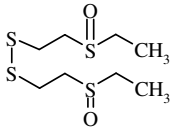
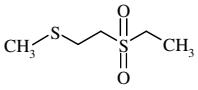
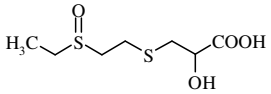
Flammability Auto ignition temperature 285°C (Eberz, 1999)

Flash point 80.5°C (Eberz, 1999)

METABOLISM AND ENVIRONMENTAL FATE

In this section the following compound codes were used.

Code	Compound	Code	Compound
ODM	 oxydemeton-methyl	M13	 2-hydroxy-3-[(2-ethylsulfonyl)ethylthio]propionic acid
M01	 demeton-S-methylsulphon	M14	 2-hydroxy-3-[(2-ethylsulfonyl-2-ethyl)-sulfinyl]-propionic acid
M02	 demeton-S-methyl	M15	 bis[2-(ethylsulfonyl)ethyl] disulfide
M03	 1-(ethylsulfinyl)-2-(methylsulfinyl)ethane	M16	 1-(ethylsulfinyl)-2-(methylthio)ethane
M04	 1-(ethylsulfonyl)-2-(methylsulfinyl)ethane	M17	 2-ethylsulfinyl-ethanol
M05	 1-(ethylsulfonyl)-2-(methylsulfonyl)ethane	M18	 2-ethylsulfonylethanol
M06	 demethyl-oxydemeton-methyl	M19	 2-ethylsulfinylethylene

Code	Compound	Code	Compound
M07	 demethyl-demeton-S-methylsulphon	M20	 S-[2-(ethylsulfonyl)ethyl] phosphorothioate
M08	 demethyl-demeton-S-methyl	M21	 2-ethylsulfinylethanthiol
M09	 2-(ethylsulfinyl)ethanesulfonic acid	M22	 2-(ethylthio)ethanesulfonic acid
M10	 2-(ethylsulfonyl)ethanesulfonic acid	M23	 2-(ethylsulfinyl)ethyl [2-(ethylsulfonyl)ethyl disulfide
M11	 bis[2-(ethylsulfinyl)ethyl] disulfide	M24	 1-(ethylsulfonyl)-2-(methylthio)ethane
M12	 2-hydroxy-3-[(2-ethylsulfinyl)ethyl)thio]propionic acid		

Plant metabolism

Apples. The metabolism of [ethylene-1-^{14}C]oxydemeton-methyl in apples was investigated in the field (McEwen, 2002). Apple trees were sprayed twice at a nominal concentration of 1.4 g ai/l applied at a nominal rate of 350 g ai/ha approximately four (pink bud stage BBCH 57) and three months before harvest (flowers fading BBCH 67).

Samples of fruit and leaves, including controls, were taken for analysis

1. 2 h after 1st application; leaves only
2. 2 h after 2nd application; leaves only
3. 1st intermediate sample (approximately 60 days before harvest); fruit and leaves
4. 2nd intermediate sample (approximately 30 days before harvest); fruit and leaves
5. Harvest; fruit and leaves

All samples were surface-washed with acetonitrile. The peel, pulp and leaves were homogenized and extracted with acetonitrile followed by acetonitrile:water (1:1). The residues were extracted with cellulase and then with hydrochloric acid. Washes and extracts containing >10% of the radioactivity in the fruit or leaves were analysed by normal phase TLC. Components were characterized by co-chromatography with authentic reference standards.

Immediately after the first application 78.9% (255 mg/kg) of the radioactivity in the surface washes and extracts was accounted for by ODM and after the second 97.5% (128.1 mg/kg).

Analysis of leaf samples at the 1st intermediate and harvest stages detected ODM, whereas at the 2nd intermediate sampling it was not detected. However all samples contained polar material (M07 and P3) (4% of the radioactivity at 1st intermediate, 9% by 2nd) but at harvest this had decreased to 2%. The main components of the residue at the 1st intermediate were ODM 5.1%, 1.3 mg/kg, and M01 3.7%, 1.0 mg/kg, but by the 2nd intermediate ODM and M01 could not be detected, and the major components were M07 (4.7% leaf radioactivity, 0.758 mg/kg) and P4 (4.8% leaf radioactivity, 1.3 mg/kg). By harvest these had decreased to 1.5% (0.3 mg/kg) and 0.5% of leaf radioactivity (0.105 mg/kg).

Analysis of pulp and peel showed ODM in the 1st and 2nd intermediate samples but not at harvest. However the 1st and 2nd intermediate samples contained M07, P2 and P3. In the 1st intermediate sample radioactivity in the pulp accounted for 17% of the radioactivity in the fruit (0.2 mg/kg) and by the 2nd 10% (0.05 mg/kg), but at harvest no components were detected.

In the peel extracts from the 1st intermediate sample, polar metabolites accounted for 3% of the radioactivity in the fruit (0.023 mg/kg) and this increased to 4% (0.026 mg/kg) at the 2nd intermediate. No components were detected at harvest. The major components detected at the 1st intermediate sampling in the pulp and peel together were ODM (26.6% of the radioactivity in the fruit, 0.245 mg/kg) and M01 (2.8% of the radioactivity in the fruit, 0.026 mg/kg). However by the 2nd intermediate sample ODM (1.7% radioactivity in the fruit, 0.012 mg/kg) and M01 (0.2%, 0.001 mg/kg) were detected only in the peel. The main component in the pulp was P4 (14.1%, 0.072 mg/kg). No compounds were identified at harvest in either the pulp or peel owing to the low levels of radioactivity in the extracted samples (<0.001 mg/kg). The results are shown in Tables 1-5.

Table 1. Concentration of total radioactive residue (mg/kg as ODM) in apples and leaves after the application of [^{14}C]oxydemeton-methyl.

Sample	Time				
	Application 1	Application 2	1st intermediate	2nd intermediate	Harvest
Fruit	-	-	0.918	0.574	0.134
Leaves	324.8	144.2	27.19	18.89	20.41

Table 2. Radioactivity in apples after the application of [^{14}C]oxydemeton-methyl.

Sample		^{14}C, % of total in fruit & (mg/kg as ODM)		
		1st intermediate	2nd intermediate	Harvest
Surface wash		4.25 (0.042)	5.57 (0.028)	ND
Peel extracts	Acetonitrile	7.57 (0.065)	8.07 (0.055)	8.59 (0.009)
	Cellulase	1.04 (0.009)	1.33 (0.007)	2.00 (0.003)
	1M HCl	0.44 (0.004)	0.58 (0.002)	0.95 (0.001)
	6M HCl	0.26 (0.002)	0.58 (0.002)	0.51 (0.001)
	Total peel extract	9.31 (0.080)	10.56 (0.065)	12.05 (0.013)
	Unextractable residue	5.26 (0.041)	7.29 (0.046)	6.15 (0.009)
Pulp extracts	Acetonitrile	47.29 (0.441)	39.99 (0.205)	22.62 (0.024)
	Cellulase	6.45 (0.057)	12.06 (0.064)	7.27 (0.009)
	1M HCl	3.73 (0.034)	7.67 (0.052)	2.90 (0.004)
	6M HCl	1.88 (0.017)	2.37 (0.015)	0.97 (0.001)
	Total pulp extract	59.35 (0.549)	62.09 (0.335)	33.77 (0.038)
	Unextractable residue	21.84 (0.201)	11.03 (0.081)	34.94 (0.057)

Sample	^{14}C, % of total in fruit & (mg/kg as ODM)		
	1st intermediate	2nd intermediate	Harvest
Juice	NS	3.47 (0.020)	13.09 (0.018)
Total fruit	(0.918)	(0.574)	(0.134)

Table 3. Radioactivity in leaves of apple trees after the application of [^{14}C]oxydemeton-methyl.

Sample		^{14}C, % of total in leaves & (mg/kg as ODM)		
		1st intermediate	2nd intermediate	Harvest
Surface wash		13.42 (3.44)	11.03 (2.02)	1.92 (0.39)
Extracts	Acetonitrile	3.54 (0.80)	10.37 (1.60)	4.26 (0.77)
	Cellulase	11.43 (2.40)	27.05 (5.72)	ND
	1M HCl	1.89 (0.38)	8.15 (1.56)	ND
	6M HCl	0.32 (0.06)	2.10 (0.41)	ND
	Total extract	13.77 (3.65)	47.68 (9.28)	4.26 (0.77)
Unextractable residue		72.82 (20.10)	41.29 (7.60)	93.8 (19.25)
Total leaf		(27.19)	(18.89)	(20.41)

Table 4. Radioactive components in wash and acetonitrile extracts of leaves after the application of [^{14}C]oxydemeton-methyl. [1]

Sample		^{14}C, % of total in leaves & (mg/kg as ODM)						
		M07	P2	P3	P4	ODM	M01	Others
1st Intermediate	Wash	0.9 (0.241)	ND (ND)	2.3 (0.584)	ND (ND)	4.1 (1.06)	3.0 (0.78)	3.0 (0.775)
	Extract	0.3 (0.071)	ND (ND)	0.8 (0.187)	ND (ND)	1.0 (0.219)	0.7 (0.167)	0.7 (0.162)
2nd Intermediate	Wash	1.3 (0.238)	ND (ND)	2.2 (0.411)	3.8 (0.700)	ND (ND)	ND (ND)	3.7 (0.760)
	Extract	3.4 (0.520)	ND (ND)	2.0 (0.310)	1.0 (0.601)	ND (ND)	ND (ND)	4.0 (0.612)
Harvest	Wash	0.2 (0.045)	ND (ND)	0.4 (0.085)	0.5 (0.105)	0.1 (0.030)	ND (ND)	0.6 (0.127)
	Extract	1.3 (0.238)	1.8 (0.323)	0.2 (0.043)	ND (ND)	ND (ND)	ND (ND)	0.9 (0.164)

Table 5. Proportion of radioactive components in acetonitrile extracts of apples after the application of [^{14}C]oxydemeton-methyl.[1]

Sample		^{14}C, % of total in fruit & (mg/kg as ODM)						
		M07	P2	P3	P4	ODM	M01	Others
1st Intermediate	Pulp Extract	2.8 (0.026)	9.7 (0.091)	4.3 (0.040)	ND (ND)	23.0 (0.214)	2.4 (0.022)	5.2 (0.048)
	Peel Extract	0.5 (0.005)	1.4 (0.012)	0.7 (0.006)	ND (ND)	3.6 (0.031)	0.4 (0.004)	1.0 (0.008)

Sample		^{14}C, % of total in fruit & (mg/kg as ODM)						
		M07	P2	P3	P4	ODM	M01	Others
2nd Intermediate	Pulp Extracts	7.9 (0.041)	ND (ND)	2.3 (0.012)	14.1 (0.072)	ND (ND)	ND (ND)	15.7 (0.081)
	Peel Extracts	1.2 (0.008)	2.6 (0.018)	ND (ND)	ND (ND)	1.7 (0.012)	0.2 (0.001)	2.4 (0.016)
Harvest	Pulp Extracts	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)
	Peel Extracts	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND (ND)

Environmental fate in soil

Aerobic degradation

Three soils, sandy loam (Laacherhof AXXa), silt loam (Laacherhof AIII) and silt (Hoefchen am Hohenseh), were stored for a maximum of 11 days in the dark at 20°C (Babczinski, 2001a). The soil characteristics are shown in Table 6.

The soil moisture corresponded to 40% of maximum water holding capacity in the three soils. [Ethylene-1-^{14}C]oxydemeton-methyl was applied at a nominal rate of 0.67 mg/kg dry soil, equivalent to the proposed single maximum annual rate of 250 g ai/ha for 2.5 cm soil depth. The experiment was conducted in accordance with EC/SETAC/OECD guidelines. Erlenmeyer flasks, attached with traps for collection of CO_2 and volatile organics, were incubated and sampled after approximately 2 h and 1, 2, 3, 4, 7 and 11 days and in a supplementary experiment after 0.1, 1, 2, 3, 4, 5, 6 and 24 h without traps for volatiles.

The samples were extracted four times with methanol, followed by a mixture of methanol and water (1:1). Analysis was by reverse-phase TLC, and components were characterized by co-chromatography with authentic reference standards and LC/MS and LC/MS/MS. During the study the total recoveries of radioactivity in individual test vessels ranged from 90.8% to 100%. In the three soils the DT_{50} and DT_{90} ranged from 0.17 to 0.22 and 0.58 to 0.74 days respectively. Furthermore the results indicated that the main metabolites were continuously degraded, that no product accumulated toward the end of the study, and unextracted residues were participating in the natural carbon cycle of soil.

Analysis of soil extracts showed two major and one less important degradation product (about 10% of the applied radioactivity (AR)) throughout the study. M09 reached a maximum on day 1 in all soils and was highest at 26.7% of the AR in Laacherhof AIII. It decreased thereafter, in the more active soils to <LOQ by day 11. M10, an oxidation product of M09, reached its maximum on day 3 in all soils and was highest at 16.8% of the AR in Laacherhof AXXa. It decreased towards the end of the study in all three soils, in the most active to almost <LOQ by day 11.

M05 reached its peak on day 4 (9.5% of the AR) in all soils and decreased thereafter. All the other detected individual ^{14}C-zones corresponded to ≤2.1% of the AR throughout the study. The total radioactivity at the TLC origin was ≤4.9% of the AR in all cases.

Bound residues in the three soils reached a maximum level at day 11 of about 50% of the AR, and all soils showed a high mineralization capacity yielding up to >30% of $^{14}CO_2$ on day 11.

The half-life in soil is expected to be about <1 day, regardless of the type of soil. The major products were further degraded and therefore would not accumulate in the soil. The results are shown in Tables 7-11.

Table 6. Characteristics of soil used for degradation experiment.

Designation	Type	Textural analysis (USDA) (%)			Org. C (%)	Org. (%) matter	Cation ex. cap. (meq/100g soil)	pH Water
		2000-50 µm	50-2 µm	<2 µm				
Laacherhof AXXa	Sandy loam	72.4	22.6	5.0	1.02	1.75	8	7.2
Laacherhof AIII	Silt loam	36.9	51.1	12.0	0.83	1.43	8	7.4
Hoefchen am Hohenseh 4a	Silt	8.5	81.3	10.2	1.55 - 2.11	2.67 - 3.63	15	7.3 - 7.6

Table 7. Distribution of radioactivity after the application of [^{14}C]oxydemeton-methyl to three soils, % of applied radioactivity.

Soil	Distribution		Interval						
			0.2 hr	1 day	2 days	3 days	4 days	7 days	11 days
Laacherhof AXXa	Volatiles	Soda lime	-	3.2	5.7	7.8	11.0	19.2	24.6
		PU Foam	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.4
		Total	-	3.2	5.7	7.8	11.0	19.2	25.1
	Extractable	Org. extract	49.7	23.7	22.7	21.6	20.2	16.0	9.9
		Water extract	18.4	20.0	17.9	16.6	14.7	10.2	5.0
		Total	68.1	43.7	40.6	38.1	34.9	26.2	14.9
	Unextr.	Soil	30.5	46.4	46.0	47.0	46.7	45.5	50.5
		Filter	1.4	1.1	0.8	0.9	0.8	0.8	0.4
		Total	31.9	47.5	46.9	47.9	47.6	46.3	50.9
	Total		100.0	94.4	93.2	93.9	93.5	91.7	90.9
Laacherhof AIII	Volatiles	Soda lime	-	1.1	2.6	4.8	6.6	12.8	19.8
		PU Foam	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.4
		Total	-	1.1	2.6	4.8	6.6	12.8	20.2
	Extractable	Org. extract	56.2	18.9	16.6	15.0	13.6	10.6	7.8
		Water extract	24.9	26.6	29.3	26.1	24.8	18.8	11.9
		Total	81.1	48.5	45.8	41.0	38.4	29.4	19.7
	Unextr.	Soil	17.4	45.3	46.9	47.9	48.4	48.0	51.3
		Filter	1.5	1.0	0.9	0.8	0.7	0.6	0.4
		Total	18.9	46.3	47.7	48.6	49.0	48.6	51.7
	Total		100.0	95.9	96.2	94.4	94.0	90.8	91.7
Hoefchen am Hohenseh 4a	Volatiles	Soda lime	-	3.8	7.4	11.3	15.0	27.1	31.6
		PU Foam	-	<0.1	<0.1	<0.1	<0.1	<0.1	0.5
		Total	-	3.8	7.4	11.3	15.0	27.1	32.1
	Extractable	Org. extract	59.7	25.7	22.9	20.8	17.8	11.7	7.4

Soil	Distribution		Interval						
			0.2 hr	1 day	2 days	3 days	4 days	7 days	11 days
		Water extract	16.7	16.3	14.3	12.2	10.2	6.3	3.1
		Total	76.5	42.0	37.2	33.0	28.1	18.0	10.5
	Unextr.	Soil	22.3	50.3	50.0	52.2	51.0	50.6	51.2
		Filter	1.2	1.2	0.8	0.8	0.6	0.4	0.2
		Total	23.5	51.6	50.8	53.0	51.5	51.0	51.5
	Total		100.0	97.4	95.4	97.3	94.6	96.0	94.0

Table 8. Distribution of radioactivity (% of the AR) after the application of [^{14}C]oxydemeton-methyl to Laacherhof AIII soil in the supplementary study.

Distribution		Interval (h)							
		0	1	2	3	4	5	6	24
Extractable	Org. extract	67.6	76.2	72.2	73.7	73.8	69.1	64.5	45.5
	Water extract	26.0	19.1	18.2	18.7	19.4	18.0	19.7	22.0
	Total	93.6	95.3	90.4	92.4	93.2	87.2	84.3	67.5

Table 9. Distribution of oxydemeton-methyl and degradation products after the application of [^{14}C]oxydemeton-methyl to three soils.

Time (days)	% applied radioactivity										
	Total extd.	Origin	ODM	M05	ROI 2	M10	M09	ROI 5	ROI 6	ROI 7	Diffuse
Laacherhof AXXa											
0	68.1	2.5	39.1	3.1	1.7	4.9	12.4	0.2	0.1	<0.1	4.1
1	43.7	1.7	1.7	8.4	<0.1	13.9	15.0	<0.1	<0.1	1.0	1.9
2	40.6	2.5	0.8	8.6	0.2	15.9	9.6	<0.1	<0.1	1.4	1.7
3	38.1	1.5	0.7	9.3	<0.1	16.8	6.3	<0.1	<0.1	1.8	1.7
4	34.9	1.6	0.5	9.5	<0.1	16.7	4.0	<0.1	<0.1	2.0	0.7
7	26.2	2.1	0.5	8.4	<0.1	11.1	<0.1	<0.1	<0.1	2.1	2.1
11	14.9	1.6	0.4	6.7	<0.1	3.7	<0.1	<0.1	<0.1	1.5	1.1
Laacherhof AIII											
0	81.1	1.6	70.9	<0.1	0.9	0.8	4.6	<0.1	<0.1	<0.1	2.3
1	48.5	3.7	2.3	2.8	0.2	7.9	26.7	<0.1	<0.1	<0.1	4.9
2	45.8	4.4	1.3	3.0	1.0	8.4	23.7	<0.1	<0.1	0.3	3.7
3	41.0	1.9	0.7	3.4	<0.1	8.9	22.8	<0.1	<0.1	0.4	2.8
4	38.4	1.9	0.4	3.5	<0.1	8.3	22.4	<0.1	<0.1	0.5	1.4
7	29.4	3.2	0.3	2.5	<0.1	2.2	17.2	<0.1	<0.1	0.6	3.3
11	19.7	2.7	0.3	1.5	<0.1	3.0	9.4	<0.1	<0.1	0.4	2.4
Hoefchen am Hohenseh 4a											
0	76.5	1.6	60.4	0.5	1.3	1.9	7.9	<0.1	<0.1	<0.1	2.7
1	42.0	1.7	1.1	7.0	<0.1	13.0	15.7	<0.1	<0.1	0.9	2.6
2	37.2	1.8	0.6	7.3	0.3	15.2	8.6	<0.1	<0.1	1.4	1.9

Time (days)	% applied radioactivity										
	Total extd.	Origin	ODM	M05	ROI 2	M10	M09	ROI 5	ROI 6	ROI 7	Diffuse
3	33.0	1.2	0.4	7.7	<0.1	15.2	5.2	<0.1	<0.1	1.4	1.8
4	28.1	1.0	0.3	8.2	<0.1	13.6	2.7	<0.1	<0.1	1.4	1.0
7	18.0	1.8	0.3	7.2	<0.1	5.4	<0.1	<0.1	<0.1	1.3	2.0
11	10.5	0.6	0.2	7.4	<0.1	0.8	<0.1	<0.1	<0.1	1.0	0.4

Table 10. Distribution of oxydemeton-methyl and degradation products after the application of [^{14}C]oxydemeton-methyl to Laacherhof AIII soil in supplementary study.

Time (h)	% applied radioactivity									
	Total extd.	Origin	ODM	M05	ROI 2	M10	M09	ROI 6	ROI 7	Diffuse
0	93.6	0.6	92.7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.4
1	95.3	0.8	92.1	<0.1	<0.1	<0.1	0.4	<0.1	<0.1	2.0
2	90.4	0.5	88.8	<0.1	<0.1	<0.1	0.2	<0.1	<0.1	0.9
3	92.4	0.5	87.0	<0.1	0.2	<0.1	0.4	<0.1	<0.1	4.2
4	93.2	1.0	87.7	<0.1	0.3	<0.1	1.2	<0.1	0.4	2.6
5	87.2	1.1	78.5	<0.1	0.6	1.8	0.9	<0.1	0.4	3.8
6	84.3	1.5	74.3	<0.1	0.8	<0.1	2.9	<0.1	0.7	4.0
24	67.5	2.2	40.0	0.6	1.0	<0.1	16.6	0.2	1.1	6.0

Table 11. Degradation parameters of oxydemeton-methyl in three soils.

Parameter	Laacherhof AXXa	Laacherhof AIII	Hoefchen am Hohenseh 4a
Main study			
K (1/day)	3.10	3.39	3.99
DT50 (days)	0.22	0.20	0.17
DT_{75} (days)	0.45	0.41	0.35
DT_{90} (days)	0.74	0.68	0.58
R^2	0.999	0.999	1.000
Supplementary study			
K (1/h)	-	0.036	-
DT_{50} (h)	-	19.4	-
DT_{75} (h)	-	38.8	-
DT_{90} (h)	-	64.4	-
R^2	-	0.980	-

Environmental fate in water/sediment systems

Hydrolysis

The hydrolysis of oxydemeton-methyl was studied in sterile 0.01M buffer solutions adjusted to pH 4, 7 and 9, stored for a maximum of 31 days in the dark at two temperatures (Babczinski, 2001b). The

experiment was carried out in compliance with GLP standards and EPA/SETAC/OECD/EC Guidelines.

The test solution was prepared with [ethylene-1-^{14}C]oxydemeton-methyl at a concentration of about 5 mg/l. A pre-test solution was incubated for 7 days under sterile conditions in the dark at 50°C with sampling at 0, 2.5 and 6 h and 1, 2 and 7 days, and in the main test the solutions at pH 4 and pH 7 were incubated for 31 days under sterile conditions in the dark at 25°C with sampling intervals of 0, 5, 11, 14, 29, 26 and 31 days deduced from the results of the pre-test. At pH 9, samples were taken after 0, 0.25, 1, 1.25, 2, 2.25, 3 and 6 days.

In the pre-test at 50°C and in the main-test at 25°C, ODM was unstable at pH 4, 7 and 9 and considerable degradation occurred. The compound was hydrolysed to M06 (maximum 20.4% of the AR) and, at pH 7 and 9, M21 (maximum 9.0% of the AR) by cleaving the P-S bond. The sulfonic acid M10 was tentatively identified as an oxidized P-S cleavage product at low percentages (maximum 2.2% of the AR).

M21 underwent dimerization to M11 (maximum 74.2% AR). This reaction is unlikely to happen in the environment since exposure would be expected to be at a significantly lower level. Realistically, this degradation product should by calculation be added to M21 making this compound the main hydrolytic degradation product at pH ≥7. Finally, desdimethyl-ODM was identified at low concentrations at 50°C and pH 9 but not quantified.

At 50°C, first-order half-lives for ODM were estimated to be 4.9, 3.5 and 0.2 days at pH 4, 7 and 9 at 25°C, 91, 42 and 2.5 days. Half-lives at 20°C were calculated from Arrhenius plots (1/T versus In k) as 174, 73 and 4.5 days. These results indicate that hydrolytic processes contribute to the degradation of ODM in the environment. The results are shown in Tables 12-15.

Table 12. Half-lives for the hydrolysis of oxydemeton-methyl in sterile aqueous solutions at 50°C (pre-test).

Solution	DT_{50} (days)
pH 4 (0.01M acetate buffer)	4.9
pH 7 (0.01M TRIS buffer)	3.5
pH 9 (0.01M borate buffer)	0.2

Table 13. Half-lives, DT_{75}s and DT_{90}s for the hydrolysis of oxydemeton-methyl in sterile aqueous solutions at 25°C.

Solution	DT_{50} (days)	DT_{75} (days)	DT_{90} (days)
pH 4 (0.01M acetate buffer)	91	182	303
pH 7 (0.01M TRIS buffer)	42	85	141
pH 9 (0.01M borate buffer)	2.5	4.9	8.2

Table 14. Half-lives and DT_{90}s for the hydrolysis of oxydemeton-methyl in aqueous solution at 20°C (calculated from experimental data at 25°C).

Solution	DT_{50} (days)	DT_{90} (days)
pH 4 (0.01M acetate buffer)	174	577
pH 7 (0.01M TRIS buffer)	73	243
pH 9 (0.01M borate buffer)	4.5	15

Table 15. Distribution of oxydemeton-methyl and degradation products (% of the AR) during the hydrolysis of oxydemeton-methyl in sterile aqueous buffer solutions at 25°C.

Solution	Interval (days)	Total radioactivity (% of applied)	ODM	M06	M11	M10	M21	Others
0.5 m g/l buffer pH 4	0	100.0	100.0	-	-	-	-	-
	5	100.3	96.3	3.4	-	-	-	0.6
	11	95.2	87.7	6.7	-	-	-	0.7
	14	96.7	87.3	8.4	-	-	-	0.9
	20	98.9	85.7	11.9	-	-	-	1.2
	26	98.3	81.9	15.1	-	-	-	1.1
	31	97.5	78.5	16.7	-	-	-	2.2
0.5 m g/l buffer pH 7	0	100.0	99.7	0.3	-	-	-	-
	5	104.0	96.7	4.6	2.4	0.3	-	0.1
	11	100.8	84.7	6.0	7.5	1.0	0.1	1.5
	14	99.9	80.4	6.2	9.2	1.3	0.3	2.5
	20	100.7	70.8	9.1	10.7	1.4	0.2	8.6
	26	102.5	68.8	20.4	11.2	1.2	-	0.9
	31	101.1	60.3	11.9	16.5	2.2	0.4	9.6
0.5 m g/l buffer pH 9	0	100.0	98.7	-	0.7	-	0.5	-
	0.25	101.5	95.6	0.3	2.1	0.2	2.8	0.6
	1	101.9	79.2	0.6	13.5	0.4	7.4	0.8
	1.25	101.6	72.8	1.1	16.9	0.5	8.9	1.5
	2	101.7	57.7	1.4	31.0	0.4	9.0	2.2
	2.25	104.2	54.1	1.6	37.3	0.4	6.8	3.9
	3	102.3	42.6	1.7	47.8	0.3	4.8	5.1
	6	101.8	16.8	2.4	74.2	0.2	0.5	7.6

Photolysis

The quantum yield of direct photodegradation of oxydemeton-methyl in water was determined according to the ECETOC method in polychromatic light (Hellpointner *et al.*, 1992). From the UV absorption data and the kinetic result of photodegradation experiments in a merry-go round irradiation apparatus the quantum yield was calculated to be 0.00078. The quantum yield and UV absorption data in aqueous solution were used to estimate the environmental half-life of ODM during photodegradation in water by two different simulation models. The results are shown in Tables 16 and 17.

Table 16. Calculated half-life of oxydemeton-methyl in water (GC-Solar program).

Season	Environmental half-life (days) at degrees latitude			
	30	40	50	60
Spring	112	117	126	143
Summer	112	117	126	143
Autumn	188	260	428	909
Winter	274	476	1110	4010

Table 17. Calculated half-life of oxydemeton-methyl in water (Frank-Klöpffer program).

Month	Photolysis constant	Environmental half-life (days)		
		Minimum	Mean	Maximum
March	0.157 10E-7	270	510	2100
April	0.291 10E-7	150	280	1100
May	0.393 10E-7	130	200	820
June	0.451 10E-7	120	180	710
July	0.401 10E-7	130	200	670
August	0.378 10E-7	140	210	710
September	0.208 10E-7	230	390	1400
October	0.101 10E-7	420	790	3600

RESIDUE ANALYSIS

Analytical methods

Many of the methods developed for the determination of residues of oxydemeton-methyl in various samples were reviewed at the 1998 Meeting under the Periodic Review Programme. The present Meeting received supplementary information on more recent methods. These still depended on oxidising ODM to demeton-S-methylsulphon as analyte.

Method 00585 (Blass *et al.*, 2001a)
This method is used to determine residues of oxydemeton-methyl, demeton-S-methyl and demeton-S-methylsulphon after oxidation to demeton-S-methylsulphon in plant materials. Oxydemeton-methyl and the other two compounds are extracted and oxidized in one step with aqueous acidic potassium permanganate solution using a microwave. Clean-up on an Extrelut column (preceded by hexane partitioning for brassica) is followed by partition against dichloromethane. The dichloromethane extract is evaporated to dryness and re-dissolved in acetone before quantification by GC-FPD. Recoveries from plant samples fortified at 0.005 to 1.0 mg/kg ranged from 70 to 110% with an RSD of ≤20%. The limit of quantification was 0.005 mg/kg for apples, potatoes and grapes and 0.01 mg/kg for Brussels sprouts.

Method 00585/M001 (Blass *et al.,* 2001)
This modification permits analysis of low-water content crops such as wheat grain, forage and straw by the addition of water and Celite before oxidation. Recoveries were within the 70 to 110% range with an RSD of ≤20% from samples of grain and straw fortified at 0.02-2.0 and 0.05-5.0 mg/kg respectively and the limits of quantification were 0.02 and 0.05 mg/kg.

Methods 00255/E001 and 00255/E002 (Seym, 1994, 1995)
Method 00255 (Ohs, 1992) was described in the 1998 Residue Evaluation. These supplements consist of validation data for the determination of residues in cauliflower, corn and sunflower (E001) and cauliflower (E002). They evaluate the sensitivity of the detector used in the quantification and confirm the limit of quantification. In E001 mean recoveries ranged from 70 to 108% after fortification with 0.01 and 0.04 mg/kg of oxydemeton-methyl and the overall mean was 89% with an RSD of 9%. In E002 recoveriesranged from 89 to 97% from cauliflower after fortification at 0.01 and 0.1 mg/kg with individual values of 85-97%. The mean recovery was 93% with an RSD of 6%. The limit of quantification for all samples was 0.01 mg/kg.

Method 00255/E004 and 00255/E005 (Seym, 1996)

These consist of a minor variation to the basic method for the determination of residues in head cabbage (E004), and Savoy and red cabbage (E005) in that the sample size is reduced to 50 g and the extraction solvent volume to 150 ml. The rest of method is unchanged. Mean recoveries from head cabbage after fortification at 0.01-0.1 mg/kg were 80-90% with an RSD of 7.0%, and from Savoy and red cabbage 73-98% and 89-92% respectively at the same fortification levels with individual recoveries ranging from 69 to 109%. The limit of quantification was 0.01 mg/kg.

Method 00255/E008 and 00255/E009 (Schoning, 1998)
In this variation to the basic method for the determination of residues in apples, pears, grapes and Brussels sprouts (E008) and wheat and barley grain (E009) the sample size is again reduced to 50 g and the extraction solvent to 150 ml. For dry samples (E009) water is added during extraction, and the extract is filtered. The rest of method is unchanged. In E008 mean recoveries at each fortification level from the four crops were 96-101% with an RSD of 0.8 to 8.1% at fortification levels of 0.01-0.1 mg/kg, and in E009 83-106% from cereal grain at levels of 0.01-0.1 mg/kg with an RSD of 9.3 to 12.9%. The limit of quantification for both modifications was 0.01 mg/kg.

Method 00255/E012 (Schoning, 2001)
In this minor variation for the determination of residues in rape foliage and seed 50 g samples are extracted with 150 ml of acetone and filtered. After filtration the extract is filtered through Celite which is washed with acetone/water (2:1) and the filtered extracts partitioned three times with dichloromethane. The remainder of the method is unchanged. Mean recoveries from foliage and seed were 80-83% and 74-79% respectively at fortification levels of 0.01-0.1 mg/kg with individual recoveries ranging from 70 to 88%. The limit of quantification was 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

The storage stability of oxydemeton-methyl in cabbage, maize, lettuce and papaya was briefly reported to the 1998 JMPR, which concluded that data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton-methyl were highly desirable as the information available was unrepresentative of the various crop groups, did not cover extended storage intervals, and suggested variable storage stability. The manufacturer reported a new study to the present Meeting.

The study was conducted to determine the storage stability of oxydemeton-methyl in spiked commercial samples of apple, dried peas, potato and oilseed rape (meal and oil) treated with formulated oxydemeton-methyl and stored at -20°C (Smith, 2002). Apple and potato samples were finely chopped in an industrial food processor (the preparation method for dried peas was not reported). Twenty g of samples of each crop were spiked with oxydemeton-methyl at nominal concentrations of 0.1, 1.0 and 10 mg/kg and stored at -20°C. Two samples from each treatment were analysed after 0, 3, 6, 12 and 24 months' storage. The analytical method (Thornton *et al.,* 1977) was validated for each crop at day 0, but after three months a modified procedure (Hill *et al.,* 1994) was used to improve the oxidation/extraction phase. The results are shown in Table 18.

The results do not show any substantial loss of residue over 24 months' storage in apple, potato or oil, but about half the residue was lost from dried peas and meal in 6 and 3 months respectively. Control samples contained ODM (apple <0.005-0.022 mg/kg, dried peas <0.005-0.016 mg/kg, potatoes <0.01-0.016 mg/kg, rape meal 0.013-0.03 and oil <0.005-0.011 mg/kg). The Meeting concluded that the storage stability data were inadequate and maintained the former requirement.

Table 18. Analyses of stored spiked samples.

Crop	Spiking level (mg/kg)	Mean oxydemeton-methyl content, mg/kg[1], and (% of initial value)			
		3 months	6 months	12 months	24 months
Apple	0.101 1.01 10.1	0.094 (92.5) 1.045 (94.0) 9.325 (92.5)	0.095 (94.5) 0.836 (82.5) 8.81 (87.5)	0.113 (111.5) 0.868 (86.0) 7.96 (79.0)	0.097 (96.0) 0.971 (95.5) 8.365 (82.5)
Dries peas	0.101 1.01 10.1	0.114 (113.5) 0.811 (80.0) 7.405 (73.5)	0.0648 (64.0) 0.620 (61.5) 5.535 (54.5)	0.051 (51.0) 0.438 (43.0) 5.80 (57.0)	0.067 (51.5) 0.473 (45.5) 4.63 (46.0)
Potato	0.101 1.01 10.1	0.029 (29.0) 0.625 (61.5) 9.81 (95.5)	0.083 (82.0) 0.786 (77.5) 8.455 (84.0)	0.071 (70.0) 0.744 (74.0) 7.000 (69.5)	0.0775 (76.5) 0.728 (72.5) 7.865 (77.5)
Oilseed rape (meal)	0.101 1.01 10.1	0.047 (47.0) 0.51 (50.5) 6.715 (66.5)	0.047 (46.5) 0.655 (64.5) 5.565 (55.0)	0.037 (36.5) 0.608 (60.0) 5.96 (59.0)	0.0774 (47.0) 0.529 (49.5) 5.325 (52.5)
Oilseed rape (oil)	0.101 1.01 10.1	0.010 (99.0) 0.913 (90.5) 8.815 (87.5)	0.081 (80.0) 0.804 (79.5) 7.815 (77.5)	0.080 (79.5) 0.918 (91.0) 8.355 (83.0)	0.089 (88.5) 0.861 (81.0) 8.525 (84.5)

[1] Corrected for apparent residue in control samples

USE PATTERN

Product labels from Europe were submitted to the Meeting together with translations into English.

Table 19. Registered uses of oxydemeton-methyl.

Crop	Country	Formulation	Dose (formulation)	Dose (ai) (kg/hl or kg/ha)	PHI (days)	No. of applns.
almond	Greece	500 g/l SL	0.10%	0.05	90	2
almond	Spain	250 g/l EC	0.10%	0.025	until petals fall	
apple	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
apple	Greece	500 g/l SL	0.10%	0.05	60	2
apple	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	90	
barley	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1
beans (horse)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	28	
beet	Spain	250 g/l EC	0.10%	0.025	30	
beet (fodder)	Austria	48.5 g/l EC	2 l/ha	0.097	35	4
beet (fodder)	Germany	265.29 g/l EC	0.6-0.8 l/ha	0.16-0.21	28	3
beet (sugar)	Germany	265.29 g/l EC	0.6-0.8 l/ha	0.16-0.21	28	3
beet (sugar)	Austria	48.5 g/l EC	2 l/ha	0.097	35	4
beet (sugar)	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	30	
broccoli	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cabbage	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cabbage (Chinese)	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cabbage (fodder)	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1

Crop	Country	Formulation	Dose (formulation)	Dose (ai) (kg/hl or kg/ha)	PHI (days)	No. of applns.
cabbage (green)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cabbage (red)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cabbage (Savoy)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cabbage (white)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
cauliflower	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
cauliflower	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
citrus fruit	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
citrus fruit	Greece	500 g/l SL	0.10%	0.05	90	2
citrus fruit	Spain	250 g/l EC	0.10%	0.025	90	
fruit trees	Finland	250 g/l EC	0.01-0.02%	0.0025-0.005	60 fruit size <20 mm	
grape vine	Germany	265.29 g/l EC	0.10%	0.027	up to inflorescences fully developed	1
kale	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
kohlrabi	Belgium	250 g/l EC	0.6 l/ha	0.15	28	1
kohlrabi	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
melon	Portugal	250 g/l EC	0.10%	0.025	before fruiting begins	
peach	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
pear	Portugal	250 g/l EC	0.20%	0.05	before fruiting begins	
pear	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	90	
plum	Spain	250 g/l EC	0.10%	0.025	until petals fall	
plum	Germany	265.29 g/l EC	0.5 l/ha	0.13	immediately after flowering	1
plum	Austria	48.5 g/l EC	0.50%	0.024	up to 2 weeks after flowering	1
pome fruit	Germany	265.29 g/l EC	0.5 l/ha	0.13	immediately after flowering	1
pome fruit	Austria	48.5 g/l EC	0.50%	0.024	up to 2 weeks after flowering	1
potato	Greece	500 g/l SL	0.10%	0.05	28	3
potato	Spain	250 g/l EC	0.10%	0.025	30	
potato	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	28	
rye	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1
salad crops (except chicory and lambs lettuce)	Germany	265.29 g/l EC	0.6 l/ha (<50cm) 0.9 l/ha (>50cm)	0.16 0.24	21	1
stone fruit	Spain	250 g/l EC	0.10%	0.025	until petals fall	

Crop	Country	Formulation	Dose (formulation)	Dose (ai) (kg/hl or kg/ha)	PHI (days)	No. of applns.
strawberries	Portugal	250 g/l EC	0.10%	0.025	before fruiting begins	
strawberries	Germany	265.29 g/l EC	2 l/ha 2000 l/ha (water)	0.53	after harvest	1
strawberries	Austria	48.5 g/l EC	0.50%	0.024	90	1
tomato	Portugal	250 g/l EC	0.10%	0.025	before fruiting begins	
triticale	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1
wheat	Portugal	250 g/l EC	0.1% 1 l/ha	0.025 0.25	until flowering	
wheat	Italy	188.7 g/l SL	0.12-0.15%	0.023-0.028	30	
wheat	Germany	265.29 g/l EC	0.5 l/ha	0.13	21	1

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised trials data on apples, pears, grapes, cabbages, Brussels sprouts, cauliflowers, field peas, potatoes, sugar beets, fodder beets, wheat, barley, rape and sunflower seeds were reported to the Meeting. Results are shown in Tables 20-33, where all residues are expressed as oxydemeton-methyl.

Table 20. Residues of oxydemeton-methyl and demeton-S-methylsulphon in apples after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)			
France (North) 1997	258EC	0.25-0.38	0.025	2 2nd fruit fall	29 44 59 74 89	0.08 0.04 0.02 0.02 0.01	RA-2157/97 0406-97
France (North) 1997	258EC	0.25-0.38	0.025	2 2nd fruit fall	30 45 60 75 90	0.08 0.05 0.04 0.02 0.02	RA-2157/97 0745-97
France (North) 1998	258EC	0.30-0.45	0.030	2 End of flowering	36 126	0.02 <u>≤0.01</u>	RA-2029/98 1361-98
France (South) 1997	258EC	0.25-0.38	0.025	2 20 mm fruit	45 60 74 91 109	0.01 <0.01 <0.01 <u>≤0.01</u> <0.01	RA-2156/97 0407-97
Italy 1997	250EC	0.25-0.44	0.025 0.029	2 20 mm fruit	30 45 60 75 90	0.10 0.06 0.03 0.02 <u>0.01</u>	RA-2109/97 0424-97

Country (Area) Year	Application				PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)			
Italy 1997	250EC	0.25-0.38	0.025	2 20 mm fruit	30 45 60 75 87	0.02 <0.01 <0.01 <0.01 <u>≤0.01</u>	RA-2109/97 0425-97
Spain[2] 1997	250EC	0.25-0.38	0.025	2 10 mm fruit	29 42 60 76 89	0.08 0.04 <0.01 <0.01 <u>≤0.01</u>	RA-2109/97 0571-97
Spain[2] 1997	250EC	0.25-0.38	0.025	2 10 mm fruit	29 42 60 76 89	0.11 0.03 0.02 <0.01 <0.01	RA-2109/97 0572-97
France (South) 1997	258EC	0.25-0.38	0.025	2 10 mm fruit	121	<0.01	RA-2156/97 0742-97
France (South) 1998	258EC	0.30-0.45	0.030	2 10 mm fruit	63 76 93 107 114	<0.01 <0.01 <u>≤0.01</u> <0.01 <0.01	RA-2030/98 1037-98
Spain 1998	250EC	0.25-0.38	0.025	3 10 mm fruit	69 90 129	<0.01 <u>≤0.01</u> <0.01	RA-2031/98 1042-98
Portugal 1998	250EC	0.25-0.38	0.025	2 20 mm fruit	49 126	0.01 <0.01	RA-2031/98 1364-98
Italy 1998	250EC	0.25-0.38	0.025	2 Flowers fading	58 150	<u>≤0.01</u> <0.01	RA-2031/98 1365-98
France (South) 1998	250EC	0.25-0.38	0.025	2 10 mm fruit	35 90	0.04 <u>≤0.01</u>	RA-2031/98 1366-98
Spain 1998	250EC	0.25-0.38	0.025	2 20 mm fruit	61 138	<u>≤0.01</u> <0.01	RA-2031/98 1367-98

[1] Expressed as oxydemeton-methyl.
[2] Trials conducted in same location.

Table 21. Residues of oxydemeton-methyl and demeton-S-methylsulphon in pears after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1998	258EC	0.30-0.45	0.030	2 2nd fruit fall	Fruit	50 91	<0.01 <u>≤0.01</u>	RA-2029/98 1036-98
France (South) 1998	258EC	0.30-0.45	0.030	2 10 mm fruit	Fruit	48 104	<0.01 <u>≤0.01</u>	RA-2030/98 1362-98

[1] Expressed as oxydemeton-methyl.

Table 22. Residues of oxydemeton-methyl and demeton-S-methylsulphon in grapes after foliar applications of oxydemeton-methyl.

Country Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	For m	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1997 (Bacchus)	250 EC	0.2	0.025	1 9 or more leaves unfolded	Berry	113	<0.01	RA-2090/97 0516-97
					Bunch	113	≤0.01	
Germany 1997 (Mueller-Thurgau)	250 EC	0.2	0.025	1 9 or more leaves unfolded	Berry	115	<0.01	RA-2090/97 0578-97
					Bunch	115	≤0.01	
Germany 1997 (Portugieser)	250 EC	0.2	0.025	1 9 or more leaves unfolded	Berry	115	<0.01	RA-2090/97 0579-97
					Bunch	115	≤0.01	

[1] Expressed as oxydemeton-methyl.

Table 23. Residues of oxydemeton-methyl and demeton-S-methylsulphon in cabbages after foliar applications of oxydemeton-methyl.

Country (Area) Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/ Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (North) 1996 (Red cabbage)	258EC	0.15	0.054	1 (30% of the head size reached)	Head	0 7 10 14 21	0.03 <0.01 <0.01 <0.01 ≤0.01	RA-2150/96 0679-96
Germany 1996 (Savoy cabbage)	258EC	0.15	0.025	1 (30% of the head size reached)	Whole plant without roots	0	2.1	RA-2150/96 0676-96
					Head	21	≤0.01	
Germany 1996 (Savoy cabbage)	258EC	0.15	0.025	1 (30% of the head size reached)	Whole plant without roots	0	1.8	RA-2150/97 0677-97
					Head	21	≤0.01	
France (North) 1996 (White cabbage)	258EC	0.15	0.054	1 (70% of the head size reached)	Head	0 7 10 14 21	0.02 <0.01 <0.01 <0.01 ≤0.01	RA-2150/96 0678-96

[1] Expressed as oxydemeton-methyl.

Table 24. Residues of oxydemeton-methyl and demeton-S-methylsulphon in Brussels sprouts after foliar applications of oxydemeton-methyl.

Country (Area)	Application	Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.

	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1997	250EC	0.15	0.025	1 (First sprouts tightly closed)	Button	0 4 7 14 21 29	0.08 0.07 0.06 0.02 0.02 0.03	RA-2088/97 0419-97
Germany 1997	250EC	0.15	0.025	1 (First sprouts tightly closed)	Button	0 4 7 14 21 29	0.09 0.09 0.07 0.04 0.03 0.02	RA-2088/97 0420-97
Great Britain 1997	250EC	0.15	0.025	1 (50% of sprouts tightly closed)	Button	0 4 7 14 21 28	0.11 0.08 0.12 0.15 0.02 0.02	RA-2088/97 0574-97
Belgium 1997	250EC	0.15	0.025	1 (70% of sprouts tightly closed)	Button	0 4 7 14 21 28	0.01 <0.01 0.01 <0.01 <0.01 <0.01	RA-2088/97 0575-97
Belgium 1998	250EC	0.15	0.025	1 (Sprouts below terminal bud tightly closed)	Button	0 21	0.09 <0.01	RA-2118/98 1044-98
Germany 1998	250EC	0.15	0.025	1 (First sprouts tightly closed)	Button	0 21	0.18 <0.01	RA-2118/98 1513-98
France (North) 1998	250EC	0.15	0.025	1 (Sprouts below terminal bud tightly closed)	Button	0 21	0.02 <0.01	RA-2118/98 1514-98
Great Britain 1998	250EC	0.16	0.025	1 (First sprouts tightly closed)	Button	0 21	0.24 <0.01	RA-2118/98 1515-98
Germany 1998	250EC	0.15	0.025	1 (50% of sprouts tightly closed)	Button	0 21	0.07 <0.01	RA-2118/98 1516-98

[1] Expressed as oxydemeton-methyl.

Table 25. Residues of oxydemeton-methyl and demeton-S-methylsulphon in cauliflower after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1996	258EC	0.15	0.025	1 (Head beginning	Whole plant without roots	0	3.2	RA-2152/96 0669-96

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
				to form)	Head	7 14 21 28	<0.01 <0.01 ≤0.01 <0.01	
Germany 1996	258EC	0.15	0.025	1 (Head beginning to form)	Whole plant without roots	0	3	RA-2152/96 0670-96
					Head	7 14 21 28	<0.01 <0.01 ≤0.01 <0.01	
Great Britain 1996	258EC	0.15	0.025	1 (Bud development)	Whole plant without roots	0	1.1	RA-2152/96 0671-96
					Head	21	≤0.01	
France (North) 1996	258EC	0.38	0.063	1 (Head beginning to form)	Whole plant without roots	0	4.8	RA-2152/96 0672-96
					Head	21	≤0.01	
France (South) 1997	258EC	0.15	0.054	1 (30% of head diameter reached)	Whole plant without roots	0	0.84	RA-2153/96 0673-96
					Head	7 10 14 21	<0.01 <0.01 <0.01 ≤0.01	
France (South) 1996	258EC	0.15	0.054	1 (Head beginning to form)	Whole plant without roots	0	1.2	RA-2153/96 0675-96
					Head	7 10 14 21	<0.01 <0.01 <0.01 ≤0.01	
France (South) 1997	258EC	0.15	0.025	1 (Head beginning to form)	Whole plant without roots	0	1.4	RA-2160/97 0409-97
					Head	21	≤0.01	
France (South) 1997	258EC	0.16	0.025	1 (30% of the head diameter reached)	Whole plant without roots	0	1.4	RA-2160/97 0750-97
					Head	21	≤0.01	

[1] Expressed as oxydemeton-methyl.

Table 26. Residues of oxydemeton-methyl and demeton-S-methylsulphon in field peas after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg) [1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South)	258EC	0.10-0.11	0.033-0.034	2 (Pods have	Whole plant without roots	0 14	2.4 0.06	RA-2161/97 0408-97

1997				reached typical size)	Seed	28	<0.01	
					Plant, dried	28	<0.01	
France (South) 1997	258EC	0.10	0.033	2 (10% of pods ripe)	Whole plant without roots	0 14	1.1 0.13	RA-2161/97 0751-97
					Seed	28	<0.01	
					Plant, dried	28	0.05	

[1] Expressed as oxydemeton-methyl.

Table 27. Residues of oxydemeton-methyl and demeton-S-methylsulphon in potatoes after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (North) 1996	258EC	0.15	0.054	2 (End of flowering)	Tuber	0 21 28	<0.01 <0.01 <u><0.01</u>	RA-2148/96 0665-96
France (North) 1996	258EC	0.15	0.054	2 (End of flowering)	Tuber	0 21 28	<0.01 <0.01 <u><0.01</u>	RA-2148/96 0773-96
France (North) 1996	258EC	0.15	0.054	2 (End of flowering)	Tuber	0 21 28	<0.01 <0.01 <u><0.01</u>	RA-2148/96 0774-96
France (North) 1996	258EC	0.15	0.054	2 (30% of total final tuber mass reached)	Tuber	0 7 14 21 28	<0.01 <0.01 <0.01 <0.01 <u><0.01</u>	RA-2149/96 0666-96
France (North) 1996	258EC	0.15	0.050	2 (50% of total final tuber mass reached)	Tuber	0 7 14 21 28	<0.01 <0.01 <0.01 <0.01 <u><0.01</u>	RA-2149/96 0667-96
France (North) 1996	258EC	0.15	0.054	2 (50% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 <u><0.01</u>	RA-2149/96 0668-96
France (North) 1996	258EC	0.15	0.054	2 (Tuber initiation)	Tuber	0 21 28	<0.01 <0.01 <u><0.01</u>	RA-2149/96 0781-96
France (North) 1996	258EC	0.20-0.25	0.050-0.063	3 (30% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 <u><0.01</u>	RA-2147/96 0664-96
Germany 1996	258EC	0.20-0.25	0.067-0.083	3 (70% of total final tuber mass reached)	Tuber	0 0 7 14 21 28	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RA-2147/96 0661-96

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Germany 1996	258EC	0.20-0.25	0.067-0.083	3 (Maximum of total final tuber mass reached)	Tuber	0 0 6 13 20 27	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	RA-2147/96 0662-96
UK 1996	258EC	0.20-0.25	0.050-0.063	3 (60% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 <0.01	RA-2147/96 0663-96
Spain[2] 1997	250EC	0.30-0.32	0.050	3 (Berry ripening)	Tuber	-1 20 27	<0.01 <0.01 ≤0.01	RA-2087/97 0426-97
Spain[2] 1997	250EC	0.30-0.32	0.050	3 (Berry ripening)	Tuber	-1 20 27	<0.01 <0.01 <0.01	RA-2087/97 0428-97
Italy 1997	250EC	0.30	0.050	3 (30% of total final tuber mass reached)	Tuber	0 21 28	0.01 <0.01 ≤0.01	RA-2087/97 0695-97
Italy 1997	250EC	0.30	0.050	3 (30% of total final tuber mass reached)	Tuber	0 21 28	<0.01 <0.01 ≤0.01	RA-2087/97 0696-97
Italy 1998	250EC	0.30	0.050	3 (60% of total final tuber mass reached)	Tuber	21 28	<0.005 ≤0.005	RA-2116/98 1046-98
Greece 1998	250EC	0.30	0.050	3 (Development of fruit)	Tuber	22 28	0.007 ≤0.005	RA-2116/98 1507-98
Spain 1998	250EC	0.30-0.32	0.050	3 (10% of berries in first fructification have reached full size)	Tuber	21 28	<0.005 ≤0.005	RA-2116/98 1509-98
France (South) 1998	250EC	0.30	0.050	3 (Development of fruit)	Tuber	21 28	<0.005 ≤0.005	RA-2116/98 1510-98
Spain 1998	250EC	0.30	0.050	3 (40% of total final tuber mass reached)	Tuber	21 28	<0.005 ≤0.005	RA-2116/98 1512-98

[1] Expressed as oxydemeton-methyl.
[2] Trials conducted in same location.

Table 28. Residues of oxydemeton-methyl and demeton-S-methylsulphon in sugar beet after foliar applications of oxydemeton-methyl.

Country Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Spain[2] 1997	250EC	0.38	0.13	3 (Beetroot has reached harvestable size)	Leaf	0 6 14 28 35	4.0 0.42 <0.04 ≤0.04 <0.04	RA-2091/97 0580-97

Country Year (Variety)	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
					Beet	0 6 14 28 35	<0.01 <0.01 <0.01 ≤0.01 <0.01	
Spain[2] 1997	250EC	0.38	0.13	3 (Beetroot has reached harvestable size)	Leaf	0 6 14 28 35	3.9 0.27 <0.04 <0.04 <0.04	RA-2091/97 0581-97
					Beet	0 6 14 28 35	<0.01 <0.01 <0.01 <0.01 <0.01	
Italy 1997	250EC	0.13	0.042	3 (Fruit developing)	Leaf	0 7 14 28 35	2.4 0.11 <0.04 ≤0.04 <0.04	RA-2091/97 0421-97
					Beet	0 7 14 28 35	<0.01 <0.01 <0.01 ≤0.01 <0.01	
Italy 1997	250EC	0.13	0.042	3 (Fruit developing)	Leaf	0 7 14 28 35	1.7 0.04 <0.04 ≤0.04 <0.04	RA-2091/97 0423-97
					Beet	0 7 14 28 35	<0.01 <0.01 <0.01 ≤0.01 <0.01	
Italy 1998	250EC	0.38	0.13	3 (Root developing)	Leaf	0 28	4.1 ≤0.01	RA-2117/98 1045-98
					Beet	0 28	0.02 ≤0.01	
Italy 1998	250EC	0.38	0.13	3 (Root devloping)	Leaf	0 28	9.4 ≤0.01	RA-2117/98 1521-98
					Beet	0 28	0.01 ≤0.01	
Spain 1998	250EC	0.38	0.13	3 (Beetroot has reached harvestable size)	Leaf	0 28	4.0 ≤0.01	RA-2117/98 1522-98
					Beet	0 28	<0.01 ≤0.01	

[1] Expressed as oxydemeton-methyl.
[2] Trials conducted same location.

Table 29. Residues of oxydemeton-methyl and demeton-S-methylsulphon in fodder beet after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1998	250EC	0.36-0.40	0.13-0.14	3 (Beetroot has reached harvestable size)	Leaf	0 28	8.4 ≤0.01	RA-2117/98 1523-98
					Beet	0 28	0.11 ≤0.01	
France (South) 1998	250EC	0.38-0.42	0.13-0.14	3 (Beetroot has reached harvestable size)	Leaf	0 28	6.1 ≤0.01	RA-2117/98 1524-98
					Beet	0 28	0.05 ≤0.01	

[1] Expressed as oxydemeton-methyl.

Table 30. Residues of oxydemeton-methyl and demeton-S-methylsulphon in wheat after foliar applications of oxydemeton-methyl.

Country Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
Italy 1997 (spring)	250EC	0.13	0.042	1 (Early milk)	Foliage	0	3.2	RA-2089/97 0417-97
					Grain	21 28	<0.01 ≤0.01	
					Straw	21 28	<0.04 ≤0.04	
Italy 1997 (spring)	250EC	0.13	0.042	1 (Early milk)	Foliage	0	3.8	RA-2089/97 0418-97
					Grain	21 28	<0.01 ≤0.01	
					Straw	21 28	0.09 ≤0.04	
France (South) 1997 (winter)	250EC	0.14	0.043	1 (Soft dough)	Whole plant without roots	0	2.6	RA-2089/97 0576-97
					Grain	21 28	<0.01 ≤0.01	
					Straw	21 28	<0.04 ≤0.04	
France (South) 1997 (winter)	250EC	0.13	0.042	1 (Soft dough)	Foliage	0	2.1	RA-2089/97 0577-97
					Grain	21 29	<0.01 ≤0.01	
					Straw	21 29	<0.04 ≤0.04	
France (South) 1997 (winter)	258EC	0.10	0.033	2 (Soft dough)	Foliage	0	1.6	RA-2162/97 0754-97
					Grain	21 29	<0.01 ≤0.01	
					Straw	21 29	<0.04 ≤0.04	

Country Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1998 (winter)	250EC	0.13	0.040	1 (Early dough)	Foliage	0	2.9	RA-2115/98 1043-98
					Grain	22 29	<0.02 <u>≤0.02</u>	
					Straw	22 29	<0.05 <u>≤0.05</u>	
France (South) 1998 (winter)	250EC	0.13	0.039	1 (Early dough)	Foliage	0	3.4	RA-2115/98 1517-98
					Grain	22 29	<0.02 <u>≤0.02</u>	
					Straw	22 29	0.10 <u>0.06</u>	
Spain 1998 (winter)	250EC	0.13	0.042	1 (Early dough)	Foliage	0	4.3	RA-2115/98 1518-98
					Grain	21 28	<0.02 <u>≤0.02</u>	
					Straw	21 28	0.64[2] 0.55[2]	
Italy 1998 (winter)	250EC	0.13	0.042	1 (Medium milk)	Foliage	0	1.4	RA-2115/98 1519-98
					Grain	21 28	<0.02 <u>≤0.02</u>	
					Straw	21 28	<0.05 <u>≤0.05</u>	
Italy 1998 (winter)	250EC	0.13	0.042	1 (Medium milk)	Foliage	0	1.8	RA-2115/98 1520-98
					Grain	21 28	<0.02 <u>≤0.02</u>	
					Straw	21 28	<0.05 <u>≤0.05</u>	
France (South) 1998 (winter)	258EC	0.12	0.040	2 (Early dough)	Foliage	0	1.1	RA-2086/98 1450-98
					Grain	21 28	<0.01 <u>≤0.01</u>	
					Straw	21 28	<0.04 <u>≤0.04</u>	

[1] Expressed as oxydemeton-methyl.
[2] Contamination suspected, since control sample had residues of 0.35 mg/kg.

Table 31. Residues of oxydemeton-methyl and demeton-S-methylsulphon in barley after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1997 (winter)	258EC	0.10	0.033	2 (Hard dough)	Whole plant without root	0	1.1	RA-2162/97 0753-97
					Grain	21 28	<0.01 <u>≤0.01</u>	
					Straw	21 28	<0.04 <u>≤0.04</u>	
France (South) 1998 (winter)	258EC	0.12	0.040	2 (Early dough)	Foliage	0	3	RA-2086/98 1039-98
					Grain	20 27	0.01 <u>≤0.01</u>	

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
					Straw	20 27	0.04 <0.04	

[1] Expressed as oxydemeton-methyl.

Table 32. Residues of oxydemeton-methyl and demeton-S-methylsulphon in rape after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report/Study no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (South) 1998	258EC	0.15	0.053	1 (15-16 leaf stage)	Foliage	0	6.8	RA-2113/98 1307-98
					Seed	248	<0.01	

[1] Expressed as oxydemeton-methyl.

Table 33. Residues of oxydemeton-methyl and demeton-S-methylsulphon in sunflower after foliar applications of oxydemeton-methyl.

Country (Area) Year	Application				Sample	PHI (days)	Residues (mg/kg)[1]	Report no.
	Form	kg ai/ha	kg ai/hl	No. (Growth stage at last application)				
France (North) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	1.6	RA-2154/97 0410-97
					Head	30	<0.04	
					Seed	93	<0.01	
France (North) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	2.5	RA-2154/97 0733-97
					Head	36	<0.04	
					Seed	97	<0.01	
France (North) 1998	258EC	0.12	0.040	1 (Inflorescence visible between youngest leaves)	Foliage	0	0.63	RA-2114/98 1040-98
					Head	35	<0.04	
					Seed	92	<0.01	
France (South) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	3.6	RA-2155/97 0411-97
					Head	29	<0.04	
					Seed	76	<0.01	
France (South) 1997	258EC	0.10	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	3.5	RA-2155/97 0737-97
					Head	24	<0.04	
					Seed	76	<0.01	
France (South) 1997	258EC	0.11	0.033	1 (Inflorescence visible between youngest leaves)	Foliage	0	5	RA-2155/97 0738-97
					Head	20	<0.04	
					Seed	72	<0.01	

[1] Expressed as oxydemeton-methyl.

FATE OF RESIDUES IN STORAGE AND PROCESSING

A number of processing studies were reported to the 1998 JMPR for evaluation, but the residues in the raw agricultural commodities and the processed products were too low to estimate the processing factors except for apple and cotton seed. All data were from supervised trials where the plants were treated in accordance with the recommended GAP.

In view of the low levels of residues in crops treated according to GAP, the manufacturer conducted a new study in which two types of peas were fortified with oxydemeton-methyl and processed. The first was a marrowfat variety (dry harvested) used exclusively for canning and the second a vining variety (green pea) used for both canning and quick-freezing (Stanley, 2002). The raw peas were spiked by soaking in solutions of an oxydemeton-methyl formulation containing 1.0 mg/l for approximately 24 h to achieve quantifiable residues. The peas were then processed as follows.

Marrowfat peas
On removal from the spiking solution the peas were drained, washed with water, blanched for three minutes at 92 to 94.5°C, drained, cooled and foreign matter (stones, split peas etc.) was removed. The peas were then canned (approximately 220 g/can), covered with a brine preparation (9.5 g salt/37.5g sugar), sealed and sterilized at 121.1°C for six minutes or more, then cooled in water, dried overnight and deep frozen (-18°C) before analysis.

Vining peas (canning)
The process was identical to that used for marrowfat peas except the sterilization at 121.1°C was extended to 13 minutes and the can weight was 285g.

Vining peas (quick frozen)
The process was identical to the canning process up to the inspection phase. Thereafter the peas were placed in a fluidised bed freezer at -34°C for three minutes. The frozen peas were then stored at -16°C before sampling, thawing and boiling in water for one minute.

Samples of peas and processing water were analysed at critical stages by an analytical method based on that of Thornton, 1977, with modifications by Hill, 1984, which was validated for peas at fortification levels of 0.051, 0.20 and 3.13 mg/kg. Recoveries ranged from 84 to 110%.

The results showed that residues in marrowfat peas were 81.57 mg/kg and in vining peas 92.52 mg/kg, appreciably higher than the intended level of 10-50 mg/kg.

The results are shown in Table 34.

Table 34. Residues of oxydemeton-methyl peas and processed fractions.

Sample	Residues (mg/kg)	Residue, % of spiked RAC
Marrowfat peas, canning process		
Treated peas	81.75	100
Treated peas after blanching	46.56	57.0
Blanching water	5.03	6.2
Treated peas after canning	2.85	3.5
Vining peas, canning process		
Treated peas	92.52	100.0
Treated peas after blanching	53.28	57.6
Blanching water	6.33	6.8

Sample	Residues (mg/kg)	Residue, % of spiked RAC
Treated peas after canning	2.23	2.4
Vining peas, freezing and cooking process		
Treated peas	92.52	100.0
Treated peas after blanching	52.97	57.3
Treated peas after freezing	51.85	56.0
Treated peas after cooking	39.69	42.9

The results show appreciable losses of oxydemeton-methyl during blanching and canning. Losses in the blanching process (treatment for three minutes at 94.9-97°C) were more than 40% of which no more than 7% was recovered in the water. Similarly, during canning (8-13 minutes at 121.1°C) losses increased to approximately 97%. In contrast blanching and freezing resulted in a loss of approximately 44% of the original residue and the final domestic cooking of frozen vining pea samples resulted in a further reduction of 13% of the original to give a total reduction of 57% of the residues in the unprocessed peas.

The domestic cooking process (1 minute at 100°C) resulted in a reduction in the residue of 13.1%.

The processing factors derived from the study are as follows:

for processed marrowfat peas (canning)	0.035
for vining peas (canning)	0.024
for vining peas (freezing)	0.56
for vining peas (freezing + domestic cooking)	0.43

NATIONAL MAXIMUM RESIDUE LIMITS

Since the evaluation of oxydemeton-methyl by the 1998 JMPR the European Community has evaluated oxydemeton-methyl and established the MRLs shown below.

Table 34. Recommended EU MRLs for oxydemeton-methyl.

Commodity		MRL, mg/kg	Commodity		MRL, mg/kg
Citrus fruit			Legume vegetables		
	Grapefruit	0.02*		Beans (with pods)	0.02*
	Lemons	0.02*		Beans (without pods)	0.02*
	Limes	0.02*		Peas (with pods)	0.02*
	Mandarins (inc. clementines and similar hybrids)	0.02*		Peas (without pods)	0.02*
	Oranges	0.02*		Others	0.02*
	Pomelos	0.02*	Brassicas		
	Others	0.02*		Flowering Brassicas	
Tree nuts				Broccoli	0.02*
	Almonds	0.02*		Cauliflower	0.02*
	Brazil nuts	0.02*		Others	0.02*
	Cashew nuts	0.02*		Head Brassicas	
	Chestnuts	0.02*		Brussels sprouts	0.05
	Coconuts	0.02*		Head cabbage	0.05

Commodity		MRL, mg/kg
	Hazelnuts	0.02*
	Macadamia nuts	0.02*
	Pecans	0.02*
	Pine nuts	0.02*
	Pistachios	0.02*
	Walnuts	0.02*
	Others	0.02*
Pome fruit		
	Apples	0.02*
	Pears	0.02*
	Quinces	0.02*
	Others	0.02*
Stone fruit		
	Apricots	0.02*
	Cherries	0.02*
	Peaches (inc. nectarines and similar hybrids)	0.02*
	Plums	0.02*
	Others	0.02*
Berries and small fruit		
	Table and wine grapes	0.02*
	Table grapes	0.02*
	Wine grapes	0.02*
	Strawberries (other than wild)	0.02*
	Cane Fruit (other than wild)	0.02*
	Blackberries	0.02*
	Dewberries	0.02*
	Loganberries	0.02*
	Raspberries	0.02*
	Other small fruits and berries (other than wild)	0.02*
	Bilberries	0.02*
	Cranberries	0.02*
	Currants (red, black and white)	0.02*
	Gooseberries	0.02*
	Others	0.02*
	Wild berries and wild fruit	0.02*
Miscellaneous fruit		
	Avocados	0.02*
	Bananas	0.02*
	Dates	0.02*
	Figs	0.02*
	Kiwifruit	0.02*
	Kumquats	0.02*
	Litchis	0.02*
	Mangoes	0.02*
	Olives (table consumption)	0.02*

Commodity		MRL, mg/kg
	Others	0.02*
Leafy Brassicas		
	Chinese cabbage	0.02*
	Kale	0.02*
	Others	0.02*
	Kohlrabi	0.05
Leafy veg. and fresh herbs		
Lettuce and similar		
	Cress	0.05
	Lambs lettuce	0.05
	Lettuce	0.05
	Escarole	0.05
	Others	0.05
Spinach and similar		
	Spinach	0.02*
	Beet leaves (Chard)	0.02*
	Others	0.02*
	Watercress	0.02*
	Witloof	0.02*
Herbs		
	Chervil	0.02*
	Chives	0.02*
	Parsley	0.02*
	Celery leaves	0.02*
	Others	0.02*
Stem vegetables		
	Asparagus	0.02*
	Cardoons	0.02*
	Celery	0.02*
	Fennel	0.02*
	Globe artichokes	0.02*
	Leeks	0.02*
	Rhubarb	0.02*
	Others	0.02*
Funghi		
	Cultivated mushrooms	0.02*
	Wild mushrooms	0.02*
Pulses		
	Beans	0.02*
	Lentils	0.02*
	Peas	0.02*
	Others	0.02*
Oilseeds		
	Linseed	0.05*
	Peanuts	0.05*

Commodity		MRL, mg/kg	Commodity		MRL, mg/kg
	Olives (oil extract)	0.02*		Poppy seed	0.05*
	Papaya	0.02*		Sesame seed	0.05*
	Passion fruit	0.02*		Sunflower seed	0.05*
	Pineapples	0.02*		Rape seed	0.05*
	Pomegranates	0.02*		Soya bean	0.05*
	Others	0.02*		Mustard seed	0.05*
Root and tuber vegetables				Cotton seed	0.05*
	Beetroot	0.02*		Others	0.05*
	Carrots	0.02*	Potato		
	Celeriac	0.02*		Early potatoes	0.02*
	Horseradish	0.02*		Ware potatoes	0.02*
	Jerusalem artichokes	0.02*	Tea		
	Parsnips	0.02*		(Dried leaves and stalks, fermented or otherwise, Camellia sinensis)	0.05*
	Parsley root	0.02*	Hops		
	Radishes	0.02*		Including hop pellets and unconcentrated powder	0.05*
	Salsify	0.02*	Cereals		
	Sweet potatoes	0.02*		Wheat	0.02*
	Swedes	0.02*		Rye	0.02*
	Turnips	0.02*		Barley	0.1
	Yams	0.02*		Sorghum	0.02*
	Others	0.02*		Oats	0.1
Bulb vegetables				Triticale	0.02*
	Garlic	0.02*		Maize	0.02*
	Onions	0.02*		Buckwheat	0.02*
	Shallots	0.02*		Millet	0.02*
	Spring onions	0.02*		Rice	0.02*
	Others	0.02*		Other cereals	0.02*
Fruiting vegetables			Products of animal origin		
			Meat, fat and preparations of meat		0.02*
	Tomatoes	0.02*	Milk and Dairy Produce		0.02*
	Peppers	0.02*	Eggs		0.02*
	Chili peppers	0.02*			
	Aubergines	0.02*			
	Others	0.02*			
Cucurbits - edible peel					
	Cucumbers	0.02*			
	Gherkins	0.02*			
	Courgettes	0.02*			
	Others	0.02*			
Cucurbits - inedible peel					
	Melons	0.02*			
	Squashes	0.02*			

Commodity		MRL, mg/kg	Commodity		MRL, mg/kg
	Watermelons	0.02*			
	Others	0.02*			
	Sweet corn	0.02*			

APPRAISAL

Oxydemeton methyl was evaluated for residues by the 1998 JMPR within the CCPR periodic review programme and then for residues and toxicology by the JMPR in 1999 and 2002, respectively.

At its 31st Session, the CCPR asked the JMPR to clarify whether demeton-*S*-methyl and demeton-*S*-methylsulfon should remain in the residue definition of oxydemeton methyl, as it was believed that registration of these compounds would not be retained. At its Thirty-second Session, the CCPR withdrew the draft MRLs for several commodities, as there was no existing GAP for them. The Committee advanced the proposed draft MRLs to Step 5 and returned the draft MRLs to Step 6 because of intake concerns, which would be considered at its next session. The Committee requested detailed information on oxydemeton methyl. The Committee discussed the definition of the residue that had been confirmed by the 1999 JMPR. It stated that, as demeton-*S*-methyl was no longer supported and there was no GAP, its use should be prevented by removing this compound from the definition of the residue. It was pointed out, however, that demeton-*S*-methyl could not be distinguished from oxydemeton methyl in analysis and that it could be generated from oxydemeton methyl during analysis. As no agreement was reached, the Committee agreed as a compromise to maintain the present residue definition but to specify that the residue definition and MRLs apply only to residues resulting from use of oxydemeton methyl. Those conditions would be met by adding a note to the residue definition, reading: "The residue definition and MRLs are based on the use of oxydemeton methyl only."

At its 33rd Session, the CCPR noted a written comment from the European Commission stating a general reservation (lack of an acute RfD) and a specific reservation on the MRLs for grape, lemon and oranges, sweet, sour (acute risk) and decided to return the draft MRLs to Step 6. The CCPR decided to return all the MRLs to Step 6 until calculations of short-term intake had been obtained from the JMPR. The Committee was informed by the manufacturer that data would be submitted to the 2004 JMPR for a review of the definition of the residue.

The Meeting received new data on physical and chemical properties (partially updated), analytical methods, fate of residues in processing, plant metabolism (apple), residue data (apples, pears, grapes, cabbage, Brussels sprouts, cauliflower, field peas, potatoes, sugar-beet, fodder beet, wheat, barley, rape and sunflower), GAP and national MRLs.

Metabolism

Plants

The metabolism of [ethylene-1-^{14}C]oxydemeton methyl in *apple* was investigated in the field. Apple trees were sprayed on two separate occasions about 4 months before harvest (pink bud stage, BBCH 57) and then about 3 months before harvest (flowers fading, BBCH 67). The formulated material was prepared at a nominal concentration of 1.4 g ai/l and applied at a nominal rate of 350 g ai/ha. Samples of fruit and leaves were taken for analysis 2 h after the first application (leaves only), 2 h after the second application (leaves only), about 60 days before harvest (first intermediate sample; fruit and leaves), about 30 days before harvest (second intermediate sample; fruit and leaves) and at harvest (fruit and leaves).

Analysis of fruit samples (pulp and peel) from the two intermediate samples showed the presence of oxydemeton methyl, but none was found at harvest. The two intermediate samples did, however, contain desmethyl-oxydemeton methyl sulfone (metabolite 7) and two polar materials (P2

and P3). In the first intermediate sample, polar radioactivity in the pulp accounted for 17% of the radioactivity in the fruit (0.2 mg/kg). In the second intermediate sample, polar radioactivity in the pulp had decreased to 10% of that in fruit (0.05 mg/kg), and by harvest no components were detected.

In the peel extracts, polar metabolites accounted for 3% of the radioactivity in the fruit (0.023 mg/kg) at the first intermediate sampling, and this had increased to 4% (0.026 mg/kg) by the second intermediate sampling. No components were detected at harvest.

The main metabolites detected at the first intermediate sampling time in both pulp and peel were oxydemeton methyl (26.6% radioactivity in the fruit, 0.245 mg/kg) and demeton-*S*-methylsulfone (2.9% radioactivity in the fruit, 0.026 mg/kg). In the second intermediate sample, oxydemeton methyl (1.7% radioactivity in the fruit, 0.012 mg/kg) and demeton-*S*-methylsulfone (0.2% radioactivity in the fruit, 0.001 mg/kg) were detected only in peel extracts. The main component detected in pulp extracts was an unidentified polar compound (14.1% radioactivity, 0.072 mg/kg). No components were detected at harvest in either pulp or peel extracts owing to the low levels of radioactivity in the extract samples (<0.001 mg/kg). The results of this study do not change the conclusions reached in the 1998 evaluation.

Environmental fate

Soil

The aerobic degradation of oxydemeton methyl was studied in three soils for a maximum of 11 days under aerobic conditions in the dark at 20°C. [ethylene-1-^{14}C]Oxydemeton methyl was applied at a nominal rate of 0.67 mg/kg dry soil, equivalent to the proposed single maximum annual use rate of 250 g ai/ha calculated for 2.5 cm depth of soil.

During the study, the total recovery of radioactivity in individual test vessels ranged from 90.8% to 100%, and the times to 50% and 90% decomposition (DT50 and DT_{90}) in the three soils ranged from 0.17 to 0.22 and from 0.58 to 0.74 days, respectively. The results also indicate that the main metabolites were continuously degraded, that no metabolite accumulated towards the end of the study and that the bound residues participated in the natural carbon cycle of soil.

Analysis of soil extracts showed two major and one semi-major degradation product, representing 10% or more of the applied radioactivity at any time during the study. The concentration of the 2-ethylsulfinyl ethane sulfonic acid metabolite reached a maximum on day 1 and then declined gradually until day 11. Its concentration was below the LOQ towards the end of the study in soils with higher microbial activity. The 2-ethylsulfonyl ethane sulfonic acid metabolite is an oxidation product of 2-ethylsulfinyl ethane sulfonic acid, and its concentration reached a maximum on day 3 in all soils; it ranked highest, at 16.8% of the applied radioactivity. The level declined towards the end of the study in all soils, and in the most active soil to below the LOQ by day 11. Significant formation of bound residues occurred during overall metabolism of parent compound. The concentration of bound residues reached a maximum on day 11 at about 50% of the applied radioactivity. Soils showed high mineralization capacity, yielding values for $^{14}CO_2$ of > 30% by day 11. The results of study demonstrate that oxydemeton methyl is quickly degraded in aerobic soils.

Water–sediment systems

The hydrolysis of oxydemeton methyl was studied in sterile 0.01 mol/l buffer solutions, which were adjusted to pH4, 7 or 9, for a maximum of 31 days in the dark at two temperatures. The experiment was carried out in compliance with good laboratory practice (GLP) and in accordance with guidelines of the US Environmental Protection Agency, the Society of Environmental Toxicology and Chemistry, the OECD and the European Commission. The test solutions were prepared with [ethylene-1-^{14}C]oxydemeton methyl at a concentration of about 5 m g/l. The pre-test solutions were incubated for 7 days under sterile conditions in the dark at 50°C. The solutions at pH 4 and pH 7 in the main test were incubated for a maximum of 31 days under sterile conditions in the dark at 25°C.

In the pre-test at 50°C and in the main test at 25°C, oxydemeton methyl was not stable at pH 4, 7 or 9, and considerable degradation occurred. Especially at higher pH values, the compound was

thoroughly hydrolysed to desmethyl-oxydemeton methyl and 2-ethylsulfinyl-ethyl mercaptan by cleavage of the P–S bound. Furthermore, 2-ethylsulfonyl ethane sulfonic acid was observed as an oxidized P–S cleavage product at low percentages (maximum of 2.2% of the applied radioactivity), although it was identified only tentatively.

By calculation from the data obtained at 50°C, orienting DT50 values (first order) for the hydrolysis of oxydemeton methyl were estimated to be 4.9, 3.5 and 0.2 days at pH 4, 7 and 9, respectively. Using the data obtained at 25°C, the DT_{50} values (first order) were estimated to be 91, 42 and 2.5 days at pH 4, 7 and 9, respectively. At 20°C, the DT_{50} values calculated from Arrhenius plots (1/T versus ln(k)) were 174, 73 and 4.5 days at pH 4, 7 and 9, respectively. The results indicate that hydrolytic processes contribute to the degradation of oxydemeton methyl in the environment.

The quantum yield from direct photodegradation of oxydemeton methyl in water was determined according to the European Centre for Ecotoxicilogy and Toxicology of Chemicals method in polychromatic light. The quantum yield calculated from the ultraviolet absorption data and the kinetics of photodegradation was 0.00078. The resulting quantum yield and data on ultraviolet absorption in aqueous solution were used to estimate the environmental half-life of oxydemeton methyl after direct photodegradation in water in two simulation models. The calculated half-lives were 112 days in summer and 274 days in winter at 30° latitude and 200 days in May and 790 days in October at 50° latitude.

Methods of analysis

A number of methods have been developed for the analysis of residues of oxydemeton methyl in various matrices, many of which were reviewed by the 1998 Meeting as part of the periodic review of this compound. The Meeting was provided with additional methods based on the same principle as those evaluated earlier, i.e. use of the oxidation process to produce demeton-*S*-methylsulfone as the analyte. The LOQs were 0.005 mg/kg for potato; 0.005–0.01 mg/kg for apples and grapes; 0.01 mg/kg for pear, Brussels sprouts, cauliflower, cabbage, corn, sunflower, rape-seed and rape (green plant material); 0.02 mg/kg for wheat grain and 0.05 mg/kg for wheat straw.

Stability of residues in stored analytical samples

The stability of oxydemeton methyl in stored cabbage, maize, lettuce and papaya was evaluated by the 1998 JMPR, which concluded that data on the stability of stored analytical samples of raw agricultural commodities containing quantifiable residues of oxydemeton methyl were highly desirable. The available information was not representative of the various crop groups, did not cover extended storage intervals and suggested variable storage stability. The manufacture submitted new study data on the storage stability of oxydemeton methyl in several crops.

A study was conducted to determine the stability of oxydemeton methyl in spiked samples of stored apple, dried peas, potato and oil-seed rape (meal and oil at-20°C). Samples for the study were obtained from a commercial source. The samples of apple and potato were prepared for use by fine chopping in an industrial food processor; the preparation of samples of dried peas was not recorded in the report. For each crop, 20 g of prepared sample were spiked with formulated oxydemeton methyl at a nominal concentration of 0.1, 1.0 or 10 mg/kg and placed in storage at-20°C. Two samples of each were removed for analysis after 0, 3, 6, 12 and 24 months of storage. The analytical method was validated for each crop at each sampling time.

There was no substantial loss of residue during 24 months' storage from apple, potato or oil; however, the residues in dried peas and rape meal apparently decreased to half the initial values after 3 or 6 months of storage. The control samples (un-spiked samples) also showed residues of oxydemeton methyl, with <0.005–0.022 mg/kg in apple, <0.005–0.016 mg/kg in dried peas, <0.01–0.016 mg/kg in potatoes, 0.013–0.03 mg/kg in rape meal and <0.005–0.011 mg/kg in rape oil. The Meeting concluded that the data submitted on storage stability were insufficient or inadequate, and the former requirement was maintained.

Definition of the residue

The Meeting received the results of new studies of plant metabolism and supplementary information on the analytical method. The new data did not, however, provide a basis for changing the current definition of the residue. The Meeting confirmed its previous recommendation.

Results of supervised trials on crops

The results of supervised field trials on apples, pears, grapes, cabbage, Brussels sprouts, cauliflower, field peas, potatoes, sugar-beet, fodder beet, wheat, barley, rape and sunflower were submitted to the Meeting. The new data were evaluated against current GAPs, and highest residue levels were estimated for commodities evaluated by the 1998 JMPR, as the 1998 JMPR did not do so. When no residues were found in any sample in older trials in which the analytical methods used had higher LOQs than current methods, the Meeting decided to use only data from the newly submitted trials in order to avoid unnecessarily high maximum residue levels.

Citrus fruit

No data from new supervised trials were submitted. From 11 trials on *orange* and *lemon*, the 1998 JMPR estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.01 mg/kg.

In the 1998 evaluations, the reported residues in pulp, in ranked order, were <0.01 (seven), 0.01, 0.02 (two) and 0.04 mg/kg. The Meeting estimated a highest residue level of 0.04 mg/kg for orange and lemon.

Pome fruit

The results of 15 new supervised trials on apples and two on pears were submitted to the Meeting

One supervised trial on *apples* in northern France in 1998 involved higher doses than used in German GAP (0.13 kg ai/ha once, immediately after flowering). Nevertheless, the residue data could be used, since the residue levels were below the LOQ. Three supervised trials on apples in southern France in 1997 and 1998, three on apples in Italy in 1997 and 1998 and four on apples in Spain in 1997 and 1998 were conducted according to Italian GAP (0.023–0.028 kg ai/hl, 90-day PHI). As two of the four Spanish trials (conducted in 1997) were carried out in the same location under almost identical trial conditions, one residue level was taken from each. The residue levels of oxydemeton methyl in apples were <0.01 (nine) and 0.01 mg/kg.

One supervised trial on *pears* in southern France in 1998 was conducted according to Italian GAP (0.023–0.028kg ai/hl, 90-day PHI), and one trial on pears in Germany in 1998 was conducted according to Austrian GAP (0.024 kg ai/hl once, up to 2 weeks after flowering). The residue level of oxydemeton methyl in pears was <0.01 (two) mg/kg.

The 1998 JMPR evaluated 10 supervised trials on apples and pears and estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg for apple and pear, based on residue levels, in ranked order, of <0.01 (seven), 0.03 and <0.04 (two) mg/kg.

The Meeting confirmed that the residue levels found in the newly submitted trials did not exceed the formerly estimated maximum and highest residue values of 0.04 mg/kg for apple and pear in the 1998 JMPR evaluations.

Grape

Three new supervised trials from Germany were conducted within German GAP (0.027 kg ai/hl, up to fully developed inflorescence). The residue levels were <0.01 (three).

The 1998 JMPR evaluated the results of five supervised trails and estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.04 mg/kg.

The Meeting confirmed that the residue levels found in the newly submitted trials did not exceed the formerly estimated maximum and highest residue values of 0.06 mg/kg for grapes in the 1998 JMPR evaluations. The residue levels, in ranked order, were <0.04 (four) and 0.06 mg/kg.

Brassica vegetables

Cabbage (head)

The results of four new supervised trials were submitted to the Meeting.

Two supervised trials on red and white cabbage in northern France in 1996 and two supervised trials on Savoy cabbage in Germany were conducted according to German GAP (0.16 kg ai/ha (<50 cm) once, 21-day PHI). The residue levels were <0.01 (four) mg/kg.

The 1998 JMPR evaluated 16 supervised trials and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.03 mg/kg. The highest value of <0.06 mg/kg was disregarded because of the high LOQ in the older trials (1976).

The Meeting confirmed that the residue levels in the submitted trials did not exceed the formerly estimated maximum and highest residue values of 0.05 mg/kg in the 1998 JMPR evaluations. The residue levels, in ranked order, were <0.01 (six), 0.02, <0.03 (three), <0.04 and <0.05 (four) mg/kg.

Kale

No data from new supervised trials were submitted to the Meeting. Four supervised trials were evaluated by the 1998 JMPR, which estimated a maximum residue level of 0.01* mg/kg and an STMR of 0.01 mg/kg.

On the basis of the reported residue level, <0.01 (four) mg/kg, the Meeting estimated a highest residue level of 0.01mg/kg.

Kohlrabi

No data from new supervised trials were submitted to the Meeting. Four supervised trials were evaluated by the 1998 JMPR, which recommended a maximum residue level of 0.05 mg/kg and an STMR of 0.02 mg/kg. The reported residue levels, in ranked order, were <0.01 (two), 0.03 and <0.06 mg/kg. The highest value was disregarded because of the high LOQ associated with the older trials (1979).

The Meeting estimated a highest residue level of 0.05 mg/kg, at the same level as the maximum residue level recommended by the 1998 JMPR.

Brussels sprouts

Five supervised trials conducted in Germany in 1997 and 1998, two conducted in Belgium in 1997 and 1998, one conducted in the United Kingdom in 1998 and one conducted in northern France in 1998 were submitted to the Meeting; however, no comparable GAP was submitted. The Meeting could not therefore estimate a maximum residue level, an STMR or a highest residue level.

Cauliflower

The results of eight new supervised trials were submitted to the Meeting. Two trials in Germany in 1996 were conducted according to German GAP (0.16 kg ai/ha (<50 cm) once, 21-day PHI) and also according to Belgian GAP (0.15 kg ai/ ha once, 28-day PHI).

Four supervised trials in southern France in 1996 and 1997 and one in the United Kingdom in 1996 were conducted according to German GAP. One of the trials in France in 1996 involved higher doses than in German GAP, but the residue data could be used for evaluation as the level was below the LOQ.

In all the trials the residue level was <u>≤0.01</u> (eight) mg/kg. The Meeting estimated a maximum residue level of 0.01* mg/kg and STMR and highest residue values of 0.01 mg/kg for cauliflower.

Field peas (dry)

Data from two supervised trials on field peas were submitted to the Meeting, but no information on GAP was provided. The Meeting could therefore not estimate a maximum residue level, an STMR or a highest residue level.

Potatoes

The results of 20 supervised trials were submitted to the Meeting.

Two supervised trials in Germany in 1996 and one in the United Kingdom in 1996 were not matched by comparable GAP. Nine supervised trials in France in 1996 and 1998, one in Greece in 1998, three in Italy in 1997 and 1998 and four in Spain in 1997 and 1998 were conducted according to Greek GAP (0.05kg ai/hl three times, 28-day PHI). As two of four Spanish trials conducted in 1997 were carried out in the same location under similar trial conditions, one residue level was taken from each. The residue levels, in ranked order, were <0.005 (five) and ≤0.01 (11) mg/kg.

The 1998 JMPR evaluated the results of 16 supervised trials and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.02 mg/kg. The reported residue levels, in ranked order, were <0.01 (seven), <0.02 (nine) and <0.05 (two) mg/kg.

The Meeting decided to use the data from the newly submitted trials and estimated a maximum residue level of 0.01*mg/kg and STMR and highest residue values of 0.01 mg/kg to replace the former recommendations.

Sugar-beet (root)

The results of seven supervised trials on sugar-beet and two on fodder beet were submitted to the Meeting.

Three supervised trials on sugar-beet in Spain in 1997 and 1998 and four in Italy in 1997 and 1998 involved higher doses than in Spanish GAP (0.025 kg ai/hl, 30-day PHI) or Italian GAP (0.023–0.028 kg ai/hl, 30-day PHI); however, the residue data could be used for evaluation as all the levels in leaf and root were below the LOQ. As two of the three Spanish trials were conducted at the same location under the same trial conditions, one residue level was taken from each.

Two supervised trials on fodder beet in southern France in 1998 involved higher doses than in Spanish GAP (0.025 kg ai/hl, 30-day PHI), but the residue data could be used for evaluation as all the levels in leaf and root were below the LOQ. Six trials on sugar-beet and two on fodder beet could be evaluated together, as all the residue levels were ≤0.01 (eight) mg/kg.

The 1998 JMPR evaluated seven supervised trials on sugar-beet and two on fodder beet in Germany, and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.04 mg/kg. The reported residue levels, in ranked order, were <0.01 (four) and <0.04 (five) mg/kg.

The Meeting decided to use the data from the newly submitted trials and estimated a maximum residue level of 0.01*mg/kg and an STMR of 0.01 mg/kg, to replace the former recommendations.

Wheat, barley and rye

Eleven supervised trials on wheat and two on barley were submitted to the Meeting.

Four supervised trials on wheat in Italy in 1997 and 1998, six in southern France in 1997 and 1998 and one in Spain in 1998 involved higher doses than in Italian GAP (0.023–0.028 kg ai/hl, 30-day PHI), but the data on residues on wheat grain could be used for evaluation as all the levels were below the LOQ. The residue levels, in ranked order, were <0.01 (six) and <0.02 (five) mg/kg.

Two supervised trials on barley in southern France in 1997 and 1998 involved slightly higher doses than in the Italian GAP for wheat. The Meeting concluded that this GAP could be applied to trials on barley, as the two crops are cultivated similarly. The residue levels were <0.01 (two) mg/kg.

The 1998 JMPR evaluated seven supervised trials on wheat and three on barley and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.04 mg/kg. The reported residue levels, in ranked order, were <0.04 (seven) and <0.05 (three) mg/kg.

The Meeting decided to use the data from the newly submitted trials to estimate the maximum residue level. The combined residue levels, in ranked order, were ≤0.01 (eight) and <0.02 (five)

mg/kg. The Meeting estimated a maximum residue level of 0.02*mg/kg and an STMR of 0.01 mg/kg, to replace the former recommendations.

Rape-seed

One supervised trial on rape was submitted to the Meeting; however, no information on GAP was provided. The Meeting could therefore not estimate a maximum residue level, an STMR or a highest residue level.

Sunflower seed

Six supervised trial data on sunflower were submitted to the Meeting; however, no information on GAP was provided. The Meeting could therefore not estimate a maximum residue level, an STMR or a highest residue level.

Sugar-beet (tops)

The residue levels in the leaves of sugar-beets and fodder beets treated according to GAP, in ranked order, were ≤0.01 (five) and <0.04 (three) mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR of 0.01 mg/kg and a highest residue level of 0.04 mg/kg, to replace the former recommendations.

Wheat, barley and rye straw and fodder

The residue levels in straw and fodder from wheat and barley, in ranked order, were ≤0.04 (eight), <0.05 (three) and 0.06 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR of 0.04 mg/kg and a highest residue level of 0.06 mg/kg, to replace the former recommendations.

Fate of residues during processing

The results of studies on residues in processed peas were provided to the Meeting. The samples were fortified with diluted oxydemeton methyl formulation by soaking because the levels of residue in samples produced under GAP conditions were expected to be too low. The reported processing factors were 0.034 for processed marrowfat peas (canning), 0.024 for vining peas (canning), 0.43 for vining peas (freezing) and 0.43 for vining peas (freezing and domestic cooking).

Residues in animal commodities

The 1998 JMPR, estimated the dietary burden of farm animals and concluded that quantifiable residues of demeton-*S*-methyl, oxydemeton methyl or demeton-*S*-methylsulfone are unlikely to occur in commodities of animal origin (meat, milk, poultry and egg). Therefore, MRLs could be set at the practical LOQ of 0.05* mg/kg for all commodities except milk and at 0.01* mg/kg for milk. The current Meeting did not recommend the addition of further feed items or an increase in the recommended residue levels. It therefore confirmed the previous maximum residue levels and STMRs for commodities of animal origin and estimated a highest residue level of 0 mg/kg for cattle fat, eggs, meat of cattle, pigs and sheep, pig fat, poultry fats, poultry meat and sheep fat.

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, as the information provided was not representative of the various crop groups, did not cover extended storage and suggested variable storage stability.

RECOMMENDATIONS

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

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

The definition of the residue and MRLs are based on the use of oxydemeton-methyl only.

Commodity		Recommendations, mg/kg			
CCN	Name	New	Previous	STMR or STMR-P	HR or HR-P
FP 0226	Apple		0.05[1]	0.01	0.04
JF 0226	Apple juice			0.01	
	Apple sauce			0.005	
GC 0640	Barley	0.02(*)	0.05(*)	0.01	
AS 0640	Barley straw and fodder, dry	0.1	2		
VB 0041	Cabbage, Head		0.05(*)[1]	0.03	0.05
MF 0812	Cattle fat		0.05(*)	0	0
VB 0404	Cauliflower	0.01(*)	W	0.01	0.01
VD 0526	Common bean (dry)		0.1	0.01	
SO 0691	Cotton seed		0.05	0.01	
OR 0691	Cotton seed oil, edible			0.002	
PE 0112	Eggs		0.05(*)	0	0
FB 0269	Grape		0.1[1]	0.04	0.06
VL 0480	Kale		0.01(*)	0.01	0.01
VB 0405	Kohlrabi		0.05	0.02	0.05
FC 0204	Lemon		0.2	0.01	0.04
MM 0097	Meat of cattle, pigs and sheep		0.05(*)	0	0
ML 0106	Milks		0.01(*)	0	
FC 0004	Oranges, Sweet, Sour		0.2[1]	0.01	0.04
FP 0230	Pear		0.05	0.01	0.04
MF 0818	Pig fat		0.05(*)	0	0
VR 0589	Potato	0.01(*)	0.05(*)	0.01	0.01
PF 0111	Poultry fat		0.05(*)	0	0
PM 0110	Poultry meat		0.05(*)	0	0
GC 0650	Rye	0.02(*)	0.05(*)	0.01	
AS 0650	Rye straw and fodder, dry	0.1	2		
MF 0822	Sheep fat		0.05(*)	0	0
VR 0596	Sugar beet	0.01(*)	0.05(*)	0.01	
AV 0596	Sugar beet leaves or tops	0.05	0.05(*)		
GC 0654	Wheat	0.02(*)	0.05(*)	0.01	
AS 0654	Wheat straw and fodder, dry	0.1	2		

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 information was not

representative of the various crop groups, did not cover extended storage, and suggested variable storage stability.

DIETARY RISK ASSESSMENT

Long-term intake

STMR or STMR-P values were estimated by the 1998 JMPR and by the present Meeting for 27 commodities. When data on consumption were available, these values were used in the estimates of dietary intake.

The dietary intake from the five GEMS/Food regional diets, on the basis of the STMR values, represented 3–30% of ADI (Annex 3 of the Report). The Meeting concluded that the intake of residues of oxydemeton methyl resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI for oxydemeton methyl was calculated for the commodities for which maximum residue levels, STMR values and highest residue levels were established and for which data on consumption (of large portions and unit weight) were available. The results are shown in Annex 4 of the Report.

The ARfD for oxydemeton methyl is 0.002 mg/kg bw. The IESTI represented 0–220% of the ARfD for children and 0–90% of that for the general population. For children, 100% of the ARfD was exceeded in apple (130%), cabbage (120%), grape (220%) and orange (120%).

The Meeting concluded that the short-term intake of residues of oxydemeton methyl from uses on commodities other than apples, cabbages, grapes and oranges that have been considered by the JMPR is unlikely to present a public health concern.

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