ETHEPHON (106)

EXPLANATION

Residue aspects of ethephon were reviewed by the JMPR in 1977, 1978, 1983 and 1985. As an ADI had not been allocated, Guideline Levels were proposed in 1977 and 1978. In view of the time since these proposals, information on current use patterns as well as further residue data and critical supporting studies were required to enable the estimation of maximum residue levels.

USE PATTERN

Ethephon is a systemic plant growth regulator belonging to the phosphonate family. It is readily absorbed by the plant and releases ethylene which is a natural plant hormone. Ethylene influences directly several physiological processes (ripening, maturation etc.) and stimulates the production of endogenous ethylene.

Ethephon is mainly formulated as a soluble concentrate (SL) and sold under several trade names corresponding to specific uses. Concentrations are between 20 and 720 g/l. Combinations with chlormequat chloride are also used for cereals. Formulations are applied as foliar sprays by either ground or aerial equipment.

Ethephon is used on cereals (wheat, barley, rye and rice) to increase resistance to lodging through straw shortening and strengthening, fruits and vegetables to promote fruit maturity (early and uniform ripening and colouring of mature fruits), ornamentals to stimulate flowering and side-branching and to reduce plant height, cotton to promote uniform boll opening and enhance defoliation, and rubber to increase yield (latex flow) and to reduce bark consumption.

Ethephon formulations are registered in more than 60 countries around the world. The national use patterns are summarized in Table 1.

RESIDUES RESULTING FROM SUPERVISED TRIALS

A number of trials were conducted on a wide variety of crops in typical geographical regions. Parent ethephon residues were determined in various crop parts. In addition, analyses for residues of monochloroacetic acid, a potential decomposition product of an impurity (the 2-chloroethyl ester) in technical grade ethephon, were carried out on blackberries (Rhône-Poulenc, 1990c), grapes (Rhône-Poulenc, 1991b), pineapples (Rhône-Poulenc, 1992l), tomatoes, (Rhône-Poulenc, 1992o) and cantaloupes (Rhône-Poulenc, 1990d). Residues of monochloroacetic acid were below the limit of determination (<0.01 mg/kg) in all samples.

In the Tables summarizing the experimental results and national MRLs the countries are indicated by the following codes: ARG-Argentina, AUL-Australia, AUS-Austria, BEL-Belgium, BRA-Brazil, CAN-Canada, COS-Costa Rica, ECU-Ecuador, FIN-Finland, FRA-France, GER-Germany, GUA-Guatemala, HUN-Hungary, ISR-Israel, ITA-Italy, JPN-Japan, NET-Netherlands, NOR-Norway, NZE-New Zealand, PER-Peru, SAF-South Africa, SPA-Spain, SWI-Switzerland, TUN-Tunis, TUR-

Turkey, UK-United Kingdom, USA-United States of America, VEN-Venezuela.

Table 1. Registered or approved uses of ethephon.

Crop	Country	Application			PHI, (days)
		Form., concn.[1]	kg ai /ha	g ai/100 l	
Almonds	Chile	Ethrel 480			
Apple	Argentina	Ethrel 480			15
Apple	Australia	Ethrel 480		24-96	7
Apple	Austria	Ethrel 39.5%		19.75	
Apple	Belgium	Ethrel F10 100 g/l		50	10-14
Apple	Canada	Ethrel 480	0.84-1.68		
Apple	Chile	Ethrel 480			
Apple	Cyprus	Ethrel 480			
Apple	Denmark	Ethrel 480		24	5
Apple	Ecuador	Ethrel 480			24-21
Apple	Egypt	Ethrel 480			
Apple	Greece	Ethrel 38.0%		28.5-57	
Apple	Israel	Ethrel 480			
Apple	Italy	Ethrel 39.5%			7
Apple	Norway	Ethrel 480			
Apple	Spain	Fruitel 480		36-72	10
Apple	Sweden	Ethrel 100			7
Apple	The Netherlands	Ethrel A 480		24-192	7
Apple	USA	Ethrel 21.7%	0.25-1.26		7-21
Barley	Australia	Cerone 480			
Barley	Denmark	Cerone 480	0.24		30
Barley	Greece	Ethrel 38.0%	0.28-0.38		
Barley	Norway	Cerone 480			
Barley	Spain	Terpal 155 (Sprayed overall)	0.31-0.39		
Barley	USA	Cerone 39.9%	0.23-0.46		
Barley (spring)	Belgium	Cerone 480	0.6		
Barley (spring)	Finland	Cerone 480	0.14-0.34		
Barley (spring)	Ireland	Cerone 480	0.24		
Barley (spring)	Italy	Cerone 39.6%	0.20-0.30		
Barley (spring)	Luxembourg	Cerone 480	0.3-0.4		
Barley (spring)	UK	Cerone 480	0.24		49
Barley (spring)	UK	Stantion 360	0.24		49
Barley (summer)	Austria	Cerone 480	0.36		
Barley (summer)	Germany	Cerone 480	0.36		49
Barley (summer)	The Netherlands	Cerone 480	0.24		
Barley (winter)	Austria	Cerone 480	0.48		49
Barley (winter)	Belgium	Vivax 180 Cerone 480	0.36-0.45 0.48-0.6		
Barley (winter)	France	Cerone RPA 480	0.72		
Barley (winter)	France	Vivax RPA 150	0.23		
Barley (winter)	Germany	Cerone	0.48		49
Barley (winter)	Ireland	Cerone 480	0.48		
Barley (winter)	Italy	Cerone 39.6%	0.3-0.495		
Barley (winter)	Luxembourg	Cerone 480	0.48-0.6		
Barley (winter)	Switzerland	Cerone 39.6%			

Crop	Country	Application			PHI, (days)
		Form., concn.[1]	kg ai /ha	g ai/100 l	
Barley (winter)	The Netherlands	Cerone 480	0.48-0.6		
Barley (winter)	UK	Cerone 480	0.48		49
Barley (winter)	UK	Stantion 360	0.48		49
Blackberries	USA	Ethrel 21.7%	1.27		
Blackcurrants	Belgium	Ethrel F10 100 g/l		50	7
Blueberries	Canada	Ethrel 240	1.32-2.04		
Bromeliaceae	Austria	Ethrel 39.5%		19.8	
Cantaloupes	USA	Ethrel 21.7%	0.762		
Cereals	Finland	Cerone 480			
Cherries (see also Morellos)	Australia	Ethrel 480		48-96	7
Cherries	Austria	Ethrel 39.5%		19.8	
Cherries	Canada	Ethrel 240	0.66-1.32		8
Cherries	Denmark	Ethrel 480		24	5
Cherries	Greece	Ethrel 38.0%		19-38	7-10
Cherries	Switzerland	Ethrel 39.6%			10
Cherries	The Netherlands	Ethrel A 480			
Cherries	USA	Ethrel 21.7%	0.085-0.76		7-14
Citrus	Greece	Ethrel 480		14.4-28.8	
Cranberries	USA	Ethrel 21.7%	1.01-1.52		21*
Coffee	Brazil	Ethrel 21.6%	0.08-0.34		14
Cotton	Australia	Prep 720	1.4-3.24		
Cotton	Greece	Ethrel 38.0%	0.87-1.33		
Cotton	USA	Prep 55.4%	0.43-1.73		
Cucumber	Hungary	Ethrel 480	0.96		14
Cucumber	Romania	Ethrel 480	0.24-0.36		
Currants	Austria	Ethrel 39.5%		19.75	
Currants	Denmark	Ethrel 480		0.24	5
Currants, red	Belgium	Ethrel F 10		50	7
Figs	Greece	Ethrel 480		0.48	
Figs	USA	Ethrel 21.7%	1.02		
Filberts	USA	Ethrel 21.7%	0.76		
Flax	Belgium	Cerone 480	0.72		
Flax	France	Cerone RPA 480	0.72		
Flax	Luxembourg	Cerone 480	0.72		
Flax	The Netherlands	Ethrel A 480	0.48-0.72		
Grapes	Australia	Ethrel 480		19.2-28.8	14
Grapes	Greece	Ethrel 38.0%		28.5-57	
Grapes	USA	Ethrel 21.7%	0.25-0.51		
Hazel nut	USA	Ethrel 480	3.51-5.85		10
Lemons	USA	Ethrel 21.7%	0.846		
Macadamia nuts	Australia	Ethrel 480		120	42
Maize	Italy	Cerone 39.6%	0.19-0.29		
Maize	USA		0.15-0.3		8
Maize	Zimbabwe	Ethrel 480			2
Mandarins	Australia	Ethrel 480		24-28.8	
Morellos	Belgium	Ethrel F10 100		50	7
Olives	Greece	Ethrel 38.0%		76-133	7-10

Crop	Country	Application			PHI, (days)
		Form., concn.[1]	kg ai /ha	g ai/100 l	
Onions	Greece	Ethrel 38.0%	1.14		
Onions	The Netherlands	Ethrel A 480	1-1.5		21
Oranges	Australia	Ethrel 480		24-28.8	
Ornamentals	Denmark	Ethrel 480		24	
Ornamentals	Greece	Ethrel 38.0%		57-380	
Ornamentals	Norway	Ethrel 480			
Ornamentals	Sweden	Ethrel 100			
Ornamentals	The Netherlands	Ethrel A 480			
Peaches	Australia	Ethrel 480		12	5
Pears	Denmark	Ethrel 480		24	5
Peppers	Austria	Ethrel 39.5%		19.75	
Peppers	Greece	Ethrel 38.0%	0.76-1.14		
Peppers	Spain	Fruitel 480	0.72-1.08	33.6-72	10
Pepper	USA	Ethrel 21.7%	0.76-1.02		5
Sweet peppers	The Netherlands	Ethrel A 480	0.72		3
Pineapples	Australia	Ethrel 480	1.2-4.32		7
Pineapples	USA	Ethrel 39.9%	0.47-0.93		
Plums	Austria	Ethrel 39.5%		19.75	
Plums	Hungary	Ethrel 480	0.29-0.38		14
Plums	Norway	Ethrel 480		19.2	
Plums	Romania	Ethrel 480	0.18		14
Raisins	USA	Ethrel 21.7%	0.25-0.51		
Rape	Finland	Cerone 480			
Rape	Germany	Cerone 480	0.72		49
Rape (winter)	Denmark	Cerone 480	0.36		30
Rose	Austria	Ethrel 39.5%		19.75	
Rye	Belgium	Cerone 480	0.72-0.84		
Rye	Belgium	Vivax 180	0.45-0.54		
Rye	Denmark	Cerone 480	0.24-0.36		30
Rye	Italy	Cerone 39.6%	0.40-0.60		
Rye	Spain	Terpal 155	0.31-0.39		
Rye	Sweden	Cerone 480			
Rye (winter)	Finland	Cerone 480			
Rye (winter)	Germany	Cerone 480	0.72		49
Rye (winter)	Ireland	Cerone 480	0.48		
Rye (winter)	Luxembourg	Cerone 480	0.72-0.84		
Rye (winter)	Switzerland	Cerone 39.6%			
Rye (winter)	The Netherlands	Cerone 480	0.48-0.72		
Rye (winter)	UK	Cerone 480	0.48		47
Rye (winter)	UK	Stantion 360	0.48		47
Sugar cane	Brazil	Ethrel 21.6%	0.43-0.86		70
Sugar cane	USA	Ethrel 39.9%	0.47		
Tangerines	USA	Ethrel 21.7%	0.84-1.27		
Tobacco	Austria	Ethrel 39.5%		19.75	
Tobacco	Canada	Ethrel 240	0.66-0.90		
Tobacco	Greece	Ethrel 38.0%	1.52-2.28		
Tomatoes	Australia	Ethrel 480	0.86		7

Crop	Country	Application			PHI, (days)
		Form., concn.[1]	kg ai /ha	g ai/100 l	
Tomatoes	Austria	Ethrel 39.5%		19.75	
Tomatoes	Brazil	Ethrel 21.6%	0.86-1.73		21
Tomatoes	Canada	Ethrel 240	0.90		3
Tomatoes	Denmark	Ethrel 480		24	5
Tomatoes	Italy	Ethrel 39.5% Florel 22 21.6% Florel 40 39.5%	0.74-1.18 1.30-1.73 1.19-1.58	197.5 86.4-108 79-98.75	10
Tomatoes	Portugal	Ethrel 480	0.72-0.96		7
Tomatoes	Spain	Fruitel 480	1.08-1.44	36-72	10
Tomatoes	Sweden	Ethrel 100			7
Tomatoes	The Netherlands	Ethrel A 480	0.72	48	3
Tomatoes	USA	Ethrel 21.7%	0.32-1.52		3
Tomatoes (industry)	Italy	Ethrel 39.5%	0.99-1.19		10
Triticale	Belgium	Cerone 480	0.24-0.72		
Triticale	Ireland	Cerone 480	0.48		
Triticale	Switzerland	Cerone 39.6%			
Triticale (winter)	UK	Cerone 480	0.48		47
Triticale (winter)	UK	Stantion 360	0.48		47
Walnuts	USA	Ethrel 21.7%	0.76-1.27		5
Walnuts-Hazelnuts	Greece	Ethrel 480		72-96	
Wheat	Belgium	Vivax 150	0.19-0.3		
Wheat	Greece	Ethrel 480	0.36-0.48		7-10
Wheat	Spain	Trepal 155	0.31-0.39		
Wheat	The Netherlands	Saldo 180+360			
Wheat (spring)	Finland	Cerone 480	0.14-0.24		
Wheat (spring)	Ireland	Cerone 480	0.36		
Wheat (spring)	U.K	Cerone 480	0.36		47
Wheat (spring)	UK	Cerone 360	0.36		47
Wheat (spring)	USA	Cerone 39.9%	0.23-0.35		
Wheat (summer)	Germany	Cerone 480	0.36		49
Wheat (summer)	The Netherlands	Cerone 480			
Wheat (winter)	Belgium	Cerone 480	0.24-0.60		
Wheat (winter)	Denmark	Cerone 480	0.36		30
Wheat (winter)	Finland	Cerone 480	0.20-0.48		
Wheat (winter)	Germany	Cerone 480	0.48		49
Wheat (winter)	Ireland	Cerone 480	0.36		
Wheat (winter)	Italy	Cerone 39.6%	0.20-0.30		
Wheat (winter)	Luxembourg	Cerone 480	0.24-0.60		
Wheat (winter)	The Netherlands	Cerone 480	0.36-0.48		
Wheat (winter)	UK	Cerone 480	0.36		47
Wheat (winter)	UK	Stantion 360	0.36		47
Wheat (winter)	UK	Strate 180+360			45
Wheat (winter)	UK	Upgrade 180+360			45
Wheat (winter)	USA	Cerone 39.9%	0.23-0.47		
Wheat (winter, hard)	France	Cerone RPA 480	0.72		
Wheat (winter, soft)	France	Cerone RPA 480	0.29		
Wheat (winter, soft)	France	Vivax RPA 150+300	0.30		

[1] g/l unless otherwise indicated
* proposed use

Apples. Residues were determined in seven apple varieties in supervised trials conducted at various locations in the USA during 1969-1983. Apples were treated at rates of 0.5 and 1 kg ai /ha with Ethrel[R] and harvested from 3 to 13 days after application. Samples treated according to GAP and taken after registered PHIs showed residues varying from 0.22 to 3.79 mg/kg. Residues of 2.56, 4.53 and 7.99 mg/kg were found after shorter PHIs than were GAP. The results are shown in Table 2.

Two trials in The Netherlands could not be evaluated because the application rates could not be related to the available information on GAP.

Cherries. Studies in the USA in 1969 and 1970 were broadly in accordance with the present use patterns. Ethephon was applied at rates of 0.2 to 1.0 kg/ha. Cherry samples taken between 7 and 14 days in trials according to GAP showed residues between 0.69 and 6.6 mg/kg. The highest residue was 12.2 mg/kg, 7-8 days after the application of 1.0 mg/kg. The results are presented in Table 3.

Table 2. Residues of ethephon in apples from supervised trials in The Netherlands* and the USA. Underlined residues are from treatments approximating GAP (USA) or at the GAP PHI (Netherlands).

Country, Variety, Year	Application			Residue, mg/kg, at interval, days, after last application				Ref.
	Form	kg ai/ha	spray l/ha	3-4	6-7	9-11	13	
Netherlands								
James Grieve, 1975	SL480	0.67			0.87 1.02 1.14 0.71			32
Cox O.P., 1975	SL480	0.93			0.78 1.49 0.43 0.79			32
USA								
McIntosh, 1969	SL480	0.5	1000	0.22	0.22	0.16		72
		1.0	1000	0.48	0.64	0.49		72
Golden d., 1969	SL480	0.5	1000	1.37	1.35	0.71		72
		1.0	1000	1.26	2.56	1.19		72
Golden d., 1969	SL480	0.5	1000		2.32			72
		1.0	1000		4.53			72
York, 1969	SL480	0.5	1000		3.79			72
		1.0	1000		7.99			72
Twenty Ounce, 1969	SL480	0.5	1000				0.64	72
Cortland, 1969	SL480	0.5	1000			1.50		72
Rome Beauty, 1969	SL480	0.5	1000			1.03		72
		1.0	1000			2.04		72
Rome Beauty, 1970	SL480	0.5	1000			1.63		72
		0.5	1000			1.76		72
Red delicious, 1983 washed fruit wet pomace dry pomace fresh unfiltered juice cold clarified juice hot clarified juice	SL240	1.6	460		0.37 0.28 0.24 0.73 0.46 0.58 0.56			38

Peaches. In a trial conducted in Australia, peaches treated with Ethrel® at rates of 0.2 and 0.4 kg a.i/ha were harvested two to three weeks after the treatment. In the three samples analysed, residues of ethephon were 0.18 and 0.21 from the 0.2 kg/ha treatment and 0.46 mg/kg from the higher rate (Ciba-Geigy, 1974a).

Plums. Plums dipped in a 1 g/l solution of ethephon were analysed for residues in fruit after storage at ambient temperature (20°C). Residues of ethephon decreased slowly from 0.96 mg/kg one day after treatment, to 0.66 mg/kg after seven days (Ciba-Geigy, 1974b).

Table 3. Residues of ethephon in cherries from supervised trials in the USA (Ref. 73).Underlined residues are from treatments approximating GAP

Year	Application			Residue, mg/kg, at interval, days, after last application			
	Form.	kg ai/ha	spray l/ha	6	7-8	10-11	13-14
1969	SL240	1	1000			2.5	
1969	SL240	0.5		0.84			
		1		2.60			
1969	SL240	1				3.05	
1969	SL240	0.5			0.69		
		1			2.66		
1969	SL240	0.5			6.55		
		1			12.2		
1969	SL240	0.5					3.93
1969	SL240	0.5			6.57		
		1			11		
1969	SL240	0.5				2.50	
1969	SL240	0.5	1000			3.54	
		1	1000			7.58	
1969	SL240	0.5	1000	3.16			
		1	1000	9.75			
1969	SL240	0.2	1000				1.10
		0.5	1000				1.79
1969	SL240	0.5	1000		1.35		1.07
1969	SL240	0.5	1000		4.67		
1970	SL240	0.5	1000				0.58
1970	SL240	0.2	1000			2.73	
1969	SL240	0.5			2.19		1.31
		0.5			1.66		1.55
		0.5			0.98		0.65
		0.5		0.03			<0.01

Blackberries. Blackberry samples from trials conducted in the USA in 1974 and 1989 with Ethrel^{R} at dose rates between 1.12 (recommended) and 2.8 kg/ha showed residues ranging from 8 to 18 mg/kg 1-3 days after application. The results are shown in Table 4.

Blueberries. Supervised trials were conducted in the USA at dose rates, approximating Canadian use patterns, from 0.56 to 2.24 kg ai/ha and with pre-harvest intervals from 0 to 55 days. The residues were 1.4-11 mg/kg and 2.1-9.1 mg/kg 14 and 39 days after the treatments, respectively. See Table 4.

Table 4. Residues of ethephon in berries from supervised trials.

Country, Year, Sample	Application			Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	spray l/ha	0-1	2-3	4-7	8-11	14 or (No.)	
USA, 1974, blackberries	SL480	1.4	1000	0.4	10				77
		2.8	1000	1.9	18				77
blackberries, fruit		1.12	1000	8		8			77
preserved fruit				3.2		3.0			77
USA, 1989, blackberries	SL240	2.8	460		16	6.5		3.2	40
		2.8	460		12	9.3		6.6	40
USA, 1977, blueberries	SL240	1.12		5.4	5.8	4.0	3.1	2.6	78
		2.2		17	17	19	11	8	78
		1.12		2.0	1.2	1.4	1.0	1.4	78
		2.2		4.2	3.2	3.2	2.7	2.0	78
		1.12					0.9-1.3		78
		2.2					2.5-3.5		78
		1.12				6.1		5.8	78
		2.24				11		11	78
		2.24						9.1 (39)	78
		1.96						5.3 (55)	78
		0.56						2.1 (39)	78
CAN, 1977, cranberries	SL240	1.12		3.3	4.8	2.4	1.1	0.1 0.15 (21)	7
		2.24		6.5	10	5.6	2.4	0.4 0.4 (21)	7
		1.12						0.07	7
		2.24						0.22	7
		1.12				0.23		0.1	7
		1.4					0.27	0.15 (17)	7
		2.24				0.42		0.18	7
		2.8					0.38	0.68 (17)	

Cranberries. Supervised trials were conducted in Canada in 1977 at application rates from 1.1 to 2.8 kg ai/ha, covering the current maximum US rate of 1.52 kg ai/ha. Samples taken between 8 and 21 days after the application of 1.1 kg/ha contained residues from 0.07 to 1.1 mg/kg. The results are in Table 4.

Grapes. Supervised trials were conducted in the USA (California) and Canada with single applications close to the maximum recommended US rate of 0.51 kg ai/ha. Grapes were harvested between 7 and 47 days (a few samples up to 108 days) after treatment. Residues decreased from 0.07 to 2.2 mg/kg with an average of 0.93 mg/kg at 7 days to 0.15 to 0.47 mg/kg with an average of 0.28 mg/kg at 45 days and <0.01 mg/kg at 92-108 days.

Residues in raisins from grapes treated about 45 days before harvest ranged from 0.21 to 1.5 mg/kg. The maximum concentration factor found from grape to raisin was 3.5. Raisin waste from the

six samples harvested 45 days after treatment showed ethephon residue levels of 1.1 to 38 mg/kg with an average of 10 mg/kg. The results are shown in Table 5.

Table 5. Residues of ethephon in grapes from supervised trials with SL 240 g/l formulation.

Sample, Country, Year	kg ai/ha	Residue, mg/kg, at interval, days, after last application								Ref.
		7	10	14	23-25	30	42-47	60-75	92-108	
Grape, CAN, 1974.	0.5								<0.01	9
Grape, CAN, 1974.	0.5								<0.01	9
Grape, CAN, 1974.	0.45								<0.01	9
Grape, USA, 1978.	0.56						0.46			13
Raisin							0.46			13
Raisin waste							9.28			13
Grape, USA, 1978.	0.56						0.47			13
Raisin							1.49			13
Raisin waste							38.0			13
Grape, USA, 1978.	0.56						0.15			13
Raisin							0.21			13
Raisin waste							3.27			13
Grape, USA, 1978.	0.56						0.24			13
Raisin							0.22			13
Raisin waste							4.72			13
Grape[1], USA, 1975	0.56					0.09				11
Juice						0.14				11
Wine						0.13				11
Dried pomace						0.15				11
Grape[1], USA, 1975	0.56				0.38					11
Juice					0.40					11
Wine					0.23					11
Dried pomace					0.53					11
Grape[1], USA, 1975.	0.56				0.20					11
Juice					0.11					11
Wine					0.10					11
Dried pomace					0.18					11
Mature fruit*, USA, 1975.	0.56				0.29					11
Grape[1]* USA, 1975.					0.35					11
Juice					0.63					11
Wine					0.37					11
Dried pomace					0.50					11
Mature fruit*, USA, 1975.	0.56				0.25					11
Grape[1]*, USA, 1975.					0.28					11
Juice					0.44					11
Wine					0.22					11
Dried pomace					0.47					11
Grape[1], USA, 1975.	0.56				0.19					11
Juice					0.31					11
Wine					0.23					11
Dried pomace					0.52					11
Grape, USA, 1989.	0.56	2.20	1.00	0.82						44, 46

Sample, Country, Year	kg ai/ha	Residue, mg/kg, at interval, days, after last application								Ref.
		7	10	14	23-25	30	42-47	60-75	92-108	
Grape, USA, 1989.	0.56	0.07	0.07	0.06						44, 46
Grape, USA, 1989.	0.56	0.53	0.42	0.36						44, 46
Grape, USA, 1989.	0.56						0.16	0.11 0.08		44, 46
Raisin waste, USA, 1989.							1.13	0.89 0.75		44, 46
Grape, USA, 1989.	0.56						0.21	0.31 0.15		44, 46
Raisin waste, USA, 1989.							3.60			44, 46

[1] Simulated grapes were prepared by adding three parts by weight of undried pomace to one part by weight of juice, in those cases where mature berries were not retained after processing, and in two additional sets (marked *) to determine the validity of the simulation.

Figs. Supervised trials were carried out in Canada in 1977 at rates up to 1.87 kg ai/ha, roughly twice the US recommended rate of 1.02 kg ai/ha. Following treatments with recommended rates, residues in dried figs treated 21-41 days before harvest were in the range 0.22 to 2.73 mg/kg and showed little variation with the PHI. The results are shown in Table 6.

Table 6. Residues of ethephon in dried figs from supervised trials in Canada in 1977 with SL 240 g/l formulation and 1000 l spray/ha (Ref. 12).

kg ai/ha	Residue, mg/kg, at interval, days, after last application				
	14 - 15	21	26 - 28	33-35	41
0.38	0.34	0.36	0.39		
0.75	0.32	0.22	0.22		
0.94	0.43	0.55	0.55		
1.87	0.43	1.00	1.00		
0.50			1.16		
0.38				0.83	
0.50				1.23	
0.38				0.25	0.34
0.75				0.29	0.47
0.94				0.29	0.34
1.87				0.49	0.73
0.25	1.54		1.18		
0.50	3.22		2.07		
1.00	8.49		2.73		
0.25	0.87		0.73		
0.50	2.31		1.25		
1.00	5.54		2.68		
0.25	1.32		0.54		
0.50	0.89		0.97		
1.00	5.58		1.60		

Pineapples. Supervised trials were conducted in major pineapple-growing areas with different climates in Hawaii from 1970 to 1990. Applications were made close to the maximum recommended rate (1.12 kg ai/ha = x) and at 0.5x, 2x and 4x rates. Ethrel was applied one to three times in different combinations of the dosages. In the case of multiple applications, the first (and second) treatments were about 6 months before harvest or earlier. The last application was shortly before harvest.

The ethephon residues from multiple applications were independent of the dosage of the early treatments. Residues decreased with increasing pre-harvest intervals. When the last treatment was at or below the maximum GAP rate, samples of whole fruit taken 7-8 days after the last application contained <0.02-0.32 mg/kg with an average of 0.17 mg/kg and standard deviation of 0.12 (9 samples), and the pulp contained 0.06-0.33 mg/kg with an average of 0.18 mg/kg and standard deviation of 0.12 (21 samples). The results are shown in Table 7.

Cucumbers. Trials were conducted in Canada in 1973 and 1974 with four varieties of cucumber at an application rate of 0.25 kg ai/ha (one or two treatments) and with PHIs between 28 and 48 days. No residues above the 0.01 mg/kg limit of determination were found in any of the 9 samples analysed (Amchem, 1975a).

Table 7. Residues of ethephon in pineapples from supervised trials in Hawaii.

Crop, part, Year	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	1	2	4	7-8	10-14	
Fruit[1], whole, 1970 Pulp	SL240	0.56				0.28 0.33		3 3
Fruit, whole Pulp	SL240	0.56				0.17 0.06		3 3
Fruit, whole Pulp	SL240	0.56					1.09 0.56	3 3
Fruit, whole Pulp	SL240	0.56					0.16 0.05	3 3
Fruit, 1989	SL39.9%	2.24 + 1.12	0.09	0.05	0.04	0.03		61
Fruit, 1989	SL39.9%	2.24 + 2.24	0.22	0.11	0.13	0.07		61
Fruit, 1989	SL39.9%	2.24 + 1.12	0.18	0.11	0.03	<0.02		61
Fruit, 1989	SL39.9%	2.24 + 2.24	0.38	0.07	0.09	0.06		61
Fruit, 1990	SL39.9%	1.12		0.29	0.32	0.32		61
		2.24		0.41	0.98	0.72		61
Fruit, 1989	SL39.9%	2.24 + 1.12	0.67	0.67	0.42	0.27		61
Fruit, 1989	SL39.9%	2.24 + 2.24	1.27	0.86	0.72	0.69		61
Fruit, 1989	SL39.9%	2.55 + 1.12	0.26	0.17	0.32	0.23		61
Fruit, 1989	SL39.9%	2.55 + 2.24	0.62	0.40	0.36	0.76		61
Fruit, 1989	SL39.9%	2.99 + 1.12	0.37	0.11	0.16	0.17		61
Fruit, 1989	SL39.9%	2.99 + 2.24	0.81	0.26	0.63	0.48		61
Fruit whole, 1989	SL39.9%	2.99 + 2.24				0.18 0.05		61
Beverage juice, 1990	SL39.9%					0.07		61
Exchange juice, 1990	SL39.9%					0.15		61
Pulp, 1990	SL39.9%					0.22		61
Bran, 1990	SL39.9%					0.95		61
Pulp, 1977	SL240	1.12, 0.56 1.12, 1.12 1.12, 2.24				0.15 0.23 0.31		71 71 71
		4.48, 0.56 4.48, 1.12 4.48, 2.24				0.16 0.22 0.40		71 71 71
		0.00, 0.56 0.00, 1.12 0.00, 2.24				0.12 0.21 0.24		71 71 71
		1.12, 0.00 1.12, 0.56 1.12, 1.12 1.12, 2.24				0.01 0.14 0.24 0.32		71 71 71 71
		4.48, 0.00 4.48, 0.56 4.48, 1.12 4.48, 2.28				0.01 0.14 0.14 0.32		71 71 71 71
		0.00, 0.56 0.00, 1.12 0.00, 2.24				0.11 0.24 0.24		71 71 71
		1.12, 0.00 1.12, 0.56				0.01 0.13		71 71

Crop, part, Year	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	1	2	4	7-8	10-14	
		1.12, 1.12 1.12, 2.24				0.33 0.50		71 71
		4.48, 0.00 4.48, 0.56 4.48, 1.12 4.48, 2.24				0.01 0.08 0.22 0.50		71 71 71 71
		0.00, 0.56 0.00, 1.12 0.00, 2.24				0.03 0.16 0.48		71 71 71
		1.12, 0.00 1.12, 0.56 1.12, 1.12 1.12, 2.24				0.01 0.03 0.08 0.08		71 71 71 71
		4.48, 0.00 4.48, 0.56 4.48, 1.12 4.48, 2.24				0.01 0.04 0.07 0.12		71 71 71 71
Juice, canned	SL240[2]	1.12, 2.24, 1.12 2.24, 2.24, 1.12 4.48, 2.24, 1.12				0.11 0.09 0.09		71
Juice, canned		1.12, 2.24, 1.12 2.24, 2.24, 1.12 4.48, 2.24, 1.12 4.48, 2.24, 2.24				0.02 0.01 0.03 0.06		71
Juice, canned, 1977	SL240	1.12, 2.24, 1.12 2.24, 2.24, 1.12 4.48, 2.24, 1.12				0.14 0.14 0.14		71
Juice, canned, 1977	SL240	1.12, 2.24, 1.12 2.24, 2.24, 1.12 4.48, 2.24, 1.12 4.48, 2.24, 2.24				0.11 0.12 0.15 0.23		71
Foliage, 1977	SL240	1.12, 1.12, 1.12 1.12, 1.12, 2.24				1.06 1.98		71
		1.12, 1.12, 1.12 1.12, 1.12, 2.24				0.80 1.75		71
		1.12, 1.12, 1.12 1.12, 1.12, 2.24				2.19 2.66		71
		1.12, 1.12, 1.12 1.12, 1.12, 2.24 4.48, 2.24, 2.24				1.77 4.54 5.35		71
Bran, 1977	SL240	1.12				1.19		71
		1.12					1.25	71
		1.12					1.11	71
		1.12				0.74		71
		1.12					0.63	71
		1.12					0.41	71
		1.12				1.08		71
		1.12					1.08	71
		1.12					1.04	71
		1.12				0.73		71
		1.12					0.66	71

Crop, part, Year	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	1	2	4	7-8	10-14	
		1.12					0.55	71

[1] Residues calculated from values measured in shell + core and pulp
[2] Three broadcast applications were made 1st 7-7.5 months, 2nd 2.5-3.5 months, 3rd 7-8 days before the anticipated harvest

Table 8. Residues of ethephon in cantaloupes from supervised trials in the USA. Underlined residues are from treatments according to GAP.

Year, Crop part	Application			Residue, mg/kg, at interval, days, after last applicn.							Ref.
	Form.	kg ai/ha	spray l/ha	0	1	2	3	4	5	6	
1975, Pulp Peel Fruit	SL240	0.90	1000	0.03 0.31	0.18 0.44	0.34 0.91	<u>0.16</u>	0.15	0.08		10
Fruit		0.96	1000				<u>0.18</u>				10
1975, Pulp Peel Fruit		1.79	1000	0.13 1.10	0.16 1.13	0.32 1.19	0.30		0.11		10
1975, Pulp	SL240	0.90	1000	0.38	0.31	0.21					10
1975, Pulp		1.79	1000	0.46	0.34	0.29					10
1975, Pulp Peel Seed Fruit		0.90	1000	0.06 0.19 0.03	0.15 0.36 0.16	0.55 0.69 0.23		<u>0.23</u>	0.23		10
1975, Pulp Fruit		1.79	1000	0.03	0.32	0.23		0.57			10
1975, Fruit		0.56	1000	0.28 0.15	0.15	<u>0.04</u>			0.07		10
Fruit		0.90	1000	0.07		0.06		<u>0.07</u>			10
Fruit		1.79	1000	0.19		0.79		0.38			10
1989, Fruit	SL240	0.97*				0.11		<u>0.07</u>		0.03	41
		0.97				0.44		<u>0.40</u>		0.06	41

* Aerial application

<u>Cantaloupes</u>. Supervised field trials were conducted in the USA between 1975 and 1989, applying ethephon at about registered (0.76 kg ai/ha) and double rates. Residues in whole fruits from recommended use were in the range 0.04 to 0.4 mg/kg 2-4 days after application. Residue ratios in the peel and pulp varied with the PHI, the peel always containing higher residues. The results are shown in Table 8.

<u>Peppers</u>. Peppers were treated at about the maximum recommended rate of 1.02 kg ai/ha in three states in the USA. Residues ranged from 3.5 to 26.2 mg/kg 5 to 8 days after application, close to the range reported in the 1977 evaluation. In Canadian trials at 0.75 kg ai/ha the residues varied from 0.7 to 1.1 mg/kg 7-8 days after treatment. Results are given in Table 9.

<u>Tomatoes</u>. Supervised field trials were conducted in Italy, The Netherlands and the USA. The dosage and sampling intervals covered the current use patterns. Samples from treatments with 1.8 kg ai/ha (the maximum recommended rate in the USA is 1.5 kg ai/ha) and taken 3 to 7 days after application

contained residues in the range 0.09 to 1.4 mg/kg. The results are shown in Table 10.

Sweet corn. In trials in the USA at 0.56 kg ai/ha, the residues in kernels plus cobs with husks removed ranged from <0.02 to 0.62 mg/kg at sampling intervals of 21-39 days, while five of the 8 samples analysed showed residues of less than 0.02 mg/kg 50-79 days after application. The residues were 0.04, 0.05 and 0.14 mg/kg in the other samples. The forage contained residues from 0.15 to 3.95 mg/kg and <0.02 to 1 mg/kg at the shorter and longer sampling intervals, respectively. Residues are shown together with those in maize and popcorn in Table 13.

Popcorn. Seven residue trials were conducted in 1985 in five States of the USA. The rates of broadcast foliar spray applications were 0.56 kg ai/ha. Six of the seven grain samples analysed showed residues of less than 0.02 mg/kg while the remaining sample contained 0.19 mg/kg. Residues in stover samples ranged from <0.02 to 0.23 mg/kg, with one exception of 1.18 mg/kg. In popcorn silage, residues varied from <0.02 to 2.44 mg/kg (Table 11).

Peas. A study was conducted in Canada with five varieties of peas to determine the residues of ethephon resulting from treatments at application rates from 0.35 to 1.68 kg ai/ha and at sampling intervals between 30 and 56 days for peas and 29 days for pea vines. The residues in peas ranged from <0.01 to 0.05 mg/kg and in pea vines from 0.22 to 1.26 mg/kg (Table 11).

Table 9. Residues of ethephon in peppers from supervised trials. Underlined residues are from treatments according to GAP.

Commodity, Country, Year	Rate, kg ai/ha	Residue, mg/kg, at interval, days, after last application					Ref.
		5-6	7-8	12-13	14-17	24-25	
Pepper, USA, 1973	0.84			0.28			8
Pepper, dehydrated				0.48			8
Pepper	1.12			0.39			8
Pepper dehydrated				0.8			8
Pepper, USA, 1973	1.12				0.23		8
Pepper	1.40				0.45		8
Pepper	1.12				<0.01		8
	1.12				0.83		8
	1.12				0.33		8
Pepper, USA, 1973	0.84				0.87		8
Pepper, dehydrated					0.08		8
Pepper	1.12		10.8				8
	1.12		9.71				8
Pepper, USA, 1973	1.12		22.3				8
	1.12		26.2				8
	1.12		8.89				8
	1.12		6.83				8
	1.12		4.45				8
	1.12		4.28				8
	1.12	3.51			0.77		8
	1.12	10.58			2.63		8
	1.12	5.65			1.10		8
	1.12	7.29			1.13		8
Pepper, CAN, 1973	0.3		0.72				9

Commodity, Country, Year	Rate, kg ai/ha	Residue, mg/kg, at interval, days, after last application					Ref.
		5-6	7-8	12-13	14-17	24-25	
	0.75		0.72				9
	0.3		0.92				9
	0.75		1.06				9
	0.75		1.06				9
	0.75			1.18			9
	0.3	1.27					9
Pepper, CAN, 1974	0.5				0.84		9
	1.5					<0.01	9
	0.5				0.73		9
	1.5					<0.01	9
	1.5					1.28	9
	1.6				1.49		9
	1.6				1.21		9

Table 10. Residues of ethephon in tomatoes from supervised trials with SL 240 g/l formulation. Underlined residues are from treatments according to GAP.

Country, Year sample	kg ai/ha	Residue, mg/kg, at interval, days, after last application									Ref.
		0-1	3-4	6-7	9-11	12-13	14-16	18-20	21-22	26-28	
ITA, 1985, Fruit	1.92								0.17		68
	2.4							0.33			68
	1.44								0.72		68
	2.40						0.76				68
	2.40							0.65			68
	1.92							0.21			68
	2.88									0.22 1.88	68
	1.92		0.41	0.41			0.06				68
NET, 1985, Fruit**	*		2.9 1.8 3.4 4.2	3.4 3.8 5.1 5.4		3.2 4.8 7.7 3.4					32
NET, 1985, Fruit**	*		2.7 1.9 2.9 3.2	4.6 4.1 3.2 6.2		5.1 3.9 4.4 6.1					32
NET, 1976, Fruit**	1.2		1.5 1.7 1.5 1.2	1.5 1.6 1.7 1.4							32
NET, 1976, Fruit**	1.2		1.6 1.3 1.4 1.1	1.5 1.5 1.0 1.1							32
USA, 1989, Fruit	1.75	0.18	0.11	0.09							47
Fruit	2.13	0.48	0.44	0.27							47
Fruit	1.79		0.66	0.92			0.69				47

Country, Year sample	kg ai/ha	Residue, mg/kg, at interval, days, after last application									Ref.
		0-1	3-4	6-7	9-11	12-13	14-16	18-20	21-22	26-28	
Fruit	2.00		0.02	<0.02			0.15				47
USA, 1989, Fruit	1.15				0.73						47
Washed fruit					0.68						47
Wet pomace	1.27				0.38						47
Dry pomace					1.39						47
USA, 1990, Fruit	1.80		<0.02	<0.02			<0.02				64
Fruit	1.80		0.32	0.06			0.06				64
USA, 1970, Mature fruit	0.90			0.46							12
	1.80			1.41							12
	0.90						0.15				12
	1.80						0.40				12
	0.90								0.16		12
	1.80								0.41		12
USA, 1969, Mature fruit	1.80					1.26					12
	0.60					0.09					12
	0.60					0.14					12
	0.60					0.09					12
USA,1969, Juice, canned	0.60			0.06	0.06		0.08				12
USA, 1969, Mature fruit	0.60				0.61						12
	1.80				1.74						12
USA, 1970, Juice, canned	0.60						0.09				12
Juice, canned	0.60							0.29			12
1970, Mature fruit	0.90	0.01		0.10	0.08	0.08	0.03	0.03			12
	1.80	0.02		0.14	0.12	0.10	0.06	0.12			12
1970, Mature fruit	1.80	0.07	0.23								12
	1.20							0.12			12
	1.80	0.11	0.37								12
1970, Sauce, canned	0.45					<0.01					12
	0.90					<0.01					12
1970, Mature fruit	0.90						0.42				12
	1.80						1.14				12
1969, Mature fruit	0.60						0.29				12
	0.90				0.78						12
	1.80				1.30						12
1970, Mature fruit	0.90						0.13				12
	1.80						0.21				12
	0.90						0.23				12
	1.80						0.50				12
	0.48					0.21					12
	0.72					0.18					12
	0.48					0.07					12
	0.72					0.10					12
	0.90			0.54			0.29		0.24		12

Country, Year sample	kg ai/ha	Residue, mg/kg, at interval, days, after last application									Ref.
		0-1	3-4	6-7	9-11	12-13	14-16	18-20	21-22	26-28	
	2.24								0.28		12

* Brushing of stems with water/480 g/l ethephon 1:1 sludge at a height of 1-1.5 m
** Replicate field trials

Table 11. Residues of ethephon in mature peas from supervised trials with SL 240 g/l formulation in Canada (ref. 9).

Year	kg ai/ha	Residue, mg/kg, at interval, days, after last application					
		Vine	Peas				
		29	30	32-34	36-38	44	56
1971	0.42		<0.01				
	0.56		<0.01				
	0.84		<0.01				
	1.12		<0.01				
	1.40		<0.01				
1972	0.56			<0.01			
	1.12			<0.01			
	1.68			<0.01			
1973	0.56					<0.01	
	1.12					<0.01	
	0.56		0.05				
	1.12		0.01				
	0.56				<0.01		
	0.56			<0.01			
	0.56				<0.01		
	0.35						<0.01
	0.56	0.22					
	1.12	0.67					
	1.68	1.26					

Cereal grains

Several studies have been conducted in the major cereal-producing countries (Australia, Belgium, Canada, Denmark, France, Germany, The Netherlands, Norway, the UK and the USA). Residues from trials conducted according to current use patterns are discussed below.

Barley. Residues in mature grain ranged from <0.01 to 0.50 mg/kg at 60-69 days after application. The straw contained residues up to 1.71 mg/kg. Residue levels in grain at harvest after 60 days were generally below 0.05 mg/kg (Table 12).

Wheat. Numerous trials with applications at 0.56 kg ai/ha, close to the maximum registered rate in the USA of 0.47 mg/kg, showed residues in the mature grain in the range 0.08 to 0.68 mg/kg at sampling intervals of 34-41 days after application. Residues in straw varied between 0.95 and 3.23 mg/kg (Table 14). Residues from European trials at other PHIs are shown in Table 15.

Oats and rye. Several trials have been reported from Finland, Germany, The Netherlands, the UK and the USA. At harvest residues in grain were <0.01-0.22 mg/kg in oats and <0.01-0.44 mg/kg in rye. Residues in straw were 0.35 to 1.38 mg/kg (Tables 16 and 17).

Maize. Formulations of Cerone^{R} (480 or 240 g/l) were used for trials conducted in the USA. A rate of 0.56 kg ai/ha was used for all the trials, with ground or aerial application. The maximum registered US rate is 0.3 kg ai/ha. The plants at application were generally at the 12-leaf stage. Fourteen of the 18 grain samples analysed showed residues of less than 0.02 mg/kg, which is the estimated limit of determination. Residues in the remaining four samples ranged from 0.03 to 0.12 mg/kg. The results are shown in Table 13. Silage and stover contained residues from 0.02 to 1.7 mg/kg and from <0.02 to 2.53 mg/kg respectively.

Table 12. Residues of ethephon in barley from supervised trials. Underlined residues are from treatments according to GAP.

Country, Year Crop part	Application			Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	spray l/ha	34-41	49-59	60-69	70-89	>90	
AUL, 1988 Grain	SL480	0.36				0.1-0.2[1]			14
		0.72				0.2-0.3[1]			14
BEL, 1978 Ears Straw Grain	SL480	0.60		1.04 4.30		 0.16			2
BEL, 1978 Ears Straw Grain	SL480	0.60		0.26 1.15		 0.05			2
BEL, 1978 Grain Straw	SL155	0.39					<0.02 0.40	<0.02 0.16	2
BEL, 1978 Grain	SL155	0.39					<0.02	<0.02	2
	SL155	0.39					0.02	0.02	2
BEL, 1978 Grain Straw Grain Straw	SL155	0.39					<0.02-0.02 0.40 <0.02-0.02 0.20		2
BEL, 1978 Grain Straw	SL155	0.31					<0.02-0.02 0.06		2
Grain Straw		0.39					<0.02 0.08		2
Grain Straw		0.47					<0.02 0.07		2
Grain Straw		0.58				<0.02-0.03 0.07			2
BEL, 1978 Grain	SL155	0.31 0.39 0.58				<0.02 <0.02 <0.02			2
BEL, 1979 Grain Straw Grain Straw	SL480	0.48					0.03 0.30 <0.02 <0.02		2
BEL, 1979 Grain	SL480	0.48				0.03			2

Country, Year Crop part	Application			Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	spray l/ha	34-41	49-59	60-69	70-89	>90	
Straw						0.60			
BEL, 1979 Grain Grain	SL480	0.48				0.04 0.04			2
BEL, 1979 Grain Straw	SL480	0.48				0.02 0.85			2
BEL, 1979 Grain Straw	SL480					<0.02-0.02 0.04			2
BEL, 1979 Grain	SL480	0.60				0.02			2
BEL, 1979 Grain Straw	SL480	0.48					<0.02 0.05		2
Grain Straw		0.60					<0.02 0.13		2
Grain Straw		0.60				0.02 0.16			2
BEL, 1979 Grain Straw	SL480	0.60					0.03 0.07		2
Grain Straw		0.48				0.10 1.20			2
Grain Straw		0.60				0.13 1.40			2
BEL, 1979 Grain Straw	SL480	0.48				0.07 0.65			2
CAN, 1981 Grain Hulls Pearls	SL480	0.56			0.62 1.01 0.54				2
CAN, 1982 Grain Straw	SL480	0.56					0.04 0.18		2
CAN, 1982 Grain Straw	SL480	0.56			0.12 0.55 0.36 1.11				2
CAN, 1982 Grain Straw	SL480	0.56				0.04 0.54			2
CAN, 1982 Grain	SL480	0.56		0.10 0.11	0.05				2
DEN, 1985 Grain	SL480	0.24			<0.05 <0.05				30
DEN, 1986 Grain	SL480	0.24				<0.05 <0.05			30
DEN, 1986 Grain Straw	SL480	0.24					<0.05 <0.05 0.16 0.14		30
FIN, 1980 Grain	SL480	0.3			<0.01	<0.01			22
FIN, 1981 Grain	SL480	0.16				<0.02			22

Country, Year Crop part	Application			Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	spray l/ha	34-41	49-59	60-69	70-89	>90	
		0.23				<0.02			22
		0.16					<0.02		22
		0.23					0.02		22
FIN, 1984 Grain	SL480	0.16			0.01				22
		0.23			0.03				22
FIN, 1982 Grain	SL480	0.36				<0.02			22
		0.24					<0.02		22
FIN, 1981 Grain	SL480	0.24				<0.02			22
		0.48				<0.02			22
		0.24					<0.02		22
		0.48					0.02		22
		1.0				<0.02			22
		1.0					0.05		22
FIN, 1982 Grain	SL480	0.5				0.02			22
		0.05					<0.02		22
FIN, 1986 Straw	SL480	0.34			0.12				22
		0.51			0.16				22
FRA, 1977 Grain Straw	SL480	0.96			<0.01 0.08				23
FRA, 1977 Grain Straw	SL480	0.96			0.05 0.45-0.43				23
FRA, 1977 Grain Straw	SL480	0.96				<0.01 <0.01			23
FRA, 1977 Grain Straw	SL480	0.96					<0.01 0.07		23
GER, 1978 Grain Straw	SL480	0.48	400			0.02 0.48			2
Grain Straw		0.48	400				0.03 0.06		2
Grain		0.48	400			0.50	0.06		2
Grain		0.48	400			0.11	0.06		2
Grain		0.48	400			0.04	<0.01		2
Grain		0.48	400		0.15	0.02			2
Grain		0.48	400			0.21	<0.01		2
Grain		0.48	400			0.05	<0.01		2
Grain		0.48	400					<0.01	2
Grain Straw Grain		0.48	400			0.04 0.36 0.06			2

Country, Year Crop part	Application			Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	spray l/ha	34-41	49-59	60-69	70-89	>90	
Straw						0.55			
Grain Straw		0.48	400				0.04 0.03		2
Grain Straw		0.48	400			0.05 1.71			2
Grain Straw		0.48	400			<0.1 <0.1			2
Grain Straw		0.48	400		<0.05 <0.05				2
Grain Straw		0.48	400			<0.05 1.17			28
Grain Straw		0.48	400			<0.05 0.10			28
Grain Straw		0.36	400		<0.02 <0.04				90
Grain Straw		0.36	400		<0.02 0.03				90
Grain Straw		0.36	400				0.06 0.30		85
Grain Straw		0.36	400		0.16 0.48				85
Grain Straw		0.36	400			0.03 0.07			85
NET, 1974 Grain Straw	SL480	0.48	400					0.27 0.74	69
NOR, 1987 Grain	SL480	0.24					<0.05	<0.05	35
UK, 1990 Grain Straw	EC480	0.24				<0.05 0.44			44
Grain Straw		0.24				<0.05 0.11			44
Grain Straw		0.24				<0.05 0.31			44
Grain Straw		0.48				<0.05 0.44			44
UK, 1982 Grain Straw	EC480	0.24					<0.05 0.16		25
Grain Straw		0.24					<0.05 <0.05-0.09		25
Grain Straw		0.48	200-250				<0.05-0.06 0.31		25
Grain Straw		0.48	200-250				<0.05-0.07 0.30		25
Grain		0.48	225				<0.05		25
Grain		0.48	200				<0.05		25

Country, Year Crop part	Application			Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	spray l/ha	34-41	49-59	60-69	70-89	>90	
USA, 1981 Grain Straw	SL480	0.56		0.13 1.85					88
Grain Straw		0.56			0.03 0.06				88
Grain		0.84			0.10				88
Grain		0.56			0.03				88
USA, 1982 Grain	SL480	0.56			0.78				88
USA, 1981 Grain	SL480	0.56				0.40			88

[1] Replicate treatments

Table 13. Residues of ethephon in maize, sweet corn and popcorn from applications of 0.56 kg ai/ha of SL 240 and 480 g/l formulations in the USA (1985).

Crop part	Residue, mg/kg, at interval, days, after last application									Ref.
	21-28	30-39	40-43	50-59	60-69	70-79	80-89	90-99	>100*	
Maize									<0.02 (102)	105
Stover									0.27 (102)	105
Silage				0.02						105
Maize								<0.02		105
Stover								0.05		105
Silage						0.09				105
Maize							0.09			105
Stover							0.07			105
Silage				0.03						105
Maize						0.03				105
Stover						0.19				105
Silage	0.60									105
Maize							<0.02			105
Stover							0.39			105
Silage		0.63								105
Maize								<0.02		105
Stover								0.08		105
Silage		0.15								105
Maize						<0.02				105
Stover						0.31				105
Silage				0.45						105
Maize								<0.02		105
Stover								0.02		105
Silage			0.03							105
Maize								<0.02		105

Crop part	Residue, mg/kg, at interval, days, after last application									Ref.
	21-28	30-39	40-43	50-59	60-69	70-79	80-89	90-99	>100*	
Stover								0.59		105
Silage		0.93								105
Maize									<0.02 (121)	105
Stover									<0.02 (121)	105
Silage						0.06				105
Maize							0.04			105
Stover							2.53			105
Silage						1.73				105
Maize						<0.02				105
Stover						0.21				105
Silage				0.08						105
Maize						<0.02				105
Stover						<0.02				105
Silage			0.10							105
Maize								<0.02		105
Stover								0.16		105
Silage			0.12							105
Maize							0.12			105
Stover							1.17			105
Maize								<0.02		105
Stover								0.62		105
Silage					0.17					105
Maize								<0.02		105
Stover								0.89		105
Silage					0.21					105
Maize						<0.02				105
Stover						0.41				105
Silage				0.26						105
Popcorn									<0.02 (101)	102
Stover									<0.02 (101)	102
Popcorn					<0.02					102
Stover					0.23					102
Silage	0.25 (14 days)									102
Popcorn							<0.02			102
Stover							0.04			102
Popcorn						<0.02				102
Stover						0.14				102
Silage				0.96						102
Popcorn							0.19			102
Stover							1.18			102
Silage			2.44							102

Crop part	Residue, mg/kg, at interval, days, after last application									Ref.
	21-28	30-39	40-43	50-59	60-69	70-79	80-89	90-99	>100*	
Popcorn									<0.02 (111)	102
Stover									0.04(111)	102
Silage				<0.02						102
Popcorn									<0.02 (121)	102
Stover									<0.02 (121)	102
Silage						<0.02				102
Sweet corn**				<0.02						104
Kernels				<0.02						104
Cannery waste***				0.14						104
Forage				0.30						104
Sweet corn						<0.02				104
Kernels						<0.02				104
Cannery waste						<0.02				104
Forage						<0.02				104
Sweet corn					<0.02					104
Kernels					<0.02					104
Cannery waste					<0.02					104
Forage					<0.02					104
Sweet corn					<0.02					104
Kernels					<0.02					104
Cannery waste					<0.02					104
Forage					<0.02					104
Sweet corn				<0.02						104
Kernels				<0.02						104
Cannery waste				<0.02						104
Forage				<0.02						104
Sweet corn				0.04						104
Kernels				0.02						104
Cannery waste				0.23						104
Forage				0.97						104
Sweet corn				0.05						104
Kernels				<0.02						104
Cannery waste				0.02						104
Forage				0.68						104
Sweet corn				0.14						104
Kernels				0.12						104
Cannery waste				0.32						104
Forage				1.01						104
Sweet corn		<0.02								104
Kernels		<0.02								104
Cannery waste		0.17								104

Crop part	Residue, mg/kg, at interval, days, after last application									Ref.
	21-28	30-39	40-43	50-59	60-69	70-79	80-89	90-99	>100*	
Forage		0.18								104
Sweet corn		0.05								104
Kernels		0.04								104
Cannery waste		0.29								104
Forage		1.00								104
Sweet corn		0.62								104
Kernels		0.13								104
Cannery waste		2.65								104
Forage		3.95								104
Sweet corn		<0.02								104
Kernels		<0.02								104
Cannery waste		0.03								104
Forage		0.15								104
Sweet corn	0.06									104
Kernels	0.05									104
Cannery waste	0.10									104
Forage	0.57									104
Sweet corn	0.22									104
Kernels	0.17									104
Cannery waste	0.93									104
Forage	1.74									104
Sweet corn	0.26									104
Kernels	0.08									104
Cannery waste	1.00									104
Forage	0.23									104

* PHI, days, in parentheses
** Kernel plus cobs with husks removed
*** Cannery waste including husk plus cob

Table 14. Residues of ethephon in wheat from supervised trials (PHIs 34-89 days). Underlined residues are from treatments according to GAP.

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application				Ref.
	Form.	kg ai/ha	34-41	49-59	60-69	70-89	
USA, 1981, grain	SL480	0.56			<0.02		88
grain		0.84		0.16			88
straw		0.84		0.28			88
grain		0.56		<u>0.05</u>			88
grain		0.84		0.07			88
straw		0.84		0.58			88
grain		0.84	0.15				88
straw		0.84	5.84				88

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application				Ref.
	Form.	kg ai/ha	34-41	49-59	60-69	70-89	
grain		0.56		0.05			88
grain		0.56		0.04			88
straw		0.56		0.36			88
grain		0.56			0.15		88
straw		0.56			3.37		88
UK, 1982, grain	EC480	0.48				<0.05	26
grain		0.48				<0.05-0.07	26
straw		0.48				0.24	26
grain		0.48				<0.05	26
straw		0.48				0.21	26
grain		0.48				<0.05	26
straw		0.48				0.33	26
USA, 1981, grain	SL480	0.56			0.41		88
grain		0.56			0.03		88
grain		0.56		0.02			88
straw		0.56		0.05			88
grain		0.84		0.34			88
straw		0.84		4.23			88
grain		0.56		0.04			88
straw		0.56		1.33			88
grain		0.56			<0.02		88
straw		0.56			0.16		88
grain		0.56			<0.02		88
USA, 1989, grain	SL39.9%	0.56	0.61				62
straw		0.56	1.53				62
grain		0.56	0.40				62
straw		0.56	1.47				62
grain		0.56			0.16		62
straw		0.56			1.30		62
USA, 1989, grain	SL39.9%	0.56	0.65				62
straw		0.56	1.33				62
grain		0.56	0.58				62
straw		0.56	1.70				62
grain		0.56			0.23		62
straw		0.56			3.37		62
USA, 1989, grain	SL39.9%	0.56	0.53				62
straw		0.56	2.73				62
grain		0.56	0.33				62
straw		0.56	1.30				62
grain		0.56			0.10		62
straw		0.56			0.78		62
USA, 1989, grain	SL39.9%	0.56	0.68				62
straw		0.56	3.23				62

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application				Ref.
	Form.	kg ai/ha	34-41	49-59	60-69	70-89	
grain		0.56	0.33				62
straw		0.56	0.94				62
grain		0.56			0.10		62
straw		0.56			0.31		62
USA, 1989, grain		0.56	0.13				62
straw		0.56	1.41				62
grain		0.56	0.12				62
straw		0.56	1.66				62
grain		0.56		0.05			62
straw		0.56		0.66			62
USA, 1989, grain	SL39.9%	0.56	0.08				62
straw		0.56	1.01				62
grain		0.56	0.08				62
straw		0.56	1.27				62
grain		0.56		<0.05			62
straw		0.56		0.29			62
USA, 1989, grain	SL39.9%	0.56	0.25				62
straw		0.56	2.00				62
grain		0.56	0.14				62
straw		0.56	1.43				62
grain		0.56			0.08		62
straw		0.56			0.33		62
USA, 1989, grain	SL39.9%	0.56	0.33				62
straw		0.56	2.73				62
grain		0.56	0.15				62
straw		0.56	1.61				62
grain		0.56			0.08		62
straw		0.56			0.20		62
USA, 1989, grain	SL39.9%	0.56	0.30				62
straw		0.56	1.20				62
grain		0.56			0.24		62
straw		0.56			1.83		62
grain		0.56				0.15	62
straw		0.56				1.33	62
USA, 1989, grain	SL39.9%	0.56	0.15				62
straw		0.56	0.95				62
grain		0.56			0.14		62
straw		0.56			0.95		62
grain		0.56				0.07	62
straw		0.56				1.47	62
USA, 1990, grain	SL39.9%	1.12			0.17		63
dust combined					0.10		63
bran					0.23		63

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application				Ref.
	Form.	kg ai/ha	34-41	49-59	60-69	70-89	
middling					<0.05		63
low grade flour					<0.05		63
patent flour					<0.05		63
shorts and germ					0.25		63
red dog					0.20		63
CAN, 1981, grain	SL480	0.56		0.35			88
bran				1.21			88
flour				0.02			88
germ				0.71			88
shorts				0.78			88
CAN, 1982, grain	SL480	0.60		0.35			86
grain		0.36		0.17			86
grain		0.56		0.62			86
grain		0.84			0.13		86
straw		0.84			0.16		86
grain		0.56				0.08	86

Rice. Rice was treated with Cerone^{R} in five Sates of the USA and Costa Rica. The crop was treated at the tilling stage and sampled at the mature stage 48-69 days after treatment. The treatment rates were 560 g/ha in the USA and 360 g/ha in Costa Rica. Residues of ethephon in grain were <0.01-0.46 mg/kg. The straw contained residues of 0.01-1 mg/kg (Table 18).

Table 15. Residues of ethephon in wheat from supervised trials in Europe (PHIs 0-71 days).

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	0	6-7	10-14	28-30	47-50 or as shown in parentheses	
GER, 1981, green plant	SL480	0.48	8.20		0.84 0.46	0.15	0.11	89
grain							0.02 (56)	89
straw							0.09 (56)	89
green plant			8.78		0.49 0.35	0.17	0.09	89
grain							0.03 (66)	89
straw							0.04 (66)	89
GER, 1982, green plant	SL480	0.48	3.84	1.54	0.90	0.47	<0.04	91
grain							<0.02 (59)	91
straw							<0.04 (59)	91
green plant	SL480	0.48	11.25	0.54	0.32	0.43	<0.04	91
GER, 1984, green plant	SL480	0.36	1.71	1.35	0.68	0.48	<0.01	101
grain							<0.02 (58)	101
straw							<0.1 (58)	101
green plant	SL480	0.36	8.54	<0.01	1.38	0.37	<0.01	101

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	Form.	kg ai/ha	0	6-7	10-14	28-30	47-50 or as shown in parentheses	
grain							<0.02 (71)	101
straw							<0.1 (71)	101
GER, 1984, green plant	SL480	0.36	4.54	1.46	1.16	0.68	0.46	98
grain							0.03	98
straw							0.24	98
green plant	SL480	0.36	6.64	2.32	1.80	0.53	0.30	98
grain							<0.02	98
straw							0.19	98
UK, 1990, leaves	EC480	0.36	14.0					44
grain							0.10 (70)	44
leaves		0.36	13.8					44
grain							0.06 (65)	44
leaves		0.36	2.37					44
grain							0.10 (58)	44
leaves		0.72	3.1					44
grain							0.47 (58)	44

Table 16. Residues of ethephon in oats from supervised trials with SL 480g/l formulation.

Country, Year, Crop part	g ai/ha	Residue, mg/kg, at interval, days, after last application							Ref.
		0	11	21-22	29-30	41-47	57-63	82-89	
GER, 1976, Green plant	0.31	9.34		<0.01	<0.01				76
Dry plant						0.08	0.07		76
Grain							<0.01		76
GER, 1976, Green plant	0.31	2.61		0.35					76
Dry plant					0.04		0.06 0.31		76
Grain					<0.01		0.03 0.03		76
GER, 1976, Green plant	0.31	3.50		1.39					76
Dry plant						0.38 0.30	0.28		76
Grain						0.06 0.19			76
GER, 1976, Green plant	0.31	4.28	<0.01						76
Dry plant								0.51 <0.01	76
Grain								<0.01 <0.01	76
USA, 1981, Grain	0.56					0.22			88

Sugar cane. A single field trial was conducted with ethephon applied aerially at 2.24 kg ai/ha, about five times the recommended US rate. The ethephon residue in the cane stalks decreased rapidly from about 4.6 mg/kg immediately after treatment to about 1.3 mg/kg one week later, then more gradually to about 0.2 mg/kg at maturity 79 days after treatment (Union Carbide, 1980b).

In another study conducted by the Hawaiian Sugar Planters' Association in 1984, no residues (<0.01 mg/kg) were detected in leaf or stalk samples 8 months to one year after a treatment with 0.56 kg/ha of Ethrel[R].

Table 17. Residues of ethephon in rye from supervised trials with SL 480 g/l formulation in Europe. Underlined residues are from treatments according to GAP.

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application										Ref
	kg ai/ha	spray l/ha	0	7	14	22-30	37-41	49-56	58-63	77-87	90-99	100-110	
GER, Green plant, 1976	0.31		1.57			0.26		0.04					76
dry plant									0.35		0.41		76
grain									<0.01	<0.01	0.04		76
Green plant, 1976	0.31		1.43			1.80							76
dry plant								0.71	0.55				76
grain								0.13	0.20				76
Green plant, 1976	0.31		3.88			1.55	0.35						76
dry plant												0.04 <0.01	76
grain												0.02	76
Green plant, 1975	0.31		9.04			0.32	0.02			<0.01			76
dry plant											0.52 0.59		76
grain											<0.01		76
Green plant, 1976	0.31		15.5			3.28		0.56	<0.01				76
dry plant										1.38			76
grain										0.24			76
Green plant, 1975	0.31		8.2			0.69		0.38					76
dry plant										0.34 0.42			76
grain										0.04			76
Green plant, 1980	0.72	400	10.5	1.70	0.45	0.10		0.05					81
grain											<0.05		81
straw											0.05		81
Green plant, 1980	0.72	400	10.8	1.60	0.60	0.15		<0.05					81
grain											<0.05		81
straw											<0.05		81
Green plant, 1980	0.72	400	1.5	0.60	0.20	0.03		<0.01					81
grain										<0.01			81
straw										<0.01			81
Green plant, 1980	0.72	400	2.4	0.40	0.40	0.03		<0.01					81
grain										<0.01			81

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application										Ref
	kg ai/ha	spray l/ha	0	7	14	22-30	37-41	49-56	58-63	77-87	90-99	100-110	
straw										<0.01			81
Green plant, 1980	0.96	400	5.4		0.80	0.04		<0.01					81
grain											<0.01		81
straw											<0.01		81
Green plant, 1980	0.96	400	4.7		0.60	0.05		<0.01					81
grain											<0.01		81
straw											<0.01		81
Green plant, 1980	0.96	400	6.3		1.10	0.20		0.04					81
grain										<0.01			81
straw										<0.01			81
Grain, 1983	1.40	400									0.25		93
straw											0.15		93
UK, Grain, 1982	0.48	300										<0.05-0.08	24
straw												0.17	24
Grain, 1982	1.92											0.44	24
straw												0.77	24
Grain, 1982	0.48	300									0.13		24
straw											0.34		24
grain	1.92										0.84		24
straw											1.05		24
Grain, 1982	0.48	220									<0.05		24
straw											0.32		24
Grain, 1982	0.48	200									<0.05-0.09		24
straw											0.21		24
FIN, Grain, 1979	0.3									0.02 (75)			22
Grain, 1980	0.3									<0.01			22
grain										0.02			22
Grain, 1982	0.72										<0.02		22
grain										0.05			22
Grain, 1978	2.5									0.5 (76)			22
Grain, 1979	2.5									0.3 (75)			22
Grain, 1980	1.4									0.07			22
grain										0.4			22
NET, 1974, Grain	0.96											0.27	32
straw	0.96											0.74	32

Hazelnuts (filberts). Filberts were treated with Ethrel^{R} and sampled after 7-39 days in the USA. Samples taken 7 days after treatment were pulled off the trees manually: all other samples were harvested "naturally" (i.e. after they had fallen to the ground). Residues in undried nuts were in the range 0.03-0.1 mg/kg following application close to the maximum recommended rate (0.76 kg ai/ha). The residues in dried nuts were in about the same range. The results are shown in Table 19.

Macadamia nuts. Supervised trials were carried out with Ethrel[R] on three commercially important varieties from the two major macadamia nut growing areas on the island of Hawaii.

Most of the analysed samples had been treated once with 500, 750, 1000, 1500 or 2000 mg/l Ethrel[R], 3, 6 or 9 days before harvest. In addition, three sets of samples were obtained from trees which had been treated twice, approximately 10 weeks apart, with 750 or 1000 mg/l Ethrel[R]. These samples were taken 3, 6 or 9 days after the second treatment. The rates of applications cover the GAP reported from Australia.

None of the 30 samples analysed showed residues of ethephon above the limit of determination of the analytical method, which was estimated to be 0.01 mg/kg (Union Carbide, 1975a).

Walnuts. Thirteen varieties of walnuts, all grown in California, were treated at the expected rate of use (500 or 750 mg/l) or at an exaggerated rate (1000 mg/l) using Ethrel[R] plant growth regulator in 1970. At PHIs between 7 and 36 days, the residues did not exceed 0.08 mg/kg. In a recent study in California at a rate of 1.4 kg ai/ha at PHIs of 5 to 20 days, the highest residue was 0.27 mg/kg and the average value was below 0.1 mg/kg. The results are shown in Table 19.

Table 18. Residues of ethephon in rice from supervised trials with SL 480 g/l formulation.

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application			
	kg ai/ha	spray l/h	48	58-59	60-62	69
USA, 1987, grain	0.56	374			0.01	
straw					1.0	
grain	0.56	374			0.46	
straw					0.50	
grain	0.56	374			0.02	
straw					0.04	
grain	0.56	93				0.03
straw						0.02
grain	0.56	93				0.02
straw						0.02
grain	0.56	187			0.04	
straw					0.16	
grain	0.56			0.13		
straw				0.30		
grain	0.56	187			<0.01	
straw					0.01	
grain	0.56	93		0.03		
straw				0.06		
grain	0.56	187			0.02	
straw					0.04	
grain	0.56	187			<0.01	
straw					0.01	
grain	0.56	244			0.05	
straw					0.18	

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application			
	kg ai/ha	spray l/h	48	58-59	60-62	69
grain	0.56	182		0.01		
straw				0.03		
COS, 1987, grain	0.36	83	0.03			
straw			0.17			

Cotton seed. Trials in the USA in 1989 used ground (10 trials) or air application (9 trials). Rates of 0.56 + 1.68 kg ai/ha and 2.24 kg ai/ha were applied in each trial. PHIs were 6-21 days. Residues in cotton seed treated at 1.68 kg/ha (the maximum recommended rate is 1.73 kg ai/ha) contained residues from <0.02 to 2.13 mg/kg. The sampling interval or mode of application did not have an observable effect on the residue levels (Table 20).

Table 19. Residues of ethephon in tree nuts from supervised trials with SL 240 g/l formulation in the USA. Underlined residues are from treatments according to GAP.

Commodity, Year	kg ai/ha	Residue, mg/kg, at interval, days, after last application						Ref.
		5-7	10	14-16	18-20	28-31	35-39	
Hazelnut meat, 1972	0.69						0.1	5
	1.87						0.17	5
dried	1.87						0.11	5
Hazelnut meat, 1972	0.69						0.03	5
	1.87						0.05	5
dried	1.87						0.04	5
Hazelnut meat, 1972	0.69					0.04		5
	1.87					0.08		5
dried	1.87					0.04		5
Hazelnut meat, 1972	0.69					0.06		5
	1.87					0.16		5
dried	2.06					0.14		5
Hazelnut meat, 1972	1.87				0.06			5
	3.74				0.31			5
	2.80				0.07			5
	4.20				0.12			5
	5.60				0.23			5
	2.80	0.01						5
dried	2.80	0.05						5
	4.20	0.06						5
	5.60	0.16						5
Walnut meat, 1969	1 g/l	0.08 0.01						74

Commodity, Year	kg ai/ha	Residue, mg/kg, at interval, days, after last application						Ref.
		5-7	10	14-16	18-20	28-31	35-39	
		0.10						
Walnut meat, 1970	0.75 g/l				0.04			74
Walnut meat, 1970	0.75 g/l				0.03			74
Walnut meat, 1970[1]	1 g/l	0.57 0.26 0.53 0.03 0.15 0.21 <0.01 0.31 0.18 0.34						74
Walnut meat, 1970	0.75 g/l			0.08				74
Walnut meat, 1970	0.5 g/l						0.08	74
Walnut meat, 1970	0.75 g/l			0.09				74
Walnut meat, 1970	0.5 g/l	0.06 0.04						74
Walnut meat, 1970	0.75 g/l			0.02				74
Walnut meat, 1991	1.40	0.27	0.23		0.12			65
Walnut meat, 1991	1.40	0.03	0.09					65
Walnut meat, 1991	1.40	0.05	0.03	0.06				65
Walnut meat, 1991	1.40	0.02	0.04		0.02			65
Walnut meat, 1991	1.40	0.02	0.03	0.02				65

[1] Different varieties from California

Rape seed. Seven trials were conducted at seven different locations in Germany to determine residues of ethephon in winter rape treated with Cerone^{R} 480 at a rate of 0.96 kg ai/ha, using 400 l/ha. At harvest, residues ranged from not detected to 1.83 mg/kg in straw and from not detected to 1.24 mg/kg in grain. In the UK winter rape treated with Cerone^{R} at rates from 0.36 to 0.72 kg ai/ha had residues of ethephon in the grain at harvest (92 and 118 days after treatment) from <0.05 to 0.09 mg/kg and up to 0.16 mg/kg at 28-35 days. The residues are shown in Table 21.

Coffee. Trials were conducted in Brazil, Costa Rica and Guatemala in 1972 at application rates from 120 to 960 mg ai per plant. Coffee beans were sampled 13 and 30 days after treatment. Residues of ethephon were between <0.01 and 0.14 mg/kg (Table 22).

Table 20. Residues of ethephon in cotton seed from supervised trials with SL 55.4% formulation in the USA. Underlined residues are from treatments according to GAP.

Sample, Year	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	kg ai/ha	spray l/ha	6	7-8	10-11	13-14	20-21	
Seed, ginned, 1978	2.24			0.01				79
Seed, ginned, 1978	2.24						0.01	79

Sample, Year	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	kg ai/ha	spray l/ha	6	7-8	10-11	13-14	20-21	
Seed, ginned, 1978	2.24			0.12				79
Seed, ginned, 1978	2.24			0.16				79
Seed, ginned, 1978	2.24		0.08					79
Seed, ginned, 1978	2.24					0.32		79
Seed, ginned, 1978	2.24		0.09					79
Seed, ginned, 1978	2.24				0.03			79
Seed, ginned, 1978	2.24						0.15	79
Seed, ginned, 1978	2.24					0.03		79
Ginned seed-hulls, 1978	2.24			<0.01	0.02		0.03	79
Cotton seed meal	2.24			<0.01	0.18	1.29	0.18	79
Crude cotton seed oil	2.24			<0.01	<0.01	<0.01	<0.01	79
Refined cotton seed oil	2.24			<0.01	0.06	0.05	0.01	79
Cottonseed soapstock	2.24			<0.03	<0.03	<0.03	<0.03	79
Ginned seed- hulls, 1978	2.24					<0.01		79
Cotton seed meal						0.19		79
Crude cotton seed oil						0.02		79
Refined cotton seed oil						0.04		79
Cottonseed soapstock						<0.03		79
Seed, 1989	0.56 + 1.68	20 aerial		0.18	0.76	0.39		48
Seed, 1989	2.24	20 aerial		0.23	0.47 0.47	0.58		48
Seed, 1989	0.56 + 1.68	167 ground		0.49	0.70	0.64		48
Seed, 1989	2.24	167 ground		0.58	0.75	0.70		48
Seed, 1989	0.56 + 1.68	47 aerial		0.40	0.44	0.59		48
Seed, 1989	2.24	47 aerial		0.55	0.95	0.45		48
Seed, 1989	0.56 + 1.68	103 ground		1.77	1.15	2.13		48
Seed, 1989	2.24	103 ground		2.37	2.17	1.93		48
Seed, 1989	0.56 + 1.68	140 ground		0.12	0.14	0.19		48
Seed, 1989	2.24	140 ground		0.10	0.18	0.24		48
Seed, 1989	0.56 + 1.68	47 aerial		0.08	0.08	0.06		48
Seed, 1989	2.24	47 aerial		0.10	0.09	0.16		48
Seed, 1989	0.56 + 1.68	138 ground		0.22	0.05	0.03		48
Seed, 1989	2.24	138 ground		0.06	0.05	0.02		48
Seed, 1989	0.56 + 1.68	19 aerial		<0.02	<0.02	<0.02		48
Seed, 1989	2.24	19 aerial		0.03 0.10	<0.02	<0.02		48
Seed, 1989	0.56 + 1.68	155 ground		0.21	0.10	0.06		48
Seed, 1989	2.24	155 ground		0.31	0.34	<0.02		48
Seed, 1989	0.56 + 1.68	21.5 aerial		0.17	0.27	0.06		48
Seed, 1989	2.24	21.5 aerial		0.65	0.35	0.36		48
Seed, 1989	0.56 + 1.68	18.6 aerial		0.70	0.89	0.78		48
Seed, 1989	2.24	18.6 aerial		0.54	0.91	1.4		48
Seed, 1989	0.56 + 1.68	132 ground		0.59	0.88	0.60		48
Seed, 1989	2.24	132 ground		0.30	0.86	0.79		48
Seed, 1989	0.56 + 1.68	139 ground		1.2	1.5	1.2		48

Sample, Year	Application		Residue, mg/kg, at interval, days, after last application					Ref.
	kg ai/ha	spray l/ha	6	7-8	10-11	13-14	20-21	
Seed, 1989	2.24	139 ground		1.5	1.1	1.5		48
Seed, 1989	0.56 + 1.68	140 ground		0.77	0.18	1.0		48
Seed, 1989	2.24	140 ground		0.50	0.09	0.1		48
Seed, 1989	0.56 + 1.68	140 ground		0.58	0.50	0.05		48
Seed, 1989	2.24	140 ground		0.61	0.42	0.07		48
Seed, 1989	0.56 + 1.68	17 air		0.40	0.35	0.10		48
Seed, 1989	2.24	17 air		0.44	0.16	0.22		48
Seed, 1989	0.56 + 1.68	51 aerial		0.21	0.18	0.08		48
Seed, 1989	2.24	51 aerial		0.35	0.21	0.05		48
Seed, 1989	0.56 + 1.68	187 ground		0.32	0.14	0.12		48
Seed, 1989	2.24	187 ground		0.36	0.16	0.19		48
Seed, 1989	0.56 + 1.68	20 aerial		<0.02	<0.02	0.06		48
Seed, 1989	2.24	20 aerial		0.03	<0.02	0.11		48

Table 21. Residues of ethephon in rape from supervised trials with SL 480 g/l formulation. Underlined residues are from treatments according to GAP.

Country, Year Crop part	Application	Residue, mg/kg, at interval, days, after last application	Ref.

	kg ai/ha	spray l/ha	0	14-17	28-35	74-83*	92-98*	115-118	
GER, 1987, Whole plant	0.96	400	2.2	1.65	0.26				108
shell					0.48				
grain							1.24		
straw							1.0		
GER, 1987, Whole plant	0.96	400	2.3	0.47	0.45				108
shell					0.79				
grain							1.22		
straw							1.83		
GER, 1987, Whole plant	0.96	400	12.4	1.73	0.16				107
shell					0.20				
grain								<0.02	
straw								0.08	107
GER, 1987, Whole plant	0.96	400	8.5	1.96	0.29				106
shell					0.39				
grain						<0.02			
straw						0.04			
GER, 1984, Green plant	0.96	400	3.2	0.62	0.11				96
shell					0.07				
grain						<0.02			
straw						<0.04			
GER, 1984, Green plant	0.96	400	12.1	4.50	0.20				99
shell					0.28				
grain						<0.05			
straw						<0.05			
GER, 1984, Whole plant	0.96	400	5.1	0.80	0.06				103
grain							<0.01		
straw							<0.01		
UK, 1982, Grain	0.48	360			0.16 <0.16				
grain	0.36	360						<0.05 <0.05	92
grain	0.48	360						<0.05 <0.05	92
grain	0.60	360						<0.05 <0.05	92
grain	0.48	300		0.17 0.12					92
grain	0.36	300					<0.05 <0.05		92
grain	0.48	300					<0.05 <0.05		92
grain	0.60	300					0.07 0.09		92
UK, 1982, Grain	0.36	360						<0.05	25
grain	0.48	360						<0.05	25
grain	0.72	360						0.08	25
grain	0.48				0.14				25
grain	0.36	300					<0.05		25

Country, Year Crop part	Application		Residue, mg/kg, at interval, days, after last application						Ref.
	kg ai/ha	spray l/ha	0	14-17	28-35	74-83*	92-98*	115-118	
grain	0.48	300					<0.05		25
grain	0.72	300					<0.05		25
grain	0.48	300		<0.16-0.16					25
FIN, 1987, Oil seed*	0.35						<0.01 (91 days)		22
oil seed	0.35					<0.01 (85 days)			22
oil seed	0.35					<0.01 (56 days)			22
oil seed	0.48						0.02 (88 days)		22

Table 22. Residues of ethephon in dried mature coffee beans from supervised trials in 1972 with SL 240 formulation (Ref. 6).

Country	mg/plant	Residue, mg/kg, at interval, days, after last application		
		13	22	30
BRA	120	0.02		
	240	0.03		
	480	0.06		
	960	0.14		
COS	240		<0.01	
	480		<0.01	
	600		<0.01	
GUA	672	0.06		
GUA	120			<0.01
	240			0.05
	360			<0.01

Animal commodities

Ten dairy cows were assigned at random into three groups of three with one control. They were fed twice a day. Ethephon, 10% w/w in corn starch, was administered orally in gelatine capsules at each feeding for 28 days at rates equivalent to 0, 1.0, 5.0 and 20 ppm ai in the feed. Milk samples were collected at the morning and evening milkings on days 0, 1, 2, 4, 7, 14, 21 and 28 (treatment period) and 29, 30, 32 and 35 (withdrawal period). The feed consumption and milk production were not affected by the treatment. Apparent residues in the milk were in the same range (0.02-0.08 mg/l) before and after the 28 days administration of ethephon, indicating that there were no detectable residues in any of the milk samples (Affiliated Medical Research, 1972).

In another experiment three groups of 3 dairy cows were administered ethephon for 28 days at 15, 50 and 150 ppm based on the feed. No ethephon residues (<0.05mg/l, the limit of determination) were detected in any of the milk samples from the 15 and 50 ppm groups. Of the fifteen samples analysed from days 19 and 27 from the 150 ppm group, ten contained residues below 0.05 mg/kg and the others 0.14, 0.1, 0.14, 0.12 and 0.11 mg/kg. The tissues analysed included muscle, heart, fat, liver and kidney. The liver of one of the three animals treated at 150 ppm contained 0.2 mg/kg ethephon, but residues were not detectable in any other samples (<0.1 mg/kg in muscle and <0.2 mg/kg in heart, fat,

liver and kidney) (Union Carbide, 1982b).

FATE OF RESIDUES

In animals

Several studies have been conducted on animals including cows, goats and poultry.

Two lactating goats were dosed with [^{14}C]ethephon at a level of 10 ppm in the diet for 7 days. Urine, faeces, milk and blood samples were collected daily. Volatiles were collected for 24 hours on the seventh day of the study. Approximately 16 hours after the last dose the animals were slaughtered and tissues collected. A major portion (31%) of the administered ^{14}C was lost as volatiles (ethylene 29%; CO_2 2%). Urine, faeces and gut contained 19.1%, 6.6% and 0.8%, respectively. Average radiocarbon levels in whole milk increased for 3.5 days and then remained at 0.38-0.42 mg/kg ethephon equivalents between 3.5 and 7 days. The total milk collected over the seven-day period contained 3.3% of the administered radioactivity. Kidney and liver had the highest residue levels, at 1.18 and 1.2 mg/kg ethephon equivalents respectively, while fat, heart and muscle contained 0.5, 0.16 and 0.1 mg/kg (Union Carbide, 1984a).

Two groups of 6 Leghorn laying hens were dosed once a day by gelatin capsule for five consecutive days with [^{14}C]ethephon at a level equivalent to 53 ppm in the feed. A third group was kept as a control. Eggs and excreta were collected from all three groups while [^{14}C]ethylene and $^{14}CO_2$ were collected from one group only.

Ten hens were killed approximately 22-23 hours after the last dose and muscle (composite of leg and breast), fat, kidneys and liver were collected for analysis. Approximately 26-30% of the administered radioactivity was recovered in the excreta and about 58% as ethylene. The ^{14}C in the CO_2, eggs and tissues accounted for less than 1% of the total radioactivity administered. The ^{14}C detected in the eggs from days 1 to 5 amounted to 0.002, 0.022, 0.082, 0.183 and 0.179 mg/kg ethephon equivalents respectively. The yolk contained approximately 80-90% of the residue, of which 72.4% could be extracted with a hexane-methanol mixture. The average total ^{14}C residues (mg/kg) were 0.3 in liver, 0.2 in kidney, 0.02 in muscle and 0.15 in fat (Rhône-Poulenc, 1992).

In plants

Metabolism studies have been conducted with ^{14}C- and ^{32}P-labelled ethephon on a wide variety of crops including apples, cherries, cantaloupes, citrus, cucumbers, figs, grapes and raisins, hazelnuts, olives, peaches, pineapples, squash, rubber, tomatoes, tobacco and walnuts, (Amchem, 1972a; Domir, 1978; Edgerton and Hatch, 1969, 1970; Martin *et al*., undated; Palmer *et al*., 1970; Union Carbide, 1968, 1981a; Yamaguchi *et al*., 1970).

All of these studies demonstrated that ethylene is the only significant metabolite of ethephon in plants. In apples, citrus, tomatoes, cucumbers, grapes, olives, walnuts, pineapples, cantaloupes and figs no other metabolites were produced.

In cherries extracts of leaves, but not of fruit, contained unidentified radioactive material accounting for about 5% of the applied ^{14}C in addition to ethephon and ethylene. Similarly an unidentified "metabolite" accounting for about 2% of the applied ^{14}C was found in the extracts from treated squash plants.

In peaches autoradiography indicated a product which was identified as an adduct of ethephon with sugars in the fruit. It was concluded that the binding of ethephon to sugars was involved in the

translocation of the compound and that it was not a metabolic reaction. A similar adduct of ethephon with glucose was obtained from excised rubber bark incubated with [^{14}C]ethephon. It therefore seems reasonable to assume that the "metabolites" seen in the squash and cherry extracts were actually adducts of ethephon with sugars in the plant.

The only apparent metabolite apart from ethylene produced in any system was â-D-glucopyranose-1-(2-chloroethyl) phosphonate, a conjugate of ethephon, which was produced to the extent of less than 4% of the applied ethephon in the excised rubber bark study. However it should be noted that the study was not conducted with an intact plant.

Since the plants studied represent a wide range of crop groups, it may be assumed that the metabolism of ethephon in almost any plant system would produce ethylene as the only significant metabolite (Union Carbide, 1981).

In processing

The effects of processing on residues were studied in apples, cranberries, grapes, pineapples, peppers, tomatoes, sugar cane, cotton seed, olives and wheat.

Apples. A complete processing trial was conducted in 1989 in the USA in order to determine the concentration factor for ethephon residues in dried pomace. The apples were treated with ethephon at 1.6 kg ai/ha seven days before harvest of the ripe fruit. Samples were analysed in triplicate. The fresh whole apples had an average residue of 0.37 mg/kg, and the dried pomace 0.73 mg/kg (Rhône-Poulenc, 1990b). The twofold concentration factor implies that a 10 mg/kg MRL would be required for dried apple pomace.

Cranberries. A study was conducted in Canada to determine possible residues of ethephon resulting from treatments with Ethrel^{R} at application rates from 1.1 to 2.2 kg ai/ha. Several sets of cranberry samples, taken at 0 or 7-10 days PHI, were processed into cranberry sauce by a method which approximated commercial practice. The process involved washing the berries, heating them with water to boiling, agitating and cooking for 5 min, preparing puree by removing skins and seeds in a "finisher", adding sugar to the puree, reheating and boiling for 5-7 min, cooling in running cold water for 10 min, canning, allowing to gel for 12 days at ambient temperature, and freezing aliquots for analysis. Residues in fruit, sauce and "cocktail" are shown in Table 23 and the average residues found in berries and puree before and after gelling in Table 24.

Processing decreased the residue level in cranberries taken at 0 or 7-10 days by average factors of 6.9 and 1.5 in freshly frozen puree, and by factors of 11.5 and 2.15 in cranberry gel, respectively.

Grapes. In a study conducted in 1978, Thompson Seedless grapes at six California locations were treated with 0.56 kg ai/ha as Ethrel^{R} 42 to 47 days before harvest (Amchem 1979). The maximum concentration factor from grapes to raisins found in this study was 5.3, the second highest being 4.5. All others were less than 3.5, which was the maximum found in an earlier study recorded in Table 5.

Pineapples. Pineapples were treated with ethephon twice at a rate of 2.24 kg ai/ha, 6 months and two days before harvest. The fruits were processed on the day of harvest in a commercial cannery. The weights of individual fruit fractions could not be obtained without disrupting the processing (Rhône-Poulenc, 1992l).

The bran, used as cow fodder, is prepared from the discarded peels by exposing them to flame at a temperature of 1500-1666°C for 1.5 hours. The fractions obtained and the residues found are given in Table 25.

During processing the ethephon residue was concentrated only in the pulp (1.2-fold) and bran (5.3-fold). Apparent monochloroacetic acid residues were extremely low (about 0.002 mg/kg) and were at the same level in the untreated controls and the treated samples.

Peppers. A study was conducted with fresh sweet and hot peppers grown at two locations in California, USA, to study the effect of dehydration on the residue levels. Peppers were treated with Ethrel^{R} at rates of 0.84 and 1.12 kg ai/ha. The residues measured are shown in Table 9. The results indicate that the ethephon residue calculated on a dry weight basis is decreased by about 79-98% when sweet and hot peppers are commercially dehydrated.

Table 23. Residues of ethephon in cranberries and their processed products from supervised trials in Canada, 1973, with SL 240 formulation (Ref. 7).

Commodity	kg ai/ha	Residue, mg/kg, at interval, days, after last application			
		0-1	4-7	8-9	10
Cranberry sauce, gelled[1]	1.12	0.58	0.50	0.33	0.32
Cranberry sauce, gelled[1]	2.24	1.28	1.41	0.10	1.17
Cranberry fruit	1.12	4.82	2.44		1.15
Cranberry sauce, ungelled[2]	1.12	0.53	0.51		0.39
Cranberry fruit	2.24	8.94	5.62		3.29
Cranberry sauce, ungelled[2]	2.24	1.16	1.51		1.17
Cranberry fruit	1.12	4.38		0.09	
Cranberry cocktail	1.12	0.02		0.01	
Cranberry fruit	2.24	7.2		1.46	
Cranberry cocktail	2.24	0.03		0.07	

[1] Samples were allowed to gel for 2 days at ambient temperature before freezing.
[2] Samples was frozen immediately after canning.

Table 24. Average ethephon residues in processed cranberry products.

Commodity	Residues, mg/kg			
Fresh berries	7.6 (0 day)	4.0 (0 day)	1.7	0.75
Puree (frozen after canning)	1.2	0.53	1.3	0.45
Puree (allowed to gel)	0.68	0.34	0.94	0.3

Table 25. Residues of ethephon in pineapple processing fractions.

Sample	Residue, mg/kg	
	Range	Average
Fresh whole fruit	0.16-0.2	0.18
Slices (canned with juice)	0.04-0.05	0.05
Beverage juice (canned)	0.06-0.08	0.07
Ion exchange juice	0.1-0.18	0.15
Pulp	0.21-0.25	0.22
Bran	0.92-0.98	0.95

Tomatoes. Processing trials were conducted in the USA (California and Florida) to determine ethephon residues in tomato processing fractions. Tomatoes treated with 2.41 kg ai/ha and untreated controls were processed. Control tomatoes were stored overnight at about 7.2°C before processing next day. Treated tomatoes were harvested 3 days after treatment and processed on the same day. Samples of the processed products were analysed for ethephon (Rhône-Poulenc, 1991d) and monochloroacetic acid (Rhône-Poulenc, 1992o). The approximate mass balance of the process and the residues found in the processed fractions are shown in Table 26.

Table 26. Residues of ethephon in processed tomatoes.

Tomato sample	% of original weight	Ratio of original wt. to wt. of processed fraction	Range of residues, mg/kg	Average residue, mg/kg
Fresh whole	100	1	0.64-0.86	0.73
Washed whole	100	1	0.67-0.70	0.68
Wet pomace	11.3	8.8	0.34-0.43	0.38
Dry pomace	1.9	53	0.98-1.6	1.39
Canned fresh juice	88.7	1.1	0.22-0.30	0.25
Canned puree	44.4	2.25	0.43-0.44	0.44
Canned paste	14.8	6.8	0.46-0.63	0.55
Canned juice from concentrate	85.5	1.2	0.26-0.33	0.29

Sugar cane. Ethrel^{R} (240 g/l) was used in trials conducted in Louisiana (Rhône-Poulenc, 1988) and Hawaii (Union Carbide, 1980b), in which cane was harvested periodically over a 79-88-day interval following aerial treatment at rates of 1.68 to 2.24 kg/ha. These were about 3½5 times the recommended US rate, and the higher rates were chosen to obtain measurable residues in processed fractions. The processing flow diagram is shown in Figure 1.

The average ethephon residues found in mature cane, sugar, and by-products after PHIs of 70 days (Hawaii) and 88 days (Louisiana) and the average concentration factors are given in Table 27. As the application rates were about four times the recommended rate only the concentration factors can be used for estimating likely residue levels in the sugar and by-products.

Analysis of the processing fractions shows that there is a substantial loss of ethephon during the step from mixed to clarified juice. The concentration factors derived from the two experiments show good agreement except for raw sugar. As most of the ethephon in the raw sugar is probably concentrated in the molasses film covering each crystal and this film is removed during the refining process, residues of ethephon in refined sugar would probably be considerably lower than in raw sugar.

Table 27. Ethephon residues and concentration factors (C_f) found in sugar cane processing[1].

Sample	Louisiana		Hawaii	
	Av. residue, mg/kg	C_f[1]	Av. residue, mg/kg	C_f[1]

[1] The concentration factor calculation was based on 0.13 mg/kg ethephon in the stalk before milling, as the residue in the mature cane did not correspond to the residues measured in bagasse and first pressed juice.

Sample	Louisiana		Hawaii	
	Av. residue, mg/kg	Cf[1]	Av. residue, mg/kg	Cf[1]
Mature cane	0.04		0.29	
Raw sugar	0.06	0.46	0.28	0.97
Molasses	0.69	5.3	2.17	7.5
Bagasse	0.05	0.38	0.1	0.34
Mixed (first pressed) juice	0.14	1.08	0.37	1.3
Clarified juice	0.07	0.54		
Syrup	0.37	2.8	0.93	3.2
Settling mud	0.14-0.27	0.46	0.22	0.76

Cotton seed. Cotton was treated with Ethrel^{R} at 2.24 kg ai/ha (Union Carbide, 1979a). Ginned cotton seed was processed and the fractions analysed. The results are shown in Table 20. Ethephon residues (mg/kg) were <0.01-0.32 in ginned cotton seed, <0.1-0.03 in hulls, <0.01-1.29 in meal, <0.01-0.02 in crude oil, <0.01-0.06 in refined oil and <0.03 in soapstock. The maximum concentration factors found were hulls/seed 0.67, meal/seed 6.33, crude oil/seed 0.67, and refined oil/seed 2.0.

Figure 1. Flow diagram of sugar cane processing.

CANE + water

MIXED JUICE + BAGASSE

CLARIFIED JUICE + CLARIFIED MUD

SYRUP + water

RAW SUGAR + MOLASSES + water

Olives (to oil). Two trials were conducted, one in Italy using Ethrel^{R} (240 g/l) at a rate of 2.70 kg/ha and the other in Tunisia using Ethrel^{R} (480 g/l) at rates from 13.53 to 21.01 g/tree. In both cases residue levels in the oil were very low when the olives were harvested 6-7 days after treatment. They varied from <0.01 to 0.012 mg/kg. (Neotron, 1989; Rhône-Poulenc, 1992).

Wheat and barley. Samples of wheat and barley grain, treated with 0.56 kg ai/ha Ethrel^{R}, 53 or 49 days before mature harvest, were milled to provide normal mill fractions for analysis. The residue level was found to be 0.02 mg/kg in the flour.

Winter wheat grain samples from a single trial in Texas were processed and the resultant fractions were analysed for residues of ethephon. Cerone^{R} plant growth regulator was applied at 1.12 kg ai/ha (twice the recommended rate). Processing was done by the Food Protein Research and Development Center at

Texas A & M University. The treated grain contained 0.17 mg/kg ethephon. Bran, shorts and germ, grain dust and red dog contained 0.23, 0.25, 0.10 and 0.20 mg/kg ethephon respectively. No quantifiable residues were found in the other fractions (middlings, low grade flour, and patent flour). The maximum concentration of the residues by processing was 1.5 times (in wheat shorts and germ) (Rhône-Poulenc, 1992n). The results are shown in Table 14.

Stability of residues in stored analytical samples

The stability of ethephon residues in stored samples has been studied in several crops. Because ethephon technical grade contains an impurity (the 2-chloroethyl ester) from which monochloroacetic acid (MCAA) can theoretically be derived by decomposition, the stability of MCAA residues were also studied. The freezer temperature generally ranged from -18 to -26°C during the storage. In apple, blackberry, cotton seed, pepper, tomato and pineapple samples the freezer temperature rose to -10°C for 10 days owing to a freezer fault. Since no appreciable breakdown of ethephon occurred in the samples this did not matter.

A study to determine the stability of ethephon in or on meat, milk and eggs during frozen storage has been completed (start date: March 9, 1992; termination date: June 15, 1993), but the results are not yet available.

In apples. Analysis of apples spiked with ethephon at 1.0 mg/kg showed that ethephon is stable in or on frozen apples for periods up to two years (Rhône-Poulenc, 1992c).

In blackberries. Analysis of blackberries spiked with ethephon at 1.0 mg/kg showed that ethephon is stable in or on fruit when stored frozen or freeze-dried and stored at room temperature for at least two years (Rhône-Poulenc, 1990c, 1992d). Blackberries spiked with MCAA at 0.1 mg/kg showed a loss of about 13% after 344 days under frozen storage conditions (Upalawanna, 1992b).

In cherries. Analysis of cherries spiked with ethephon at 1.0 mg/kg showed that ethephon is stable in or on fruit when stored frozen or freeze-dried and stored at room temperature for at least 24 months. (Rhône-Poulenc, 1992f).

In grapes. Grapes were spiked with ethephon at 0.5 mg/kg. Ethephon was stable in or on the fruit when stored frozen or freeze-dried and stored at room temperature for at least two years (Rhône-Poulenc, 1992g). Grapes and raisin waste spiked with MCAA at 0.1 mg/kg showed a loss of about 3% on grapes after 351 days and about 60% on raisin waste after 356 days under frozen storage conditions (Upalawanna, 1992d).

In pineapples. Analysis of pineapples spiked with ethephon at 0.5 mg/kg showed that ethephon is stable in or on fruit when stored frozen or freeze-dried and stored at room temperature for at least two years (Rhône-Poulenc, 1992i). Pineapple spiked with MCAA at 0.1 mg/kg showed a loss of about 18% after 359 days under frozen storage conditions (Upalawanna, 1992e).

In cantaloupes. Analysis of cantaloupes spiked with ethephon at 1.0 mg/kg showed that ethephon is stable in or on frozen fresh fruit for a period of at least six months. Ethephon appears to be stable in freeze-dried cantaloupes stored at room temperature for up to four months, but breakdown averaging about 50% occur after six months (Rhône-Poulenc, 1992e). Cantaloupes spiked with MCAA at 0.1 mg/kg showed a loss of about 13.5% after 359 days under frozen storage conditions (Upalawanna, 1992c).

In peppers. When whole fresh peppers were spiked with ethephon at 1.0 mg/kg and stored frozen, the residue was stable for at least two years. Ethephon in or on whole peppers stored at room temperature after freeze-drying showed degradation with a half-life of about 10 months. (Rhône-Poulenc, 1992h).

In tomatoes. Analysis of tomatoes spiked with ethephon at 0.5 mg/kg showed that ethephon is stable in or on fruit when stored frozen or freeze-dried and stored at room temperature for at least two years (Rhône-Poulenc, 1992j). Tomatoes spiked with MCAA at 0.1 mg/kg showed a loss of about 21% after 344 days under frozen storage conditions (Upalawanna, 1992f).

In wheat. Analysis of wheat grain and wheat straw spiked with ethephon at 0.5 mg/kg showed that ethephon is stable in or on frozen wheat grain and straw for up to six months (Rhône-Poulenc, 1992a,b). Wheat grain and wheat straw spiked with MCAA at 0.1 mg/kg showed a loss of about 12% and 15% respectively after 1 year under frozen storage conditions (Upalawanna, 1992a).

In walnuts. Weighed samples of walnuts were spiked with ethephon at 0.20 mg/kg. Some samples were stored frozen, others at room temperature and others were freeze-dried and then stored at room temperature. Ethephon was stable for at least three months in or on walnuts when stored frozen, but appeared to be degraded slowly on freeze-dried walnuts stored at room temperature and rapidly when stored at room temperature on nut-meats that had been removed from the shell. Losses were more than 60% after one week of storage.

After walnuts had aged for about nine months, it apparently became impossible to obtain an adequate recovery of 0.2 mg/kg from samples spiked immediately before analysis. It was concluded that walnut samples should be analysed within three months of harvest until a better analytical method is found. (Rhône-Poulenc, 1991b).

In cotton seed. When cotton seed was spiked with ethephon at 1.0 mg/kg, residues were stable in seed stored frozen for six months. Cotton seed spiked with MCAA at 0.1 mg/kg showed a loss of about 24% of the residue after 341 days under frozen storage conditions. (Rhône-Poulenc, 1992k; Uppalawanna, 1992g).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The results of surveys carried out in The Netherlands since 1975 on some fruits and vegetables are summarized in Table 28 (Netherlands, 1993)

Table 28. Residues (mg/kg) of ethephon found in commodities moving in commerce in The Netherlands.

Commodity, year	Number of samples falling in range (mg/kg)										
	<LOD	>LOD-0.5	>0.5-1	>1-2	2-3	3-4	4-5	5-6	6-7	7-8	>8
Tomato, 1975	34	16		12	2	1	0	0	1	1	1
Tomato, 1976	27	29		6	2	1	6	1	1		
Tomato, 1977	52	6		1							
Tomato, 1978	84	22		5		3					
Tomato, 1982	15	5				0					
Tomato, 1983	53	1	1								
Pears, 1983	4	2	1								
Apples, 1983	24	4									
Other vegetables	6										

METHODS OF RESIDUE ANALYSIS

The principle of the analytical method for the determination of residues has always been the same since

the early 1970s. Samples are divided into separate components as may be necessary. They are then hard frozen and, unless liquid or sauce, ground to give a homogeneous preparation. This is followed by freeze drying unless the sample has a very low moisture content.

The analysis consists of extraction with methanol, pH adjustment, precipitation of interfering materials, esterification and final quantification by gas chromatography using a flame-photometric detector in the phosphorus mode or an alkali flame-thermionic detector.

The method is sensitive to 0.05 mg/kg for all crops (5 to 20 g sample size). Recoveries in the 70-120% range, depending on the crop or the fraction analysed, are acceptable. General and matrix-specific methods are available (Rhône-Poulenc, 1989b,c; Union Carbide, 1984b).

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs reported to the Meeting are summarized below. The limits are expressed as the parent ethephon.

Commodity/Country	ARG	AUL	AUS	BEL	BRA	CAN	ECU	FRA	GER	HUN	ISR
Apple	2	1	2				5				5
Barley		1	1	0.1		0.5	2	0.05	1		
Cherries (sweet)	1	15	5			8	10			1	
Cherries (sour)		15				2					
Citrus	0.5										
Coffee	0.1				0.1		0.1				
Cotton (seed)	2	1									
Cotton (seed oil)		0.1									
Cucumber										1	
Flax				0.1				0.05			
Gherkins										1	
Grape		10				1					2
Kiwi fruit		0.1									
Lemon	2										
Macadamia nuts		0.1									
Mandarins		2									
Meat, mammalian		0.1									
Milk of cattle, goats and sheep		0.05									
Morellos										1	
Oat							2				
Olives	0.1										0.1
Oranges		2									
Peaches	2	0.5									
Pears	0.1										
Peppers										2	
Pimento	2										
Pineapple		2			0.5		2				

Commodity/Country	ARG	AUL	AUS	BEL	BRA	CAN	ECU	FRA	GER	HUN	ISR
Plums										1	2
Rape									0.1		
Redcurrants										1	
Rice							0.3				
Rye				0.1					1		
Sugar cane	2				0.5		0.1				
Tobacco	0.1										
Tomatoes	2	2	3	3	1.5	3	2			1	2
Triticale		1		0.1							
Walnuts							0.5				
Wheat		1		0.1		0.5	2	0.05	1		
Winter									1		

Commodity/Country	ITA	JPN	NET	NZE	PER	SAF	SPA	SWI	USA	VEN
Apple	2		2	2	5	3			5	
Bananas					2					
Barley	0.5	0.5	0.5					0.5	2	
Cantaloupes									2	
Cherries (sweet)		2	1						10	
Cherries (sour)			1			3				
Cherries (yellow)		2				3				
Citrus						2				
Coffee	0.5								0.1	
Cotton (seed)									2	
Cotton (seed meal)									5	0.2
Cranberries									5	
Cucumber					0.1					
Figs					5					
Filberts									0.5	
Garlic					5					
Grape		2			2	5			2	
Macadamia nuts									0.5	
Mangoes					2					
Melon					0.1					
Olives					2					
Onion			0.5		5					
Oranges					2					
Peaches		2				3				
Pears		2								
Peppers									30	
Persimmon		2								

Commodity/Country	ITA	JPN	NET	NZE	PER	SAF	SPA	SWI	USA	VEN
Pineapple		2				1			2	
Plums					5	3				
Potatoes					5					
Pumpkin					0.1					
Rice										20
Rye	0.5		0.5					0.5		
Sugar cane						0.05				
Sweet peppers			3							
Tomatoes	3	3	3	1					2	
Triticale								0.5		
Walnuts									0.5	
Wheat	0.5	0.5	0.5			2			2	
Zucchini					0.1					

APPRAISAL

The present evaluation is part of the CCPR periodic review programme.

Residue aspects of ethephon were reviewed by the JMPR in 1977, 1978, 1983 and 1985. As an ADI had not been allocated, Guideline Levels were estimated in 1977 and 1978. In view of the time since these estimates, information on current use patterns as well as further residue data and critical supporting studies were required to enable the estimation of maximum residue levels.

Ethephon is a systemic plant growth regulator belonging to the phosphonate family. It is readily absorbed by the plant and releases ethylene which is a natural plant hormone. Ethylene influences directly several physiological processes (e.g. ripening, maturation) and stimulates the endogenous production of ethylene.

Use patterns, usually including one or two treatments at various growing stages of the plants, were reported for a wide range of crops from many countries.

A number of supervised field trials were conducted on several crops in typical geographical regions. Parent ethephon residues were determined in various crop parts and residues of monochloroacetic acid (MCAA), a potential decomposition product of an impurity (the 2-chloroethyl ester) in technical grade ethephon, were also determined in blackberries, grapes, pineapples, tomatoes and cantaloupes. Residues of MCAA were below the limit of determination (<0.01 mg/kg) in all samples.

Residues of ethephon were stable in the treated crops and did not show substantial changes with time, so the PHI usually had little influence on the estimated maximum residue levels.

Apples were treated at rates of 0.5 and 1 kg ai/ha, within the recommended range in the USA, with Ethrel® and harvested from 3 to 13 days after application. Samples taken at day 7 (the PHI corresponding to the lower rate) after treatments with 0.5-0.67 kg ai/ha showed residues varying from 0.37 to 2.32 mg/kg. The highest residues found at longer PHIs (corresponding to the higher rate) were 1.19, 1.76 and 2.04 mg/kg irrespective of the rate. The Meeting concluded that the residue data supported a limit of 5 mg/kg.

Analyses of cherry samples taken 7-8 and 13-14 days after applications in accordance with current use patterns showed residues of 0.69-6.6 mg/kg and <0.01-3.93 mg/kg respectively. The results support the previous estimate of 10 mg/kg.

In a trial conducted in Australia, peaches were treated at rates of 0.2 and 0.4 kg ai/ha and analysed at harvest two to three weeks after treatment. In the three samples analysed residues of ethephon were 0.18 and 0.21 mg/kg from 0.2 kg/ha and 0.46 mg/kg from the higher rate. There were no results at the current PHI of 5 days. The Meeting withdrew the previous estimate.

Blackberry samples from trials in the USA in 1974 and 1989 at rates of 1.12-2.8 kg/ha showed residue levels ranging from 8 to 18 mg/kg 1-3 days after application. Thee recommended rate is 1.27 kg ai/ha. The data are not sufficient to estimate maximum residue level. The Meeting withdrew the previous estimate.

The residues in blueberries from US trials according to Canadian use patterns were 1.4-19 and 2.1-9.1 mg/kg 4 and 39 days after the treatments respectively. The GL recorded previously (20 mg/kg) covers these residues and is now recommended as an MRL.

Samples of cranberries taken 4-7 and 8-14 days after applications at 1.1 kg/ha contained residues of 0.2-2.4 and 0.1-1.1 mg/kg respectively. Residues were below 0.4 mg/kg at 17-21 days after application. No GAP was reported for cranberries. The Meeting withdrew the previous estimate.

Grapes should be treated at least 14 days before harvest. Residues were in the range 0.09-0.82 mg/kg in samples harvested between 14 and 47 days after treatment at 0.56 kg ai/ha. Residues decreased from the range 0.07-2.2 mg/kg with an average of 0.93 mg/kg at day 7 to 0.16-0.47 mg/kg with an average of 0.28 mg/kg at 45 days and <0.01 mg/kg at 91-108 days.

Residues in raisins from grapes treated about 45 days before harvest ranged from 0.21 to 1.49 mg/kg. The maximum concentration factor found from grapes to raisins was 3.5. Raisin waste from grapes harvested 45 days after treatment showed ethephon residue levels of 3.27 to 38 mg/kg with an average of 15.1 mg/kg. In view of the current use patterns the Meeting estimated a maximum residue level of 1 mg/kg in grapes.

Following treatments with 0.38-1 kg ai/ha in Canada, residues in dried figs derived from fruits treated 14-15 days before harvest were in the range 0.32-8.5 mg/kg. The application conditions correspond to US registered uses. Samples taken at 21-41 days contained residues between 0.22 and 2.73 mg/kg and did not show any significant differences at different PHIs. The new residue data support a limit of 10 mg/kg for dried figs. As no residue data were available for fresh figs, the Meeting withdrew the previous estimate.

The concentration of ethephon residues in pineapples following two or three applications did not depend on the rate of the early treatments. When the last treatment was at or below the maximum rate, whole fruit samples taken 7-14 days after the last application contained residues between <0.02 and 1.1 mg/kg with an average of 0.17 mg/kg (9 samples). The residues in 21 pulp samples ranged from 0.06 to 0.33 mg/kg with an average of 0.18 mg/kg. Following two applications at double rates (2.55 + 2.24 kg ai/ha), the maximum residue in the whole fruit was 1.3 mg/kg. On the basis of current US GAP, the Meeting estimated a maximum residue level of 1 mg/kg.

Trials were conducted with four varieties of cucumber at application rate of 0.25 kg ai/ha (one or two treatments) and at PHIs between 28 and 48 days. No residues were found in any of the 9 samples analysed above the 0.01 mg/kg limit of determination. As the trial conditions do not correspond to reported GAP, the Meeting was not able to estimate a maximum residue level.

Residues in whole cantaloupes from recommended treatments were in the range 0.04-0.4 mg/kg 2-4 days after application. Residues of 0.55 mg/kg in the pulp and 0.69 mg/kg in the peel at day 2 indicate that residues in the whole fruit would be between 0.5 and 1 mg/kg. Residue levels reported in the 1977 evaluation were in the same range when the applications were made at the currently recommended rates. The ratio of the residue in the peel to that in the pulp varied with the time after application. The peel always contained higher residues. The Meeting estimated a maximum residue level from current use patterns of 1 mg/kg.

As no residue data were reported for other varieties of melon, the Meeting withdrew the previous estimate for melons, except watermelon.

In Peppers treated at 1.12 kg ai/ha, close to the recommended rate, residues ranged from 3.5 to 26.3 mg/kg 5 to 8 days after application. This is similar to the range reported in the 1977 evaluation. In Canadian trials at 0.75 kg ai/ha the residues varied from 0.72 to 1.1 mg/kg 7-8 days after treatment. The new results support the previously recorded GL of 30 mg/kg, which is now recommended as an MRL.

Tomatoes treated with 1.8 kg ai/ha (the maximum recommended rate in the USA is 1.5 kg ai/ha) contained residues in the range 0.09-1.4 mg/kg 3 to 7 days after application. The rate and sampling intervals also cover the current use patterns established in other countries. On the basis of the new results and those reported in the 1977 Evaluations the Meeting estimated a maximum residue level of 2 mg/kg according to current GAP.

In sweet corn treated at 0.56 kg ai/ha, the residues (in kernels plus cobs with husks removed) ranged from <0.02 to 0.62 mg/kg at sampling intervals of 21-39 days, while five of the eight samples analysed showed residues of less than 0.02 mg/kg 50-79 days after application. The residues were 0.04, 0.05 and 0.14 mg/kg in the other samples. The forage contained residues from 0.15 to 3.95 mg/kg and <0.02 to 1 mg/kg at the shorter and longer sampling intervals respectively. No GAP was reported for sweet corn.

The residues in peas ranged from <0.01 to 0.05 mg/kg and for pea vines from 0.12 to 1.26 mg/kg between 30 and 56 days after application. No GAP was reported.

Residues in mature barley grain ranged from <0.02 to 0.69 mg/kg 35-90 days after application. In one trial 0.78 mg/kg was detected 7 weeks after treatment, but residue levels in the grain at harvest were generally below 0.05 mg/kg. The straw contained residues up to 1.7 mg/kg.

Numerous trials conducted at a rate of 0.56 kg ai/ha, close to the maximum rate registered in the USA, showed residues in mature wheat grain in the range 0.08-0.68 mg/kg at sampling intervals of 34-41 days after application. Residues in the straw varied between 0.95 and 3.23 mg/kg.

Following recommended uses, residues at harvest ranged from <0.01 to 0.3 mg/kg in oat and rye grain and from 0.35 to 1.4 mg/kg in the straw.

In view of the similar use patterns on barley, rye and wheat, the residues were assessed together. It was concluded that the results were mutually supportive and the Meeting estimated maximum residue levels of 1 mg/kg in barley, rye and wheat grain, and 5 mg/kg in the corresponding straws.

As no GAP was reported for oats, no limit could be recommended.

Fifteen of 19 maize grain samples analysed showed residues of less than 0.02 mg/kg (limit of determination). Residues in the remaining four samples ranged from 0.03 to 0.12 mg/kg. At harvest, the residues in popcorn kernels were <0.02 mg/kg in 6 samples and 0.19 mg/kg in one sample. Silage and stover contained residues from 0.02 to 2.44 and 0.05 to 1.18 mg/kg respectively. As the trial conditions

did not correspond to reported GAP, residue limits could not be recommended.

Rice was treated with 0.36-0.56 kg ai/ha at the tilling stage and sampled at the mature stage 48-69 days later. Residues in the grain were <0.01-0.46 mg/kg. The straw contained residues between 0.04 and 1 mg/kg. No GAP was reported.

A single field trial was conducted on sugar cane with ethephon applied aerially at 2.24 kg ai/ha, about five times the recommended rate. The ethephon residue in cane stalks decreased rapidly from about 4.6 mg/kg immediately after treatment to about 1.3 mg/kg one week later, then more gradually to about 0.2 mg/kg at maturity, 79 days after application. The available information is not sufficient to estimate a maximum residue level.

Residues in hazelnuts (filberts) were in the range 0.03-0.1 mg/kg 28 to 39 days after application at 0.69 kg ai/ha, close to the recommended rate of 0.76 kg/ha. The residues in dried nuts after treatment at 1.87 kg ai/ha were in about the same range. The Meeting was informed that GAP corresponding to the trial conditions was under consideration in the USA. When the recommended use pattern is followed the residues would be below 0.2 mg/kg, which was estimated as a maximum residue level.

Thirteen varieties of walnuts were treated at recommended rates (500 or 750 mg/l) or at an exaggerated rate (1000 mg/l) in California. At PHIs between 7 and 36 days the residues were below 0.3 mg/kg and the average residue was below 0.1 mg/kg. The results support the previously estimated GL of 0.5 mg/kg.

Macadamia nuts were treated once with 500 to 2000 mg/l Ethrel^{R}, 3, 6 or 9 days before harvest or twice, approximately 10 weeks apart, with 750 or 1000 mg/l. None of the 30 samples analysed showed residues of ethephon above the limit of determination (0.01 mg/kg). The trial conditions cannot be related to the reported use pattern, so a maximum residue level could not be estimated.

Residues in cotton seed treated at about the maximum recommended rate (1.68 kg ai/ha) contained residues from 0.12 to 2.1 mg/kg. Neither the sampling interval (7-14 days) nor the mode of application (ground or aerial) had an observable effect on the residue levels. The results indicate that residues in cotton seed are unlikely to exceed 2 mg/kg when GAP is followed. The Meeting estimated 2 mg/kg as a maximum residue level.

In rape at harvest, residues ranged from undetected to 1.8 mg/kg in the straw and from undetected to 1.2 mg/kg in the seed. As samples were not taken at registered PHIs (30-49 days), a maximum residue level could not be estimated.

Coffee beans were sampled 13 and 30 days after treatment at rates from 120 to 960 mg ai per plant. The residues were between <0.01 and 0.15 mg/kg. The data could not be evaluated because the GAP application rate is expressed in kg ai/ha and the trial data in mg ai/plant. The Meeting agreed to withdraw the previous estimate for coffee beans, 0.1 mg/kg.

No residue data were provided for black currants, lemons, limes, mandarins or onions. The estimates recorded as GLs by the previous Meetings are therefore withdrawn.

Metabolism studies have been conducted with ^{14}C- and ^{32}P-labelled ethephon on a wide variety of crops, including apples, cherries, cantaloupes, citrus, cucumbers, figs, grapes and raisins, hazelnuts, olives, peaches, pineapples, squash, rubber, tomatoes, tobacco and walnuts. These studies demonstrated that ethylene and phosphoric acid are the only significant metabolites in plants. The latter is taken up into the plant phosphate cycle. No other metabolites were produced in apples, citrus, tomatoes, cucumbers, grapes, olives, walnuts, pineapples, cantaloupes or figs.

Extracts of cherry leaves, but not of fruit, contained unidentified radioactive material accounting for about 5% of the applied ^{14}C in addition to ethephon and ethylene. Similarly an unidentified "metabolite" accounting for about 2% of the applied ^{14}C was found in the extracts from treated squash plants.

In peaches autoradiography indicated a product which was identified as an adduct of ethephon with sugars in the fruit. It was concluded that the binding of ethephon to sugars was involved in the translocation of the compound and was not a metabolic reaction. A similar adduct of ethephon with glucose was produced (to the extent of less than 4% of the applied ethephon) when excised rubber bark was incubated with [^{14}C]ethephon. It was identified as â-D-glucopyranose-1-(2-chloroethyl) phosphonate, a conjugate of 2-chloroethylphosphonic acid.

The excretion of ethephon and the levels of its residues in products of animal origin were studied in cows, goats and poultry.

Ten dairy cows were fed twice a day for 28 days at rates of 0, 1, 5 or 20 ppm in the feed. Milk samples were collected at the morning and evening milkings on days 0, 1, 2, 4, 7, 14, 21 and 28 (the treatment period) and on days 29, 30, 32 and 35 in the withdrawal period. No residues were detected in any milk samples after 28 days feeding.

In another experiment dairy cows were administered ethephon at levels equivalent to 15, 50 or 150 ppm in the feed for 28 days. No ethephon residues (<0.05 mg/kg) were detected in any of the milk samples from the 15 and 50 ppm groups. Of the fifteen samples analysed during days 19 and 27 from the 150 ppm group, residues in ten were below 0.05 mg/kg while the other samples contained 0.14, 0.1, 0.14, 0.12 and 0.11 mg/kg. The tissues analysed were muscle, heart, fat, liver and kidney. The liver of one of the three animals treated at 150 ppm contained 0.2 mg/kg ethephon but residues were not detectable in any other samples (<0.1 mg/kg in muscle and <0.2 mg/kg in heart, fat, liver and kidney).

Two lactating goats were dosed with [^{14}C]ethephon at a level equivalent to 10 ppm in the diet for 7 days. Urine, faeces, milk and blood samples were collected daily. Volatiles were collected for 24 hours on the seventh day of the study. Approximately 16 hours after the last dose the animals were slaughtered and tissues collected. Analysis for total radiocarbon showed that a major proportion (31%) of the administered dose was lost as volatiles (ethylene 29%, CO_2 2%). Urine, faeces and gut contained 19.1%, 6.6% and 0.8% respectively. Average radiocarbon levels in whole milk increased for 3.5 days and then reached a plateau (0.38-0.42 mg/kg ethephon equivalents) between 3.5 and 7 days. The total milk collected over the seven-day period contained 3.3% of the administered radioactivity. Kidney and liver had the highest residue levels, at 1.18 and 1.2 mg/kg ethephon equivalents respectively, while fat, heart and muscle contained 0.5, 0.16 and 0.1 mg/kg.

Two groups of Leghorn laying hens, each consisting of 6 birds, were dosed once a day by gelatin capsule for five consecutive days with [^{14}C]ethephon at a level equivalent to 53 ppm in the feed. Ten hens were slaughtered approximately 22-23 hours after the last dose, and muscle (composite of leg and breast) and fat samples, the kidneys and the liver were collected for analysis. All samples were analysed for their radioactive contents. Approximately 26-30% of the administered radioactivity was recovered in the excreta, and about 58% was recovered as ethylene. The $^{14}CO_2$ trap, eggs and tissues accounted for less than 1% of the total radioactivity administered. The ^{14}C (as ethephon equivalents) in the eggs on days 1 to 5 was about 0.002, 0.022, 0.082, 0.183 and 0.179 mg/kg. The yolk contained approximately 80-90% of the residue of which 72.4% could be extracted with a hexane-methanol mixture. The average total ^{14}C residues (mg/kg) were 0.3 in liver, 0.2 in kidney, 0.02 in muscle and 0.15 in fat. Phosphonic acid was not identified in any of the samples.

In view of the residue levels in feed grains and plant by-products (e.g apple pomace, cotton seed meal, pineapple bran, sugar cane molasses, stover etc), the maximum ethephon residue in composite cattle or goat feed is unlikely to exceed 2 mg/kg. Even less would be expected in poultry feed. Consequently

animal tissues, milk and eggs should not contain any ethephon residues above the limits of determination of current analytical methods. The Meeting estimated the LODs as maximum residue levels.

The effect of processing on residues was studied in apples, cranberries, grapes, peppers, tomatoes, sugar cane, cotton seed, olives and wheat.

Fresh whole apples containing an average residue of 0.37 mg/kg yielded dried pomace containing an average of 0.73 mg/kg, showing a concentration factor of about 2.

Several sets of cranberry samples were processed into cranberry sauce by a method which approximated commercial practice. Processing reduced the residue level in cranberries sampled 0 and 7-10 days after treatment by average factors of 6.9 and 1.5 in freshly frozen puree, and of 11.5 and 2.15 in cranberry gel, respectively.

The highest concentration factors from grapes to raisins found in a processing study were between 3.5 and 5.3. Residues in wine were at about the same level as in the corresponding grape samples or somewhat lower.

The ethephon residue was reduced by about 79% and 98%, calculated on a dry weight basis, when sweet and hot peppers were commercially dehydrated.

The average residues (mg/kg) found in processed fractions of fresh tomato containing 0.73 mg/kg ethephon were as follows: wet pomace 0.38, dry pomace 1.39, canned fresh juice 0.25, canned puree 0.44, canned paste 0.55, canned juice from concentrated puree 0.29.

Sugar cane was treated at three to four times the proposed label rate to obtain measurable residues during processing. The average ethephon residues (mg/kg) found in two studies were: mature cane 0.13, 029; raw sugar 0.06, 0.28; molasses 0.69, 2.17; mixed juice 0.14, 0.37; syrup 0.37, 0.93. There was a substantial loss of ethephon during the clarification step.

As the application rate was four times the recommended maximum rate only the concentration factors can be used to estimate the residue levels in processed sugar and by-products.

Ginned cotton seed was processed and the fractions analysed. Ethephon residues (mg/kg) were <0.01-0.32 in ginned cotton seed, <0.01-0.03 in cotton seed hulls, <0.01-0.19 in cotton seed meal, <0.01-0.02 in crude oil, <0.01-0.06 in refined oil and <0.03 in soapstock. Since the initial residues in the cotton seed were much lower than the estimated maximum residue level and the apparent ethephon residues were higher in oil from untreated than from treated plants, the reported studies do not provide sufficient data to estimate residue levels in crude or refined oil.

Apparent monochloroacetic acid residues were extremely low and at the same level in the control and treated samples, showing that no additional monochloroacetic acid was derived from the ethephon application.

It is unlikely that residues would ever be found in alkali-refined oil or in soapstock because ethephon is extremely unstable in bases.

Residue levels in olive oil prepared from seeds harvested 6-7 days after treatment varied from <0.01 to 0.012 mg/kg.

Treated wheat grain contained 0.17 mg/kg ethephon. The wheat bran, wheat shorts and germ, and wheat grain dust derived from it contained 0.23, 0.25 and 0.10 mg/kg ethephon respectively. No

quantifiable residues were found in the other fractions (middlings, low grade flour and patent flour). The maximum concentration factor from processing was 1.5 times (in wheat shorts and germ). The residues in the wheat were too low compared with the likely maximum residue level to obtain a realistic estimate of maximum residues in the processed fractions.

Storage stability tests with ethephon and monochloroacetic acid (MCAA) were carried out with several crops. The results showed that ethephon is stable at about -20°C or at room temperature after freeze-drying in or on spiked apples, blackberries, cherries, grapes, pineapples, peppers and tomatoes for at least two years. Studies are being conducted on wheat, cotton seed and cantaloupes. Until a new method is available, walnut samples should be analysed within three months of harvest. Samples of grapes, pineapples, tomatoes and cotton seeds spiked with MCAA showed a loss of 3-24% after one year under frozen storage conditions.

The principle of the analytical method for the determination of residues has not changed since the early 70s. It consists in extraction with methanol, pH adjustment, precipitation of interfering materials, esterification and final quantification by gas chromatography using a flame-photometric detector or an alkali flame-thermionic detector in the phosphorus mode. The limits of determination are between 0.01 and 0.05 mg/kg for all crops, 0.05 mg/kg for milk, 0.1 mg/kg for muscle and 0.2 mg/kg for other animal tissues. Recoveries range from 70 to 120%.

The Meeting agreed to maintain the definition of the residue as ethephon because the parent compound amounts to >95% of the residue and the analytical method used for determining the residues in supervised trials measures the parent compound.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting estimated the maximum residue levels listed below. As an ADI has now been established they are recommended for use as MRLs.

Definition of the residue: ethephon

Commodity		Recommended limit (mg/kg)		PHI on which based, days
CCN	Name	New MRL	Previous GL	
FP 0226	Apple	5	5	6-11
GC 0640	Barley	1		35-90
AS 0640	Barley straw and fodder, dry	5		
FB 0264	Blackberries	W	30	
FB 0020	Blueberries	20	20	0-14
VC 4199	Cantaloupe	1	2[1]	0-4
FS 0013	Cherries	10	10	0-14
PE 0840	Chicken eggs	0.2*		
SB 0716	Coffee beans	W	0.1	

Commodity		Recommended limit (mg/kg)		PHI on which based, days
CCN	Name	New MRL	Previous GL	
SO 0691	Cotton seed	2		7
FB 0265	Cranberry	W	5	
FB 0278	Currant, Black	W	5	
MO 0096	Edible offal of cattle, goats, horses, pigs and sheep	0.2*		
FT 0297	Fig	W	5	
DF 0297	Figs, dried	10		
FB 0269	Grapes	1	10	7-14
TN 0666	Hazelnuts	0.2	0.5	
FC 0002	Lemons and Limes	W	2	
FC 0003	Mandarins	W	0.5	
MM 0096	Meat of cattle, goats, horses, pigs and sheep	0.1*		
VC 0046	Melons, except Watermelon	W[2]	2	3-4
ML 0107	Milk of cattle, goats and sheep	0.05*		
VA 0385	Onion, Bulb	W	0.5	
FS 0247	Peach	W	0.5	
HS 0790	Peppers	30	30	5-8
FI 0353	Pineapple	1	2	1-14
PO 0111	Poultry, edible offal of	0.2*		
PM 0110	Poultry meat	0.1*		
GC 0650	Rye	1		49-99
AS 0650	Rye straw and fodder, dry	5		
VO 0448	Tomato	2[3]	3 Po	3-7
TN 0678	Walnuts	0.5	0.5	5-7
GC 0654	Wheat	1		34-41
AS 0654	Wheat straw and fodder, dry	5		

* residues at or about the limit of determination.
[1] For Melons, except Watermelon
[2] Replaced by separate limit for cantaloupe
[3] Currently listed uses do not include Po

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