ETHION (034)

EXPLANATION

Ethion, originally evaluated by the JMPR in 1968 and subsequently re-evaluated for residues several times up to 1983, is included in the CCPR periodic review programme. The TADI was replaced by a lower ADI by the 1990 JMPR. GAP and residue data were requested in order that consideration might be given to the reassessment of existing CXLs (ALINORM 91/24A, para 93; CL 1991/15-PR).

The Meeting evaluated new information from the manufacturer on plant and animal metabolism, storage stability and residues in citrus. Data on the processing of citrus and grapes, and residue data on apples, maize, curcurbits, grapes, pears and plums were also reviewed.

IDENTITY

ISO common name: Ethion

Chemical name

IUPAC: *O*,*O*,*O*′,*O*′-tetraethyl *S*,*S*′-methylenebis(phosphorodithioate)

CA: *S*,*S*′-methylene bis(*O*,*O*′-diethyl phosphorodithioate)

CAS Registry No: 563-12-2

Structural formula:

Molecular formula: $C_9H_{22}O_4P_2S_4$

Molecular weight: 384.5

Physical and chemical properties

Pure active ingredient

Vapour pressure:	0.2 mPa (25°C)
Melting point:	-12 to -15°C
Octanol/water partition coefficient:	log P_{ow} = 5.07
Solubility:	sparingly soluble in water, miscible with most organic solvents, e.g. acetone, methanol, ethanol
Specific gravity:	1.22 (20°C)
Hydrolysis:	hydrolysed by aqueous acids and alkalis

USE PATTERN

No information on GAP has been reviewed since 1972. The Meeting received additional or revised information which is summarized in Table 1.

Table 1. Registered uses of ethion.

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray conc., kg ai/hl	
Apple	Greece			0.048-0.072	28
	Canada	3	1.7 -2.3		30
	Spain		0.71-1.4	0.047-0.094	28
Apricot	Greece		0.048-0.072		28
	Spain		0.71-1.4	0.047-0.094	28
Cherry	Spain		0.71-1.4	0.047-0.094	28
			1.7-2.3		AH
Citrus	Cyprus			0.05 -0.1	20
	Spain		3.8 -4.8	0.095-0.12	28
Coffee	Kenya		2.4		
Cotton	Greece		0.6 -0.72		-
	Spain		0.28-0.56	0.047-0.094	28
Cucumber	Greece		0.36-0.96		7
Egg plant	Greece		0.36-0.96		7
French beans	Greece		0.36-0.96		15
	Kenya		0.48		2
Fruit trees	Cyprus		0.05 -0.1		20
Grapefruit	USA	3	2.8 -3.4	0.12 -0.15	0
Grapes	Canada	1	2.5*		30

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray conc., kg ai/hl	
		2	0.81-1.4**		30
	Cyprus			0.05 -0.1	20
	Greece			0.072-0.096	28
	Spain		0.47-0.94	0.047-0.094	28
Lemon	Greece			0.048-0.096	28
	USA	3	2.8 -3.4	0.12 -0.15	15
Lime	USA	3	2.8 -3.4	0.12 -0.15	15
Melon	Greece		0.36-0.96		7
Nectarine	Greece			0.048-0.072	28
Olive	Greece			0.072-0.096	28
Onion	Greece		0.36-0.96		15
	Canada		1.1 -4.2		FT
Orange	Greece			0.048-0.096	28
	USA	3	2.8 -3.4	0.12 -0.15	0
Peach	Greece			0.048-0.072	28
	Canada	3	1.7 -2.3		30*, 15**
	Spain		0.71-1.4	0.047-0.094	28
Pear	Greece			0.048-0.072	28
	Canada	3	1.7 -2.3		30
	Spain		0.71-1.4	0.047-0.094	28
Pepper	Greece		0.36-0.96		15
Plum	Greece			0.048-0.072	28
	Canada	3	1.7 -2.3		30*, 15**
	Spain		0.71-1.4	0.047-0.094	28
Strawberry	Cyprus			0.056-0.07	2
	Greece		0.36-0.96		15
	Canada		0.56-1.1		8
Tangelo	USA	3	2.8 -3.4	0.12 -0.15	0
Tangerine	Greece			0.048-0.096	28
	USA	3	2.8 -3.4	0.12 -0.15	0
Tea	Kenya			2.4	
Tomato	Kenya			0.48	2
	Greece		0.36-0.96		15
Vegetables	Cyprus			0.056-0.07	2

* Eastern Canada
** British Columbia
AH Apply only after harvest
FT Furrow treatment: apply at planting by drilling the product directly into the seed furrow at the same depth as the seed.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting reviewed supervised trial data for citrus, apples, grapes, maize, pears, plums and cucurbits (one study).

Tables in this section

Table No.

Underlined residues in Tables 2-15 are from treatments according to GAP.

Crops

Apples. Available data are summarized in Table 2. Apples from 6 US trials were treated in 1984 with one delayed dormant application of Ethion 8 EC or Ethion Superior 70 Oil Insecticide followed by two foliar cover sprays of Ethion 25 WP (Leppert, 1985). Two US trials involving the use of Ethion 25 WP were carried out in 1971 (Hinstridge, 1972b).

Table 2. Residues of ethion in apples, USA.

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1984	Ethion 8 EC	3	1.7 (1)		0	4.7	23
	Ethion 25 WP		3.4 (2)		7	2.5	
			3.4 (3)		14	1.9	
					21	1.3	
					28	1.1	
					35	0.88	
1984	Ethion 8 EC	3	1.7 (1)		0	4.2	23
	Ethion 25 WP		3.4 (2)		7	3.0	
			3.4 (3)		14	2.7	
					20	2.7	

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
					28	2.8	
					34	2.4	
1984	Ethion	3	1.7 (1)		0	2.0	23
	Superior				7	2.1	
	70 Oil				14	2.5	
	Ethion 25 WP		3.4 (2)		21	1.3	
			3.4 (3)		28	0.72	
					35	0.98	
1984	Ethion	3	1.3 (1)		0	4.4	23
	Superior				7	3.0	
	70 Oil				14	1.6	
	Ethion 25 WP		3.4 (2)		21	1.8	
			3.4 (3)		28	1.7	
					35	1.8	
1984	Ethion 8 EC	3	1.7 (1)		0	6.4	23
	Ethion 25 WP		3.4 (2)		7	5.6	
			3.4 (3)		14	3.6	
					21	2.3	
					28	3.8	
					35	2.9	
1985	Ethion Superior	3	1.7 (1)		0	4.2	23
	70 Oil				10	1.7	
	Ethion 25 WP		3.4 (2)		20	1.8	
			3.4 (3)		30	1.5	
1971	Ethion 25 WP	2	2.8	0.08	28	0.68	15
1971	Ethion 25 WP	2	2.8	0.08	20	0.6	15

(1) First application
(2) Second application
(3) Third application

Pears. Trees from 1 eastern and 2 western US trials received 1 delayed dormant application of Ethion 8 EC or Ethion Superior 70 Oil Insecticide followed by 2 foliar cover sprays of Ethion 25 WP (Leppert, 1985). Two US trials involving the use of Ethion 25 WP were undertaken in 1971 (Hinstridge, 1972b).

Table 3. Residues of ethion in pears, USA.

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1984	Ethion 8 EC	3	1.7 (1)		0	3.4	24
	Ethion 25 WP		3.4 (2)		7	3.0	
			3.4 (3)		14	2.4	
					21	1.9	
					28	1.6	
					35	1.1	
1984	Ethion 8 EC	3	1.4 (1)		0	7.7	24
	Ethion 25 WP		3.4 (2)		10	5.1	
			3.4 (3)		21	3.8	
					30	2.5	
					41	1.0	
1984	Ethion Superior	3	1.7 (1)		0	2.1	24
	70 Oil				10	1.7	
	Ethion 25 WP		3.4 (2)		20	1.5	
			3.4 (3)		30	1.1	
					40	0.52	
1971	Ethion 25 WP	1	2.8	0.08	23	1.0	15
1971		2	3.4 (1)	0.08	20	1.9	15
			2.8 (2)				

Plums and prunes. Residues in harvest samples of plums and prunes are shown in Table 4. Both crops had received 3 applications of WP formulation and were sampled 21 days after the final treatment (Hinstridge, 1972c).

Table 4. Residues of ethion in plums and prunes, USA, 1971 (Ref. 16).

Form	Application			PHI, days	Residue, mg/kg
	No	kg ai/ha	kg ai/hl		
Ethion 25 WP	3	2	0.05	21 23	plums: 0.31 0.28
	3	2	0.05	21	prunes: 0.21

Grapes. Available data are summarized in Table 5. Grapes in 9 trials in 4 US states conducted during 1984 and 1985 were treated with 2 foliar applications of Ethion 25 Spray (WP). 5 grape varieties were involved (Leppert, 1986).

Table 5. Residues of ethion in grapes, USA (Ref. 25).

Year	Form	Application		PHI, days	Residue, mg/kg
		No	kg ai/ha		
1984	Ethion 25 WP	2	2.2	0	14.3
				9	4.0
				20	4.3
				30	3.6
				40	1.8
1984	Ethion 25 WP	2	2.2	0	15.7
				10	6.9
				20	5.6
				30	4.4
				40	3.9
1984	Ethion 25 WP	2	2.2	0	14.0
				10	6.4
				20	5.9
				30	2.5
				40	2.2
1985	Ethion 25 WP	2	2.2	0	5.6
				10	2.5
				19	1.7
				30	1.2
				40	1.1
1985	Ethion 25 WP	2	2.2	0	3.9
				10	2.4
				20	2.7
				30	2.2
				41	1.2
1985	Ethion 25 WP	2	2.2	0	2.1
				10	1.2
				20	0.71
				30	0.57
				40	0.44
1985	Ethion 25 WP	2	2.2	0	2.5
				10	1.1
				20	0.39
				30	0.47
				40	0.57

Year	Form	Application		PHI, days	Residue, mg/kg
		No	kg ai/ha		
1985	Ethion 25 WP	2	2.2	0	3.8
				11	2.0
				21	2.4
				31	2.1
				40	2.1
1985	Ethion 25 WP	2	2.2	0	6.0
				10	2.8
				20	1.9
				30	1.9
				40	2.6

Grapefruit. During the 1989 growing season 8 trials were carried out in Florida and Texas. Parts of grapefruit groves were treated with foliar applications of Ethion 4 Miscible using airblast sprayers (Stenzel, 1992b). In another study (Kaiser, 1991a), also in Florida and Texas, field trials were with Ethion 4 Miscible or 8 EC. During 1984 and early 1985 (Witkonton and Barge, 1985) in 4 trials with Ethion 4 EC and 1 with Ethion 8 EC, trees were sprayed in Florida (aerial application) and California (ground application). Results are shown in Table 6.

Table 6. Residues of ethion in grapefruit, USA.

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1989	Ethion 4	3	3.4	0.15	0	0.62	36
	Miscible				15	0.61	
					30	0.14	
					45	0.59	
					60	0.35	
1989		2	3.4	0.15	70	0.19	36
					90	0.23	
					120	0.08	
					150	0.07	
1989		3	3.4	0.15	0	0.85	36
					15	0.85	
					30	0.96	
					45	0.77	
					60	0.61	
1989		2	3.4	0.15	60	0.57	36
					90	0.21	
					120	0.28	
					150	0.23	
1989		3	3.4	0.15	0	1.0	36

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
					15	0.84	
					30	0.60	
					45	0.51	
					60	0.65	
1989		2	3.4	0.15	60	0.22	36
					90	0.21	
					120	0.18	
					141	0.15	
1989		3	3.4	0.18	0	1.6	36
					15	1.5	
					30	1.1	
					45	0.53	
					60	0.73	
1989		2	3.4	0.18	60	0.63	36
					90	0.25	
					120	0.21	
					150	0.13	
1989	Ethion 4	3	3.4	0.15	0	1.7	20
	Miscible				15	0.21	
	or 8 EC				30	1.0	
					90	0.92	
					150	0.53	
1989		2	3.4	0.15	90	0.21	20
					150	0.13	
1989		3	3.4	0.15	0	1.3	20
					15	1.3	
					30	1.2	
					90	0.54	
					150	0.15	
1989		2	3.4	0.15	90	0.51	20
					150	0.16	
1989		3	3.4	0.15	0	2.8	20
					15	1.8	
					30	1.2	
					90	0.52	
					150	0.26	
1989		2	3.4	0.15	90	<0.10	20
					150	<0.10	
1989		3	3.4	0.15	0	2.0	20
					15	1.4	
					30	1.6	
					90	1.0	

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
					150	0.52	
1989		2	3.4	0.15	90	0.78	20
					150	0.46	
1984	Ethion 4 EC	3	6.3*		0	0.97	42
					7	0.43	
					14	0.4	
					21	0.24	
1984		3	6.3*		0	0.84	42
					7	0.48	
					14	0.88	
					21	0.38	
1984		3	3.4		0	1.7	42
					7	1.0	
					14	1.0	
					21	1.2	
1984		3	3.4		0	1.2	42
					7	1.3	
					14	1.9	
					21	1.2	
1984	Ethion 8 EC	4	6.3		0	1.6	42
					7	2.6	
					14	1.6	
					21	1.3	

* Foliar aerial application

Lemons. Data from 8 US trials are shown in Table 7. Parts of lemon groves were treated with 2 or 3 foliar applications of Ethion 4 Miscible using a speed sprayer (Goldsmith, 1992b; Stenzel, 1992c). In 4 trials in 1984 and 1985 single foliar sprays of Ethion 4 EC or 8 EC were applied (Witkonton and Barge, 1985).

Table 7. Residues of ethion in lemons, USA.

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1989	Ethion 4	3	3.4	0.15	0	1.1	37
	Miscible				15	1.7	
1989		2	3.4	0.15	45	0.46	37
					60	0.34	
					90	0.21	
1990		3	3.4	0.15	0	4.7	12
					15	2.9	
					29	2.8	
					45	3.0	
					59	1.9	
					91	1.2	
1990		2	3.4	0.15	60	1.6	12
					91	2.1	
					120	0.07	
					150	0.08	
					180	0.1	
1984	Ethion 4 EC	1	3.4		0	1.9	42
					7	2.3	
					14	1.4	
					21	1.5	
					28	1.2	
1984	Ethion 8 EC	1	6.3		0	4.2	42
					7	2.9	
					14	<0.02	
					21	0.96	
					28	0.24	
1984		1	6.3		0	1.7	42
					7	1.6	
					14	1.2	
					21	1.2	
					28	1.3	

Limes. In US studies Ethion 4 Miscible was applied 2 or 3 times to lime groves in Florida in 1989 and 1990 (Goldsmith, 1992; Stenzel, 1992d) and, in 2 trials in 1984, Ethion 4 EC was applied once (Witkonton and Barge, 1985). See Table 8.

Table 8. Residues of ethion in limes, USA.

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1989	Ethion 4	3	3.4	0.15	0	2.8	38
	Miscible				15	1.6	
					30	1.3	
					45	0.75	
					60	0.59	
					76	0.40	
1989		3	3.4	0.15	0	3.5	38
					15	2.1	
					30	1.5	
					45	1.4	
					60	0.71	
					76	0.73	
1989		3	3.4	0.15	0	2.9	38
					15	1.8	
					30	1.2	
					45	0.88	
					60	0.40	
1989		2	3.4	0.15	45	0.81	38
					60	0.23	
					90	0.14	
					120	0.08	
					150	<0.05	
					166	0.07	
1989		2	3.4	0.15	45	0.86	38
					60	0.2	
					90	0.21	
					120	<0.05	
					150	<0.05	
					166	<0.05	
1989		2	3.4	0.15	45	0.78	38
					60	0.39	
					90	0.29	
					120	0.16	
					150	<0.05	
1990	Ethion 4	3	3.4	0.15	0	2.1	13
	Miscible				15	1.4	
					30	1.0	
					46	0.64	
					60	0.21	
					90	0.29	
1990		3	3.4	0.15	0	3.1	13
					15	2.3	

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
					30	1.4	
					46	1.0	
					60	0.82	
					90	0.43	
1990		3	3.4	0.15	0	3.8	13
					15	2.2	
					30	1.4	
					46	1.4	
					60	0.81	
					90	<0.05	
1990		2	3.4	0.15	44	0.46	13
					61	0.5	
					92	0.28	
					122	0.3	
					154	0.11	
					184	<0.05	
1990		2	3.4	0.15	44	0.67	13
					61	0.52	
					92	0.29	
					122	0.12	
					154	0.06	
					184	0.08	
1990		2	3.4	0.15	44	0.54	13
					61	0.39	
					92	0.15	
					122	0.10	
					154	0.07	
					184	<0.05	
1984	Ethion 4 EC	1	3.4		0	3.3	42
					7	3.4	
					14	2.4	
					21	2.6	
					28	2.2	
1984		1	3.4		0	3.5	42
					7	2.4	
					14	2.1	
					21	1.1	
					28	0.98	

Oranges. Residue studies involving 2 formulations of ethion were conducted on Valencia oranges grown in the San Joaquin Valley of California (Hinstridge, 1972a). Valencia oranges in Florida received 3 foliar sprays of Ethodan 4 EC as a summer scalicide, an autumn miticide and a post-blooming scalicide in the spring. Valencia oranges have a 12-month growing season, so mature oranges may be present at blooming. Mature oranges were sampled on the same day after the post-blooming application (Shuttleworth, 1971a). Results of these and other US studies (Goldsmith, 1992a; Stenzel, 1992a; Witkonton and Barge, 1985) on oranges treated with Ethion 4 Miscible and Ethion 4 EC and 8 EC are shown in Table 9.

Table 9. Residues of ethion in oranges, USA.

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1972	Ethion 25 Spray	1	5.6	0.1	0	0.75	14
					7	0.29	
					14	0.26	
1972		1	8.4	0.15	0	1.5	14
					7	0.78	
					14	0.52	
1972	Ethion 8 EC	1	5.6	0.1	0	1.4	14
					7	0.66	
					14	0.85	
					21	0.62	
1972		1	8.4	0.15	0	1.5	14
					7	1.0	
					14	1.0	
					21	0.64	
1970	Ethodan 4 EC	3	2.2, 2, 1.8		0	0.42	31
1970		3	1.8, 1.8, 1.1		0	0.6	31
1984	Ethion 4 EC	3	6.3*		0	0.83	42
					7	0.48	
					14	0.56	
					21	0.51	
1984		3	6.3*		0	0.64	42
					7	0.59	
					14	0.34	
					21	0.10	
1984	Ethion 8 EC	4	6.3		0	2.7	42
					7	3.5	
1984		4	6.3		0	1.6	42
					7	1.2	
					14	1.0	
					21	1.1	
1990	Ethion 4	3	3.4	0.15	15	1.2	11
	Miscible				30	1.6	
					60	1.1	
					191	0.34	
1990		4	3.4	0.15	0	2.9	11

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
					7	1.4	
					14	2.7	
					21	1.0	
1990		3	3.4	0.15	15	1.2	11
					30	1.2	
					60	0.91	
					191	<0.05	
1990		4	3.4	0.15	0	2.6	11
					7	2.2	
					14	1.6	
					21	1.2	
1990		3	3.4	0.15	15	2.3	11
					30	1.8	
					60	1.6	
					191	0.2	
1990		4	3.4	0.15	0	3.8	11
					7	2.1	
					14	0.95	
					21	1.8	
1989		3	3.4	0.15	0	1.5	35
					15	0.97	
					30	1.2	
					45	1.8	
					60	0.93	
1989		2	3.4	0.15	70	0.07	35
					90	<0.05	
					120	<0.05	
					150	<0.05	
1989		3	3.4	0.15	0	1.4	35
					15	1.4	
					30	1.5	
					45	1.0	
					60	0.95	
1989		2	3.4	0.15	60	0.37	35
					90	0.19	
					120	0.16	
					150	0.11	

Year	Form	Application			PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl			
1989		3	3.4	0.15	0	2.5	35
					15	2.2	
					30	1.6	
					45	1.3	
					60	1.7	
1989		2	3.4	0.15	60	0.44	35
					90	0.22	
					120	0.29	
					150	0.20	
1989		3	3.4	0.18	0	3.4	35
					15	2.1	
					30	1.4	
					45	0.77	
					60	0.71	
1989		2	3.4	0.18	60	0.66	35
					90	0.29	
					120	0.19	
					150	0.12	

* Foliar aerial application

Tangerines and tangelos. The results of trials on tangerines and tangelos (Witkonton and Barge, 1985) are shown in Tables 10 and 11.

Table 10. Residues of ethion in tangerines, USA, 1984.

Form	Application		PHI, days	Residue, mg/kg	Ref.
	No	kg ai/ha			
Ethion 4 EC	3	3.4	0	2.6	42
			7	2.4	
			14	2.4	
			21	1.6	
	3	3.4	0	1.9	42
			7	1.9	
			14	2.0	

Form	Application		PHI, days	Residue, mg/kg	Ref.
	No	kg ai/ha			
			21	2.2	
	3	3.4	0	2.5	42
			7	1.4	
			14	1.1	
			21	1.0	

Table 11. Residues of ethion in tangelos, USA, 1984.

Form	Application		PHI, days	Residue, mg/kg	Ref.
	No	kg ai/hl			
Ethion 4 EC	3	3.4	0	2.7	42
			7	2.3	
			14	2.8	
			21	1.8	
	3	3.4	0	2.3	42
			7	1.6	
			14	1.2	
			21	1.3	
	3	3.4	0	1.9	42
			7	2.6	
			14	1.7	
			21	1.5	

Citrus residue distribution study. In addition to the analyses of whole citrus fruit, subsamples from four Florida plots (2 orange, 1 grapefruit, 1 lemon) and one California plot (lime) were analysed to determine the distribution of residues between the edible pulp and inedible peel. Samples from the 0- and 21- or 0- and 28-day PHIs were examined. These analyses revealed that ethion and its two oxygen analogue metabolites remained in the peel (Witkonton, 1985b; Witkonton and Barge, 1985).

In two of the above trials, a 0-day sample yielded trace total ethion residues above the LOD of 0.02 mg/kg in the pulp (0.04 and 0.05 mg/kg) which accounted for about 2% of the total residue in the

whole fruit. Since the residues were found in only two of the five 0-day pulp samples, it is likely that they were the result of contamination by hand-peeling rather than of actual transfer of residue through the peel. The fact that no residue above the LOD was found in the pulp from the longest pre-harvest intervals (21 or 28 days), further supports the conclusion that no inward movement of residue from peel to pulp occurred. Results are shown in Table 12.

Table 12. Distribution of residues of ethion in citrus, USA (refs. 40,42).

Year	Form	Application		PHI, days	Sample	Residue, mg/kg
		No	kg ai/ha			
1984	Ethion 4 EC	3	3.4	0	orange	2.1
				7		2.0
				14		1.9
				21		1.8
				0	pulp	0.02
				21		0.02
				0	peel	6.6
				21		5.6
	Ethion 8 EC	4	6.3	0	orange	2.6
1985				7		3.2
				14		1.1
				21		1.7
				0	pulp	0.05
				21		0.02
				0	peel	4.2
				21		5.0
1984	Ethion 4 EC	3	3.4	0	grapefruit	1.6
				7		1.6
				14		0.85
				21		1.3
				0	pulp	<0.02
				21		<0.02
				0	peel	4.2
				21		3.8
1984	Ethion 4 EC	1	3.4	0	lemon	2.5
				7		1.7
				14		1.1
				21		1.2
				28		0.78
				0	pulp	0.02
				28		<0.02
				0	peel	4.9
				28		4.0
1984	Ethion	1	6.3	0	lime	1.9
				7		1.4

Year	Form	Application		PHI, days	Sample	Residue, mg/kg
		No	kg ai/ha			
	8 EC			14		0.97
				21		1.3
				28		0.98
				0	pulp	0.04
				28		0.02
				0	peel	4.8
				28		5.8

Cucurbits. Cucumber, melon and summer squash samples from 12 residue trials were analysed after single foliar applications of Ethion 8 EC (Martin, 1985b). See Tables 13-15.

Table 13. Residues of ethion in cucumbers treated with Ethion 8 EC, USA, 1984 (Ref. 27).

Application		PHI, days	Residue, mg/kg
No	kg ai/ha		
1	1.1	0	2.8
		3	0.24
		7	0.22
1	1.1	0	0.44
		3	0.12
		7	0.04
1	1.1	0	0.35
		3	0.24
		7	0.42
1	1.1	0	0.30
		3	0.17
		7	0.03

Table 14. Residues of ethion in summer squash treated with Ethion 8 EC, USA, 1984 (Ref. 27).

Application		PHI, days	Residue, mg/kg
No	kg ai/ha		
1	1.1	0	0.64
		3	0.08
		7	0.02
1	1.1	0	0.16

Application		PHI, days	Residue, mg/kg
No	kg ai/ha		
		3	0.11
		7	0.05
1	1.1	0	0.86
		3	0.18
		7	0.05
1	1.1	0	0.18
		3	0.10
		7	0.04
1	1.1	0	0.16
		3	0.11
		7	0.05

Table 15. Residues of ethion in melons and cantaloupes treated with Ethion 8 EC, USA, 1984 (Ref 27).

Application		PHI, days	Residue, mg/kg
No	kg ai/ha		
1	1.1	0	0.58*
		3	0.20
		7	0.17
1	1.1	0	0.72*
		3	0.67
		7	0.58
1	1.1	0	0.48*
		4	0.40
		7	0.30
1	1.1	0	1.2**
		3	0.18
		7	0.19

* Melons
** Cantaloupes

Animals

Cows. A feeding study on dairy cows was conducted using unlabelled ethion. Groups of four mature Holstein cows were fed ethion at 5, 10 or 20 ppm in their daily feed ration for 30 consecutive days. Milk samples were taken periodically during the feeding period. At the end of the 30-day feeding period, three cows from each group were slaughtered and tissue samples taken. A 30-day recovery period followed during which milk samples were collected at weekly intervals. At the end of the recovery period the remaining cows were killed and tissue samples taken.

Ingestion of the test material produced no mortalities or abnormal behavioral reactions during the test period. Food consumption, milk production and gross pathological findings were normal for all animals.

The report on the feeding portion of this study was independently audited and found to be complete and valid (Ellison, 1981a). Milk analyses showed only ethion as a residue (residues of ethion mono-oxon metabolite <0.005 mg/kg, ethion dioxon <0.01 mg/kg). Residues of ethion in milk occurred rapidly, reached a maximum in 4 to 8 days and remained relatively constant thereafter. The highest ethion residue found in milk from the 20 ppm feeding level was 0.034 mg/kg (0.17% transfer) and occurred at the 4-day interval.

Analysis of tissue samples also showed only ethion as a residue. Liver and kidney showed no detectable ethion (<0.005 mg/kg) at any feeding level and none was detected in muscle at the 5 or 10 ppm levels, while a maximum of 0.008 mg/kg (0.04% transfer) was found at 20 ppm. Fat contained ethion at all feeding levels and showed a maximum 0.22 mg/kg (1.1% transfer) at 20 ppm. Results are shown in Table l6 (Shuttleworth, 1971b).

Table 16. Residues of ethion in cow milk and tissues, USA 1971 (Ref. 32).

Sample	Application, ppm in diet	Days from start	Residue, mg/kg
Milk	5	0	<0.005
		2	<0.005
		4	<0.005
		8	0.006
		14	<0.005
		22	<0.005
		30	<0.005
	recovery period	37	<0.005
		44	<0.005
		51	<0.005
		60	<0.005
Milk	20	0	<0.005
		2	0.006
		4	0.018*
		8	0.018+
		14	0.012
		22	0.016
		30	0.013
	recovery period	37	<0.005
		44	<0.005
		51	<0.005
		60	<0.005
Muscle	5	30	<0.005
	10	30	<0.005
	20	30	0.008
Kidney	5	30	<0.005
	10	30	<0.005
	20	30	<0.005
Liver	5	30	<0.005
	10	30	<0.005
	20	30	<0.005
Fat	5	30	0.014
	10	30	0.10
	20	30	0.18**

* milk residue day 4: 0.018 mg/kg, average of 0.012, 0.02, 0.034, 0.006
\+ milk residue day 8: 0.018 mg/kg, average of 0.022, 0.018, 0.019, 0.011
** fat residue: 0.18 mg/kg, average of 0.22, 0.18, 0.15

Poultry. White leghorn hens were fed unlabelled ethion at 10 ppm in their daily feed ration for nine weeks. Egg samples collected from day 54 to 60 of the feeding study were analysed. Tissue samples (gizzard, liver, muscle and fat) were analysed at the end of the 9-week period. No ethion (<0.01 mg/kg) was found in any of the egg or tissue samples analysed (Shuttleworth, 1969).

The report on the feeding portion of this study was independently audited and found to be complete and valid (Ellison, 1981b).

Table 17. Residues of ethion in eggs and chicken tissues after feeding ethion technical ai at 10 ppm in diet, USA, 1969.

Days from start	Residue, mg/kg
54-60	eggs: <0.01
63	liver: <0.01
63	gizzard: <0.01
63	fat: <0.01
63	muscle: <0.01

FATE OF RESIDUES

Nomenclature of metabolites

Ethion mono-oxon

Structural formula:

Ethion dioxon

Structural formula:

diethyl hydrogen phosphate	ESOP
O,O′-diethyl hydrogen phosphorothionate	ESOP
O,O′-diethyl hydrogen phosphorodithioate	ESSP
O,O′-diethyl S-[(methylthio)methyl] phosphorodithioate	FMC 78153

In animals

Goats. Two metabolism studies have been conducted in goats. In the first study, for each of seven consecutive days, two lactating goats were orally administered [^{14}C]ethion labelled in the methylene group at a rate equivalent to 1 ppm in the diet, corresponding to an estimated daily intake of 45-70 mg/kg bw. Each dose contained ca. 0.5 mCi of [^{14}C]ethion. The goats were slaughtered within 24 hours of the final dose. Milk, sampled daily during dosing, and tissues (liver, muscle, kidney, heart and fat) were assayed for total ^{14}C residues. The maximum radiocarbon residue in milk was 1.5 mg/kg, expressed as ethion, at day 6 of dosing. The maximum residues in tissues were 14.7 mg/kg in liver, 1.3 mg/kg in muscle, 9.7 mg/kg in kidney, 1.6 mg/kg in heart and 0.2 mg/kg in fat (Jobsis and Zietlow, 1985).

Analysis of milk samples showed that 50% of the ^{14}C was in the casein and 10% in lactose, indicating substantial degradation of the parent molecule and incorporation into natural products. Hexane/acetone and methanol extraction of tissues followed by protease digestion released 77-93% of the radioactivity. Most of these residues were polar, water-soluble and volatile. Low levels of the parent chemical (<0.01-0.03 mg/kg) were detected in milk and tissues. Ethion mono-oxon and dioxon were not detected at any significant levels.

In the second study, two lactating goats were again treated with [^{14}C]ethion, but labelled in the ethyl groups, for seven days. This time the nominal treatment levels were equivalent to 25 ppm in the feed (Gohre, 1988).

Representative urine and faeces samples were analysed for ethion and its related metabolites. The major components identified in the excreta were unchanged ethion and its oxidation and cleavage products EOOP, ESOP and ESSP. Ethion mono-oxon, ethion dioxon, and FMC 78153 were detected at low levels. 95% of the total residue in urine was identified as the acid metabolites. Most of the residue in the faeces was identified as ethion or the oxygenated acid metabolites ESOP and EOOP, with the remainder consisting of ESSP, FMC 78153 and several other fractions (all <5% of the total residue). The metabolism of ethion in goats and rats was similar since the principal metabolites identified in goat and rat excreta were the same (Gohre, 1991a).

The pattern of ^{14}C distribution in milk (total residue 0.31 mg/kg ethion equivalents) showed that the majority of the radioactivity (ca. 75% of the total residue) was incorporated into individual fatty acids, which were labelled according to the normal fatty acid distribution found in goat milk. The milk contained minimal amounts of radioactivity in metabolites structurally related to ethion (ca. 4% of the total) and small proportions were incorporated into the major milk sugar, lactose (ca. 9% of the total) or were associated with protein (5%). Incorporation of the radiolabel into natural constituents resulted from desethylation of ethion to release radiolabelled ethanol which entered intermediary metabolism.

The distribution pattern of ^{14}C residues in peritoneal fat (total residue 0.51 mg/kg) was similar to that in milk. About 75% of the total residue was incorporated into the longer chain fatty acids generally associated with triglyceride storage. A significant amount (ca. 18% of the total residue) was associated with bioaccumulated ethion.

In liver most of the extractable activity (ca. 32% of the total residue) was associated with the acid metabolites ESSP, ESOP and EOOP, with <2% of the total associated with ethion and related nonpolar metabolites. The high proportion of acid metabolites and the low levels of nonpolar compounds related to ethion suggest that ethion is metabolized efficiently and extensively by the liver. The high proportion of bound residues (54% of the starting radioactivity) is consistent with the ability of ethion as an OP insecticide to bind to esterase enzymes. The release of bound residues with an alkaline phosphatase enzyme and identification of EOOP as the major released product (ca. 14% of the total residue) agreed with this observation. Some incorporation of the label into natural products was observed (lipids ca. 2.3% of the total residue; sugars ca. 6.6%). Incorporation occurs by the release of [^{14}C]ethanol which enters intermediary metabolism as labelled acetate.

Incorporation of the label into natural products in the liver was less extensive and less specific than in milk (where >90% of the label was incorporated into lactose, lipids, and proteins, all molecules of nutritional significance). Several polar metabolites (released from the bound residues, 4.1-8.4% of the total residue) remained unidentified after extensive analyses (Gohre, 1991b).

In kidney ca. 66% of the total residue was identified as the acid metabolites EOOP, ESOP and ESSP. This is consistent with the metabolite profile in urine (where ca. 95% of the total ^{14}C was associated with acid metabolites) since the kidneys function as the major excretory organ. The small amount of ethion and related nonpolar metabolites (<1% of the total residue) confirms that ethion is extensively metabolized. As with liver, a proportion of the bound activity was released with alkaline phosphatase and identified as EOOP and ESOP.

The residue distributions in goat heart and loin muscle were very similar. There was little bioaccumulated ethion (7 and 8.7% of the total residue respectively), and only minor amounts of extractable acid metabolites were identified (ca. 15 and 16% of the total in heart and loin muscle respectively). The majority of the activity (48 and 42% respectively) was in unextractable bound residues. The low specific activity of the tissues prevented further analysis.

The levels of unidentified and/or bound tissue residues in liver, kidney, heart, and loin muscle were not significant, and would be much lower from the ethion residues expected to result from normal agricultural practice. The unidentified residues in liver ranged from 0.06 to 0.32 mg/kg. In kidney, heart and loin muscle the unidentified residues ranged from 0.001 to 0.026 mg/kg. The unextractable or unreleased bound residues in liver, kidney, heart and loin muscle were 0.2, 0.025, 0.038, and 0.014 mg/kg respectively. In general, residues that were extractable were either very polar (acid metabolites) or probably incorporated at low specific activity into natural products. The overall metabolic pattern suggested by these studies is that the minor amounts of activity retained in the goat tissues (ca 1.1% of the total dose) or excreted in the milk (ca. 1% of the total dose) are the result of extensive metabolism of ethion to the acid metabolites and incorporation into natural products. The activity in the excreta (majority of the dose) was associated with ethion or the acid metabolites.

Poultry. Laying hens were dosed orally with [^{14}C]ethion labelled in the methylene group at a rate equivalent to 2 ppm in the diet for 10 consecutive days. This corresponded to an estimated intake of 17 mg/kg bw/day. Eggs (sampled daily) were separated into yolks and whites. Tissues (liver, muscle, fat) were sampled at slaughter. Samples were assayed for total ^{14}C residues (expressed as mg/kg ethion equivalents). The maximum ^{14}C residues in egg yolk and white were 1.2 mg/kg at day 9 and 0.2 mg/kg at day 4 respectively. Total ^{14}C residues in tissues amounted to 3.1 mg/kg in liver, 0.2 mg/kg in muscle and 0.1 mg/kg in fat (Bodden and Zietlow, 1985).

The analysis of tissues and eggs by gas and thin-layer chromatography showed that residues of ethion, ethion mono-oxon, and ethion dioxon were below 0.01 mg/kg. Organosoluble residues contained 5 to 40% of the total tissue ^{14}C. Water-soluble residues accounted for 7-70%, while non-extractable radiocarbon ranged from 13 to 60%. The extractable residues were polar and some fractions were volatile. In liver, one volatile component was isolated and identified as [^{14}C]formaldehyde (Bodden and Zietlow, 1985).

In plants

Citrus fruits. In an outdoor study, orange leaves and immature and mature Valencia oranges were treated with [^{14}C]ethion labelled in the methylene group formulated as an emulsifiable concentrate and diluted with water to an approximate concentration of 0.045 kg ai/hl. The material was applied uniformly with a paint brush. Oranges harvested immediately after treatment showed total ^{14}C residues of 1.9-5.8 mg/kg (ethion equivalents). These declined slowly to 1.1-2.3 mg/kg by 90 days after application. Radiocarbon residues were almost entirely (>99%) in the peel. The total ^{14}C in or on the leaves was higher than in the fruit, declining from 86 mg/kg at day 0 to 1.4-5.2 mg/kg by day 90. No significant levels (<0.01-0.02 mg/kg ethion equivalents) of ^{14}C were translocated into the fruit from the treatment of adjacent leaves, indicating that ethion and its degradation products were essentially non-systemic (Patterson, 1985).

Extractable residues accounted for ca. 90% of the total recovered ^{14}C in orange peel at all pre-harvest intervals. Organosoluble (in ethyl acetate) and water-soluble residues also remained constant at about 80% and 10% of the total ^{14}C over all intervals. Ethion was the predominant product in the ethyl acetate fractions (90% of the organosoluble residues). Ethion mono-oxon and ethion dioxon increased from 1% and <1% respectively at day 0 to 11% and 4% by day 90. The results indicated that ethion degradation on oranges may be attributable to environmental factors (i.e. weathering) rather than metabolism.

The breakdown of ethion on citrus leaves was faster than on fruit. Extractable ^{14}C residues declined markedly from 97% of the total recovered ^{14}C at day 0 to 60% by day 90, and organosoluble ^{14}C decreased from ca. 90% to 44% during the same period. Water-soluble radiocarbon increased to 16% by day 90. Ethion declined from 90% of the organosoluble fraction at day 0 to 62% at day 90, while ethion mono-oxon and dioxon increased from 2% and 1% respectively to 27% and 4% (Selim, 1985).

A study of the extractability of plant-incorporated residues from mature oranges treated with ^{14}C ethion revealed that 90% of the total residue could be extracted with acetone (Arabinick and Witkonton, 1985).

In processing

Apples. McIntosh apples treated with four applications of Ethion 8 EC at 4.5 kg ai/ha were harvested 30 days after the last application. The mature fruit was processed in accordance with commercial procedures on a pilot-plant scale. The unwashed whole fruit and the processed fractions were analysed for ethion, ethion mono-oxon, and ethion dioxon. No ethion residue was transferred from apples to cider, but there was a 4.1- and 13-fold concentration into wet pomace and dry pomace respectively. Ethion accounted for about 98% of the total residue found in whole apples and apple products and ethion mono-oxon for the remaining 2%. No dioxon was found (Shuttleworth, 1972).

Citrus fruits. In addition to the residue distribution study (Table 12) the results of further studies were

made available.

Florida Valencia oranges sprayed with Ethion 4 Miscible were processed by commercial procedures. The mature oranges were treated with a single application of Ethion 4 Miscible at 0.045 kg ai/hl and 5 kg ai/ha. The mature fruit was harvested seven days after treatment, then processed on a pilot-plant scale. The citrus fractions analysed for ethion, ethion mono-oxon and ethion dioxon included unwashed fruit, washed fruit, finisher pulp, dry pulp, syrup, juice and oil (Witkonton and Arabinick, 1985).

A total ethion residue (ethion plus mono-oxon and dioxon) of 0.84 mg/kg was found on unwashed whole oranges. This residue was concentrated 11 times in orange oil and 4.3 times in dry pulp, but decreased to 0.25-fold in syrup and 0.02-fold in finisher pulp. No ethion or oxygen metabolite residue was found in the orange juice (<0.02 mg/kg). Ethion parent compound accounted for 85 to 90% of the total residue in whole oranges and their processed products. The only metabolite detected was ethion mono-oxon (the limit of detection for the dioxon was 0.02 mg/kg). Results are shown in Table 18.

Table 18. Residues of ethion in oranges and orange processing fractions from a supervised processing trial in the USA, 1984 (Witkonton and Arabinick, 1985). Ethion (4EC) was applied once to Valencia orange trees at 5 kg ai/ha at a spray concentration of 0.05 kg ai/hl. Oranges were harvested 7 days later and processed.

Commodity	Residues, mg/kg
Fruits, unwashed	0.76
Fruits, washed	0.64
Juice	<0.02
Syrup	0.21
Dry pulp	3.3
Finisher pulp	0.02
Oil	7.7

Grapes. Thomson seedless grapes from 4 US trials which had been treated with 2 foliar applications of Ethion 25 Spray (WP) were subsampled in the field after 30 days and dried into raisins. Grape, raisin and raisin waste samples were analysed (Leppert, 1986). Results of these and older trials (Hinstridge, 1972d) are shown in Table 19. The average the total residue showed a maximum concentration into raisins of 2.7 times and into raisin waste of 9.9 times (see Table 20).

Table 19. Residues of ethion in grape processing, USA.

Year	Form	Application			Sample	PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl				
1984	Ethion 25 WP	2	2.2		grapes	0	16	25
						10	6.9	
						20	5.6	
						30	4.4	
						40	3.9	

Year	Form	Application			Sample	PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl				
					raisins	30	7.2	
1984		2	2.2		grapes	0	14	25
						10	6.4	
						20	5.9	
						30	2.5	
						40	2.2	
					raisins	30	4.8	
1985		2	2.2		grapes	0	2.1	25
						10	1.2	
						20	0.71	
						30	0.57	
						40	0.44	
					raisins	30	0.69	
					raisin waste	30	1.9	
1985		2	2.2		grapes	0	2.5	25
						10	1.1	
						20	0.39	
						30	0.47	
						40	0.57	
					raisins	30	1.1	
					raisin waste	30	1.9	
1971	Ethion 4 EC	2	1.5	0.06	grapes			17
						32	0.83	
					wet pomace	32	2.6	
					dry pomace	32	6.1	
					raisin stems	32+35	2.0	
					raisins	32+35	9.1	
1971	Ethion 25 WP	2	1.5	0.06	grapes	30	1.1	17
					raisin stems	30+18	0.41	
					raisins	30+18	0.30	
1971	Ethion 8 EC	2	1.5	0.06	grapes	30	0.34	17
					raisin stems	30+18	0.43	
					raisins	30+18	0.36	

Table 20. Concentration factors for residues of ethion in grape processing.

Trial No.	Average residue, mg/kg	Concentration factor	Ref.

	Grapes	Raisins	Raisin waste	Raisins	Raisin waste	
2	4.7	9.5	-	2	-	25
3	2.7	6.4	-	2.3	-	
6	0.62	0.96	3.9	1.6	6.4	
7	0.53	1.4	5.3	2.7	9.9	
Average			2.15	8.15		

Maize. Maize plots in New York, Colorado and Nebraska were sprayed with Ethodan 4 EC using a boom sprayer in New York and Nebraska and aerial spraying in Colorado (Shuttleworth, 1971c). Trials by Hinstridge (1969) were undertaken to obtain residue data on maize and maize silage. In 6 other trials across the USA maize was treated with a single application of Ethion 4 or 8 EC after the ear formation stage. Maize ears were sampled 50 days later at normal harvest (Barrett, 1986). Results are shown in Table 21.

Table 21. Residues of ethion in maize forage, USA.

Year	Form	Application			Sample	PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl				
1970	Ethodan 4 EC	1	1.1*			0	17	33
						7	4.4	
						13	2.4	
						21	1.3	
1970		1	1.1**			0	1.3	33
						7	1.1	
						14	0.45	
						21	0.96	
1970		1	1.1*			0	12	33
						21	0.49	
1967	Ethion 4 EC	1	1.1	0.25	silage	22	3.9	18
						29	1.6	
						36	0.9	
						43	1.1	
					dry stalk	64	5.2	
					dry husk	64	0.28	
					dry kernel	64	<0.05	
					dry cob	64	<0.05	
1967		1	2.2	0.5	silage	22	9.8	18
						29	7.1	
						36	7.2	
						43	6.6	
					dry stalk	64	20	
					dry husk	64	0.36	
					dry kernel	64	<0.05	
					dry cob	64	<0.05	
1967		1	1.1	0.25	silage	0	35	18
						7	13	
						14	8.6	
						21	11	
					dry stalk	42	13	
					dry husk	42	2.6	
					dry kernel	42	<0.05	
					dry cob	42	<0.05	
1967		1	2.2	0.5	silage	0	50	18

Year	Form	Application			Sample	PHI, days	Residue, mg/kg	Ref.
		No	kg ai/ha	kg ai/hl				
						7	27	
						14	21	
						21	11	
						42		
					dry stalk		1.8	
					dry husk	42	3.8	
					dry kernel	42	<0.05	
					dry cob	42	<0.05	
1967		2	1.1	0.25	dry stalk		30	18
					dry husk	21	11	
					dry kernel	21	<0.05	
					dry cob	21	<0.05	
1967		2	2.2	0.5	dry stalk	21	75	18
					dry husk	21	36	
					dry kernel	21	<0.05	
					dry cob	21	0.06	
1967		1	1.1**	1.25	silage	0	14	18
						7	3.5	
						14	0.36	
						50	0.38	
1985	Ethion 4	1	1.1*			50	<0.01	3
1985	Miscible	1	1.1*			51	<0.01	3
1985		1	1.1*			52	<0.01	3
1985		1	1.1*			56	<0.01	3
1985		1	1.1*			50	<0.01	3
1985	Ethion 8 EC	1	1.1*			50	<0.01	3

* Foliar spray, ground
** Foliar spray, aerial

Stability of residues in stored analytical samples

A frozen storage stability study was conducted at -18°C with laboratory-fortified samples. Apples, oranges, grapes, cucumbers, maize silage, maize stover and grain, as well as a variety of soil types (sandy loam, silt loam and sandy clay loam), were included in the study. Samples were fortified with ethion, ethion mono-oxon and ethion dioxon and analysed after 0, 3 and 6 months storage.

The results showed ethion and its oxygen analogue metabolites to be stable in the freezer for at least six months. The residue found in a given matrix varied between sampling intervals, but did not show any consistent pattern of decline in crop or soil samples. The maximum variation for a given matrix was

less than 25% (Martin, 1985a).

In additional studies the stability of ethion and its metabolites was determined in various crops and soils for periods up to 38 months (Akari, 1989; Kaiser, 1992).

Results are shown in Table 22.

Table 22. Storage stability of ethion in or on various crops and soils, USA.

Year	Sample	Application		Storage period, months	Residue, mg/kg	Ref.
		No	mg/kg			
1990-1991	Grapefruit	1	2	0	1.7	22
				3	1.8	
				9	1.6	
				15	1.9	
1990-1991	Lemon	1	2	0	1.7	22
				3	1.9	
				9	1.8	
				15	1.8	
1985-1988	Apple	1	2	0	2.0	1/26
				3	1.7	
				6	1.7	
				16	1.9	
				26	2.0	
				38	2.3	
1985-1988	Orange	2		0	2.0	1/26
				3	1.5	
				6	1.8	
				16	2.2	
				26	2.3	
				38	2.0	
1985-1988	Grape	1	2	0	2.0	1/26
				3	1.9	
				6	1.8	

Year	Sample	Application		Storage period, months	Residue, mg/kg	Ref.
		No	mg/kg			
				16	1.9	
				26	2.2	
				38	2.1	
1985-1988				0	0.5	
				3	0.43	
				6	0.46	
				16	0.52	
				26	0.55	
				38	0.52	
1985-1988	Cucumber	1	0.5	0	0.5	1/26
				3	0.43	
				6	0.46	
				16	0.52	
				26	0.55	
				38	0.52	
1985-1988	Maize silage	1	5	0	5.0	1/26
				3	3.8	
				6	5.8	
				16	4.7	
				26	5.3	
				38	5.7	
1985-1988	Maize stover	1	5	0	5.0	1/26
				3	4.8	
				6	4.8	
				16	6.1	
				26	5.0	
				38	4.2	
1985-1988	Maize grain	1	0.5	0	0.50	1/26

Year	Sample	Application		Storage period, months	Residue, mg/kg	Ref.
		No	mg/kg			
				3	0.54	
				6	0.45	
				16	0.57	
				26	0.60	
				38	0.53	
1985-1988	Sandy loam soil	1	0.5	0	0.50	1/26
				3	0.47	
				6	0.41	
				16	0.51	
				26	0.59	
				38	0.59	
1985-1988	Silt loam soil	1	0.5	0	0.50	1/26
				3	0.47	
				6	0.43	
				16	0.41	
				26	0.55	
				38	0.56	
1985-1988	Sandy clay loam soil	1	0.5	0	0.50	1/26
				3	0.50	
				6	0.52	
				16	0.60	
				26	0.53	
				38	0.55	

Residues in the edible portion of food commodities

See Table 12.

METHODS OF RESIDUE ANALYSIS

Apples and pears. For the determination of ethion and its oxygen analogue metabolites apple and pear samples were extracted with acetone, and the extract diluted with water and cleaned up by dichloromethane partition. Quantification was accomplished by gas chromatography employing a 530 m capillary column for apples and a 2 mm packed column for pears with a methyl silicone stationary phase and a nitrogen-phosphorus detector. Processed apple products, except cider, were extracted with acetone, diluted and partitioned as before; the residue was transferred into hexane and partitioned into acetronitrile. Cider was partitioned with dichloromethane and the residue transferred via hexane into acetonitrile. Quantification was by gas chromatography with a packed methyl silicone column and a flame-photometric detector.

The analytical methods used for determining ethion residues in apples, pears and processed apple products were validated by spiking untreated fruit or processed products with known levels of ethion, ethion mono-oxon, and ethion dioxon before the initial solvent blending. Average recoveries from apples (Leppert, 1985a) and pears (Leppert, 1985b) ranged from 83 to 103% depending on which of the three compounds was determined. Recoveries from processed apple fractions (Shuttleworth, 1972) varied from 65 to 123% depending upon the sample analysed and the compound determined.

The limit of determination was assessed on the basis of the lowest fortification level from which an adequate recovery was obtained. The limit of detection was defined as the sample size giving the minimum visual recognition of detector response. For apples and pears, the limit of determination was established as 0.1 mg/kg and the limit of detection as 0.05 mg/kg for ethion, ethion mono-oxon and ethion dioxon. For processed apple products, limits of determination ranged from 0.02 mg/kg in cider to 0.5 mg/kg in dry apple pomace for ethion and ethion mono-oxon. The limit of determination for ethion dioxon ranged from 0.04 mg/kg in cider to 1.25 mg/kg in dry apple pomace. The limits achieved for the processed fractions were more than adequate for the level of residues being measured.

Citrus fruits. Samples were prepared for the determination of ethion and its oxon metabolites by acetone extraction, solvent evaporation, aqueous dilution, dichloromethane partition and silica-gel Sep-Pak clean-up of the dichloromethane extract. Quantification was by gas chromatography on a 530 m capillary methyl silicone column attached to a nitrogen-phosphorus detector (Witkonton, 1985a).

The analytical methods used in determining ethion residues in or on citrus were validated (Kaiser, 1991b) by spiking untreated fruit and processed products with known levels of ethion, ethion mono-oxon, and ethion dioxon before the initial solvent blending. Average recoveries ranged from 73 to 110% depending on the sample analysed and the compound determined.

Limits of determination and detection were established and defined as above. A limit of determination of 0.1 mg/kg was found for ethion, ethion mono-oxon and ethion dioxon in citrus fruits and all processed products except citrus oil (0.5 mg/kg). The limit of detection was 0.02 mg/kg for ethion and its two oxons in all citrus samples except citrus oil, for which it was 0.1 mg/kg.

The efficiency of extraction of ethion and all of its degradation products including its oxygen analogue metabolites from citrus fruits was validated with citrus samples from the outdoor metabolism study which had been treated with [^{14}C]ethion labelled in the methylene group. The citrus fruits were treated with formulated [^{14}C]ethion at a concentration equivalent to 0.045 kg ai/hl and sampled 90 days after treatment. The whole citrus fruit sample was blended with acetone and the extractable ^{14}C radioactivity was determined by radioassay. The total acetone-extractable radioactivity, based on triplicate analyses, averaged 90 $\pm$ 6%.

Animal products. For milk and cow tissue samples an acetone extraction (acetonitrile extraction for fat)

was followed by solvent exchange into hexane, acetonitrile partition, polyethylene alumina column clean-up, aqueous dilution and dichloromethane partition. For liver a Florisil column clean-up was used in place of the polyethylene alumina column. Quantification was by gas chromatography on a packed methyl silicone column connected to a flame-photometric detector.

Eggs and poultry tissues were analysed in the same way as liver except that the initial extraction was with acetonitrile.

For milk and cow tissue analyses (Shuttleworth, 1971b), average method recoveries ranged from 66 to 87% depending on which of the three compounds was determined and which sample was analysed. Eggs and poultry tissues (Shuttleworth, 1969) yielded an overall average method recovery of 77% for ethion.

The limit of determination in milk and meat was 0.005 mg/kg for ethion and ethion mono-oxon and 0.01 mg/kg for ethion dioxon. The limits of detection were one-half of the limits of determination. For eggs and poultry tissues, the limits of determination and detection for ethion were 0.01 mg/kg and 0.005 mg/kg.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Commodity	MRL, mg/kg
Australia	Cattle, fat	2.5
	Citrus fruits	1
	Grapes	2
	Milk	0.5
	Milk products	0.5
	Pome fruits	1
	Stone fruits	1
	Tea, dry manufactured	5
Canada	Apple	2
	Grapes	2
	Peach	1
	Pear	2
	Plum	1
	Strawberry	1
	Onions	n.r.*
Germany	Cattle, fat	2
	Cattle, meat	2
	Citrus fruits	2
	Citrus juices	0.1
	Eggs	0.2
	Goat, fat	0.2
	Goat, meat	0.2
	Grapes	0.5
	Pig, fat	0.2
	Pig, meat	0.2
	Pome fruits	0.5
	Poultry, fat	0.2
	Poultry, meat	0.2
	Milk	0.5
	Sheep, fat	0.2
	Sheep, meat	0.2
	Stone fruits	0.5
	Other fruits	0.1

Country	Commodity	MRL, mg/kg
	Other food commodities of plant origin	0.05
	Vegetables	0.1
	Other food of animal origin	0.01
Netherlands	Citrus fruits	2
	Grapes	0.5
	Pome fruits	0.5
	Stone fruits	0.5
	Other fruits	0.1
	Vegetables	0.1
	Other food commodities	0.02
Spain	Citrus fruits	2
	Cotton	0.1
	Grapes	0.5
	Pome fruits	0.5
	Stone fruits	0.5
USA	Almonds	0.1
	Almonds, hulls	5
	Apple	2
	Apricot	0.1
	Beans	2
	Cattle, fat	2.5
	Cattle, meat by-products	1
	Cattle, meat	2.5
	Cherries	0.1
	Chestnuts	0.1
	Citrus fruits	2
	Cotton, seed	0.5
	Cucumber	0.5
	Egg plant	1
	Eggs	0.2
	Filberts	0.1
	Goat, fat	0.2
	Goat, meat by-products	0.2
	Goat, meat	0.2
	Grapes	2
	Horse, fat	0.2
	Horse, meat by-products	0.2

Country	Commodity	MRL, mg/kg
	Horse, meat	0.2
	Maize, fodder	14
	Maize, forage	14
	Maize, grain	0.1
	Melon	2
	Milk	0.5
	Nectarine	1
	Onion	1
	Peach	1
	Pear	2
	Pecan	0.1
	Pepper	1
	Pig, fat	0.2
	Pig, meat by-products	0.2
	Pig, meat	0.2
	Pimento	1
	Plum	2
	Poultry, fat	0.2
	Poultry, meat byproducts	0.2
	Poultry, meat	0.2
	Sheep, fat	0.2
	Sheep, meat by-products	0.2
	Sheep, meat	0.2
	Sorghum, forage	2
	Sorghum, grain	2
	Squash, summer	0.5
	Strawberry	2
	Tomato	2
	Walnuts	0.1

* negligible residue, residue expected to be <0.1 mg/kg

APPRAISAL

Ethion, previously evaluated for residues by the JMPR in 1968, 1969, 1972, 1975 and 1983, is included in the CCPR periodic review programme.

Ethion is used as an insecticide and acaricide in pre-harvest application on a variety of crops, especially citrus, to control aphids, scales, mites, leaf miners and leaf hoppers. It is formulated as WP, EC or GR and applied between one and four times at a rate of 0.36-2.3 kg ai/ha. Residue data from supervised trials on

apples, pears, plums, grapes, citrus, cucumbers, melons and summer squash have been submitted to the Meeting.

Ethion is essentially non-systemic. Metabolism studies on citrus using radiolabelled ethion have shown the degradation of the parent compound to occur on leaf and fruit surfaces. The oxygen analogue metabolites, ethion mono-oxon and ethion dioxon, were identified products (up to 11% and 4% of the total residue respectively). A metabolism study in goats with labelled ethion showed incorporation of significant levels of radiocarbon into casein and lactose, indicating degradation and reincorporation into natural products, and low to negligible levels of the parent chemical in milk. A study on laying hens using the radiolabelled compound showed residues of ethion and its oxygen analogue metabolites to be less than 0.01 mg/kg in eggs and tissue.

Methods of residue analysis in plants involved solvent extraction with acetone followed by dichloromethane/water partition and silica gel Sep-Pak clean-up of the dichloromethane extract. Quantification was accomplished by gas chromatography on a 530 m capillary methyl silicone column connected to a nitrogen-phosphorus detector. For the analysis of milk, cow and poultry tissues and eggs the sample was blended with acetone (acetonitrile for fat), and transferred to hexane. This was followed by acetonitrile partition, clean-up on a polyethylene alumina column, aqueous dilution and dichloromethane partition. For liver, a florisil column was used in place of the polyethylene alumina column. Quantification was by gas chromatography with a packed methyl silicone column connected to a flame-photometric detector. The limits of determination were 0.005 mg/kg in milk and cattle meat, 0.01 mg/kg in eggs and poultry tissue, 0.05 mg/kg in cucurbits, 0.1 mg/kg in apples and pears and 0.5 mg/kg in dry apple pomace.

There was insufficient information on GAP and residues from supervised trials made available to the Meeting for the following commodities covered by Codex CXLs: almonds, apricots, cherries, chestnuts, common beans, cotton seed, egg plant, garlic, hazelnuts, maize, nectarines, onions, peaches, pecans, peppers, sweet peppers, strawberries, tea, tomatoes, walnuts, winter squash and the meat and edible offals of pigs and sheep. The Meeting therefore proposed withdrawal of the relevant MRLs. Residues in other commodities were evaluated as follows.

Citrus fruits. Ethion residues in citrus fruits are quite persistent and small differences in the PHI have little effect on residue levels. Decreases are probably mostly accounted for by growth dilution.

Numerous US trials according to GAP (3 applications at 3.4 kg ai/ha, with a PHI of 0 days for grapefruit, oranges, tangerines and tangelos and of 15 days for lemons and limes) showed residues which exceeded the present CXL of 2 mg/kg. There were 11 residues in grapefruit over the range 0.62-2.8 mg/kg, 12 in lemons and limes at 1.1-2.9 mg/kg, 6 in oranges at 0.42-3.4 mg/kg and three results each for tangerines and tangelos with a minimum of 1.9 mg/kg and a maximum of 2.7 mg/kg. On the basis of these data the Meeting estimated a maximum residue level of 5 mg/kg for citrus fruits to replace the previous recommendation (2 mg/kg).

The residue in the whole fruit was concentrated primarily in orange oil (11 times) and dry pulp (4.3 times). The residue was reduced in syrup (0.25 times that in the fruits) and finisher pulp (0.02 times). No residues of ethion or its oxygen analogues were found in orange juice (<0.02 mg/kg).

Pome fruits. Results of 9 US trials (6 on apples, 3 on pears) at a maximum rate of 3.4 kg ai/ha, with a 28-day PHI and 3 applications, showed that the present MRL of 2 mg/kg for apples and pears does not suffice since the minimum residue was 0.72 mg/kg and the maximum 3.8 mg/kg. There were no data from trials according to current GAP. The Meeting agreed to withdraw the previous recommendation for apples and

pears.

No residues were transferred from apples to cider, but the residue was concentrated in wet and dry pomace, 4.1 and 13 times respectively.

Plums and prunes. The two trials available did not suffice for an evaluation. The Meeting agreed to withdraw the recommendation for plums (including prunes) of 2 mg/kg.

Grapes. Residues in some samples from 9 supervised US trials with two applications of 2.24 kg ai/ha and a PHI of 30 days exceeded the present MRL of 2 mg/kg, with a range of 0.47-4.4 mg/kg. There were no data from trials according to current GAP. The Meeting agreed to withdraw the previous recommendation.

Raisins. Average total residues in raisins and raisin waste showed maximum concentration factors of 2.7 and 9.9 times respectively. The evaluation of processing studies using an application rate of 2.2 kg ai/ha showed large variations of residues in raisins (minimum 0.69 mg/kg, maximum 7.2 mg/kg). The Meeting could not recommend an MRL for raisins because it had withdrawn the previous recommendation for grapes.

Cucurbits. There were 4 values available for cucumbers and melons, and 5 for summer squash, from US trials based on 3 applications, a 7-day PHI and a application rate of 1.1 kg ai/ha.

Residues in cucumbers ranged from 0.03 to 0.42 mg/kg. Summer squash residues were lower (minimum 0.02 mg/kg, maximum 0.05 mg/kg). In melons the few results were below the previous MRL of 2 mg/kg (minimum O.17 mg/kg, maximum 0.58 mg/kg).

Because of the lack of US GAP the Meeting agreed to withdraw the recommendations for MRLs for cucumbers (0.5 mg/kg), summer squash (0.5 mg/kg) and melons (2 mg/kg).

Meat and edible offal of cattle. Analysis of samples of liver and kidney showed no detectable ethion (<0.005 mg/kg) at feeding levels of 5, 10 and 20 ppm. No ethion was detected in muscle at the 5 and 10 ppm feeding levels, while a maximum of 0.008 mg/kg (0.04% transfer) was found at 20 ppm. Fat from cattle fed at all levels contained ethion and showed a minimum of 0.1 mg/kg and a maximum of 0.22 mg/kg (1.1% transfer) at the 20 ppm feeding level.

Milk. Ethion residues in milk appeared rapidly, peaked in 4 to 8 days, and remained relatively constant thereafter. The highest ethion residue found in milk at the 20 ppm feeding level was 0.034 mg/kg (0.17% transfer) and occurred after 4 days.

Meat and edible offal of poultry. White Leghorn hens were fed unlabelled ethion at 10 ppm in their daily feed ration for nine weeks. Tissue samples (gizzard, liver, muscle and fat) taken after nine weeks of feeding were analysed. No ethion residue (<0.01 mg/kg) was found in any of the samples analysed.
Eggs. Egg samples collected from days 54 to 60 of the feeding study were analysed. No ethion was detected (<0.01 mg/kg).

In the absence of MRLs for animal feed items (and in the case of cattle meat and milks information on veterinary uses) the Meeting agreed to withdraw the previous recommendations for eggs, milks, and the meat and edible offal of cattle, goats, horses, pigs, poultry and sheep.

The Meeting noted that the oxygen analogue metabolites were usually only a minor part of the

residue. The animal transfer study on lactating cows provided evidence that ethion tends to partition into the fat. The Meeting therefore agreed that the residue should continue to be defined as ethion (fat-soluble).

RECOMMENDATIONS

On the basis of the residue data from supervised trials the Meeting concluded that the residue level for citrus fruits shown below is suitable for establishing a maximum residue limit, and that the previous MRLs for the other listed commodities should be withdrawn.

Definition of the residue: ethion (fat-soluble).

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New	Previous	
TN 0660	Almonds	W	0.1*	
FP 0226	Apple	W	2	
FS 0240	Apricot	W	0.1*	
MM 0812	Cattle meat	W	2.5 (fat) V	
MO 0812	Cattle, Edible offal of	W	1	
FS 0013	Cherries	W	0.1*	
TN 0664	Chestnuts	W	0.1*	
FC 0001	Citrus fruits	5	2	0, 15[1]
VP 0526	Common bean (pods and/or immature seeds)	W	2	
SO 0691	Cotton seed	W	0.5	
VC 0424	Cucumber	W	0.5	
VO 0440	Egg plant	W	1	
PE 0112	Eggs	W	0.2*	
VA 0381	Garlic	W	1	
MM 0814	Goat meat	W	0.2* (fat)	
MO 0814	Goat, Edible offal of	W	0.2*	
FB 0269	Grapes	W	2	
TN 0666	Hazelnuts	W	0.1*	
MM 0816	Horse meat	W	0.2* (fat)	
MO 0816	Horse, Edible offal of	W	0.2*	
GC 0645	Maize	W	0.05*	
VC 0046	Melons	W	2	
ML 0106	Milks	W	0.02 F V	
FS 0245	Nectarine	W	1	
VA 0385	Onion, Bulb	W	1	
FS 0247	Peach	W	1	
FP 0230	Pear	W	2	
TN 0672	Pecan	W	0.1*	
VO 0051	Peppers	W	1	
VO 0445	Peppers, Sweet	W	1	
MM 0818	Pig meat	W	0.2* (fat)	
MO 0818	Pig, Edible offal of	W	0.2*	
FS 0014	Plums (including Prunes)	W	2	
PM 0110	Poultry meat	W	0.2* (fat)	
PO 0111	Poultry, Edible offal of	W	0.2*	
MM 0822	Sheep meat	W	0.2* (fat)	

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New	Previous	
MO 0822	Sheep, Edible offal of	W	0.2*	
VC 0431	Squash, Summer	W	0.5	
FB 0275	Strawberry	W	2	
DT 1114	Tea	W	5	
VO 0448	Tomato	W	2	
TN 0678	Walnuts	W	0.1*	
VC 0433	Winter squash	W	0.5	

[1] PHI 0 days: grapefruit, orange, tangerine, tangelo
PHI 15 days: lemon, lime
W: withdrawal

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