DELTAMETHRIN (135)

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EXPLANATION

Deltamethrin is a non-systemic synthetic pyrethroid insecticide used in agriculture and public and animal health as a broad spectrum insecticide in a wide range of fruit, vegetable and field crops. It is also recommended for use against locusts, indoor insects and pests of stored grain and timber and for the control of ticks, mites and insect pests of livestock.

Deltamethrin was first reviewed by JMPR in 1980 with subsequent residue reviews between 1984 and 1992. Full periodic reviews were conducted for toxicology in 2000 and for residues in 2002. Residues resulting from the veterinary uses of deltamethrin were evaluated by JECFA in 1999 and in 2003.

The 2000 JMPR established an ADI of 0–0.01 mg/kg bw/day and an acute RfD of 0.05 mg/kg bw for deltamethrin and the residue definition established by the 2002 JMPR for plant and animal commodities, for both compliance with MRLs and for dietary intake assessment is the *sum of deltamethrin and its α-R- and trans-isomers*. The 2002 JMPR also concluded that the residue is fat-soluble but that residues in milk should be measured on the whole milk.

Specifications for deltamethrin technical material and relevant formulations have been established by the JMPS, most recently in January 2015, and published on the AGP-FAO Specifications webpage.

The 47th Session of the CCPR (2015) listed deltamethrin for further evaluation by the 2016 JMPR for additional MRLs and the current Meeting received GAP information and supporting residue information from the manufacturer for rape seed (canola).

METHODS OF RESIDUE ANALYSIS

Analytical methods

The 2002 JMPR reviewed and summarized analytical method descriptions and validation data for deltamethrin and its α-R, cis- and trans–isomers, generally involving extraction with varying proportions of polar and non-polar solvents (depending primarily upon the nature of the matrix being extracted and its water content), a primary liquid – liquid partition to transfer deltamethrin residues to less polar solvents prior to column clean-up and residue determination by GC with an electron capture detector (ECD).

The analytical methods used in the supervised field trials on rape seed were similar to the above, involving hexane or hexane/acetone extraction, clean-up of reduced extracts by gel permeation chromatography (GPC) or GPC plus alumina column and residue determination by GC with ECD. The LOQ of these methods was 0.012 to 0.02 mg/kg for deltamethrin, α-R-deltamethrin and trans-deltamethrin.

Table 1 Summary of deltamethrin analytical methods developed for rape seed

Matrix	Analyte	Method	Principle	LOQ (mg/kg)	Reference
Soya bean Soya bean fractions Rape seed	α-R deltamethrin cis-deltamethrin trans--deltamethrin	XM-10	Hexane extraction GPC+alumina column clean-up GC-ECD analysis	0.012 0.015 0.019	B002857
Rape seed Rape seed fractions	α-R deltamethrin cis-deltamethrin trans--deltamethrin	BP/01/98	Acetone/hexane extraction Hexane:water partition GPC clean-up GC-ECD analysis	0.02 0.02 0.02	B002972

Data collection methods

XM-10

This method was developed for soya beans and with minor modifications was used to measure residues of α-R, cis- and trans–deltamethrin in rape seed. Seed samples were extracted with hexane in the presence of sodium chloride with the extracts filtered under vacuum, reduced to dryness using a rotary evaporator and reconstituted in 85:15/cyclohexane:ethyl acetate. The reconstituted solutions were subjected to gel permeation chromatography (GPC) followed by a second alumina column clean-up. After evaporation and reconstitution in hexane, residues were analysed by GC using an ECD. LOQs for rape seed were 0.012 mg/kg (α-R deltamethrin), 0.015 mg/kg (cis-deltamethrin) and 0.019 mg/kg (trans-deltamethrin).

In validation study reported by Crofts, 2000 [Ref: B002857] and Beriault & Belyk (2001) [Ref: B003360], mean recovery rates in rape seed samples spiked with 0.012–1.5 mg/kg of α-R, cis- and trans-deltamethrin isomers ranged from 80-108% (RSD ≤ 19%).

Table 2 Deltamethrin analytical validation recovery rates for Method XM-10 in rape seed

Analyte	Fortification (mg/kg)	Recovery (range) (%)	Average recovery (%)	No	Mean recovery (%)	RSD (%)	Reference
Deltamethrin	0.015	91	91	1			
	0.15	84, 89	86	2	89	3.9	
	1.5	91	91	1			
α-R deltamethrin	0.015	95	95	1			B002857
	0.15	59, 80	69	2	80	19	
	1.5	86	86	1			
trans-deltamethrin	0.015	94	94	1			
	0.15	83, 92	87	2	89	5.3	
	1.5	89	89	1			
Deltamethrin	0.015	98-100	99	3			B003360
	0.15	101-107	103	3	101	3.1	
α-R deltamethrin	0.012	101-103	102	3			
	0.12	101-104	103	3	102	1.2	
trans-deltamethrin	0.019	105-107	106	3			
	0.19	106-113	109	3	108	2.7	

BP/01/88

In this method (referenced in the 2002 JMPR evaluation for use in apples, plums, tomatoes and rape seed/meal/oil), residues of α-R-deltamethrin, cis-deltamethrin and trans–deltamethrin were extracted by blending with acetone:hexane (50:50). The extracts were filtered, diluted with de-ionised water, partitioned and dried before reconstitution in hexane:dichloromethane (50:50) and gel permeation chromatography clean-up. After evaporation and reconstitution in toluene, residues were analysed by GC using an ECD. LOQs for α-R deltamethrin, cis-deltamethrin and trans-deltamethrin were 0.02 mg/kg in rape seed and meal and 0.05 mg/kg in oil.

Analytical (concurrent) recoveries in the supervised field residues averaged 82–101% in samples spiked with 0.02-0.05 mg/kg of α-R, cis- and trans-deltamethrin isomers.

Table 3 Deltamethrin analytical concurrent recovery rates for Method BP/01/88 in rape seed, meal and oil

Matrix	Fortification level	%Recovery (mean)			Reference
	(mg/kg)	Deltamethrin	*trans*-isomer	α-*R*-isomer	
Seed	0.02 (n = 1)	71	77	63	B002998
	0.05 (n = 6)	73-107 (91)	58-99 (77)	61-107 (83)	
	0.5 (n = 1)	86	99	84	
Seed	0.02 (n = 2)	80, 76	75, 80	77, 68	B002972
Meal	0.02 (n = 1)	112	104	114	

Matrix	Fortification level	%Recovery (mean)			Reference
	(mg/kg)	Deltamethrin	*trans*-isomer	α-*R*-isomer	
Seed	0.02 (n = 1)	71	77	63	B002998
	0.05 (n = 6)	73-107 (91)	58-99 (77)	61-107 (83)	
	0.5 (n = 1)	86	99	84	
Oil	0.05 (n = 1)	88	83	71	

NA = Not Applicable

Enforcement methods

Multi-residue method DGM F01/97-0

The 2002 JMPR reviewed the multi-residue method DGM F01/97-0 for measuring residues of deltamethrin and reported validation data for rape seed, showing mean recovery rates of 110–112% in samples spiked with 0.02 mg/kg and 0.2 mg/kg deltamethrin (LOQ of 0.02 mg/kg).

Stability of residues in stored analytical samples

Plant matrices

The 2002 JMPR reviewed information on the stability of residues of deltamethrin and its *trans*- and α-*R*- isomers in various substrates, including cotton seed products, grain and soya bean seed. In these matrices, residues were stable for at least 9 months (13–38 months in cotton seed products, with no significant isomerisation occurring during frozen storage.

USE PATTERNS

Information on GAP in 37 countries in Europe, North America, Asia and the Pacific was provided to the Meeting on the use of deltamethrin on oilseed rape. The following table summarizes the representative critical national or regional GAPs for this use.

Table 4 Representative authorised uses of deltamethrin on oilseed rape

Country	Form	Application				Max/season		PHI (days)	Remarks:
		method	max rate (g ai/ha)	water L/ha	RTI (days)	no	g ai/ha		
Australia	EC	foliar	5.5 - 13.75					7	
Austria	EC	foliar	6.25- 7.5	200-400	14	2		45	
Belarus	OD	foliar	7.5	200-400		2		30	
	WG	foliar	7.5	200-400	15	1		20	
	EC	foliar	7.5-10	200-400	15	2		30	
Belgium	EC	foliar	5			2		GS	Apply at BBCH 10-13 and BBCH 50-75
Bosnia, Croatia Herzegovina	EC	foliar	7.5		14	4		45	
	OD	foliar	5.0 -7.0	200-600		2			At start of infestation
Bulgaria	OD	foliar	6.25 5.0	200-500 200-250	14 14	2 2		 45	Up to BBCH 19 Up to BBCH 79
Canada	EC	foliar	5.0-10.0	100 11-22 (air)				7	Max 7.5 g ai/ha, 14d PHI for eastern Canada
China	EC	foliar	5.6-9.4	600-900		3			Use is for early season aphid control
Czech Republic	OD	foliar	5.0 -7.5	200-600		2		45	Up to BBCH 79
	EW	foliar	5.0 -7.5	200-600		1		GS	Up to BBCH 69
Estonia	EW	foliar	6.2 - 7.5	200-400		2		20	
	OD	foliar	6.0 - 7.5	200-300		2		45	

Country	Form	Application				Max/season		PHI (days)	Remarks:
		method	max rate (g ai/ha)	water L/ha	RTI (days)	no	g ai/ha		
Finland	EW	foliar	2.5-7.5	100-400	14	2		45	Up to late bud stage
France	EC, EW	foliar	5			4		45	
	OD	foliar	5-6.25		14	2		45	
Germany	EC	foliar	5.0 7.5 5.0	200-400 200-400 200-400		1 1 1		 90 90	From BBCH 12-29 From BBCH 11-69 From BBCH 55-69
Hungary	EW	foliar	7.5 7.5	250-400 250-400	 10	1 3		 45	Autumn Spring (Up to BBCH 75)
Ireland	EC	foliar	6.25-7.5	200		4		45	Up to BBCH 69
Italy	EC, EW	foliar	7.5		14	4		45	
Latvia, Lithuania	EC	foliar	6.0 - 7.5	200-400		2		20	
	OD	foliar	6.0 - 7.5	200-300		2		45	
Luxembourg	EC	foliar	5.0 5.0			1 1		45	From BBCH 10-13 From BBCH 50 Up to BBCH 75
Macedonia	EC	foliar	7.5		14	4		45	
Malta	EC, EW	foliar	7.5	600-1000	14	4		45	
Moldova	EC	foliar	12.5	200-300 50 (air)		2		30	
	WG	foliar	12.5			1		45	
Netherlands	EC	foliar	5			3		45	
Norway	EW	foliar	5-7.5	200-400		1		45	
Poland	EC, EW	foliar	5-7.5	300	14-21	1 2		 45	Autumn Spring
	OD	foliar	5-6	200-300	5-7	2		45	
Romania	EW	foliar	7.5	200-400	14	3		45	
	OD	foliar	3.5 - 6	200-400 50-100 (air)	14	2		GS	Up to BBCH 51
Russian Federation	WG	foliar	7.5	200-400		2		44	During vegetative period
Serbia, Montenegro	EC	foliar	5.0 - 7.5	200-400 50-100 (air)		2		42	Up to BBCH 59
Slovakia	EW	foliar	5.0 - 7.5	200 - 600	7	2		45	Up to BBCH 69
	OD	foliar	5.0 - 7.0	200 - 600		1		GS	Up to BBCH 19
Slovenia	EC	foliar	7.5	300-400				45	
Spain	EC	foliar	12.5		14	3		30	
	EC	foliar	7.5			3		45	
Switzerland	EW	foliar	7.5			1		42	Up to BBCH 59
Ukraine	WG	foliar	7.5-17.5	150-200		2		30	
	EC	foliar	6.25-12.5	200-300 50 (air)		2		30	
United Kingdom	EC	foliar (winter crops)	7.5	200 min	14	4	30	45	Up to BBCH 69
	EC	foliar (spring crops)	7.5	200 min		3	22.5	45	Up to BBCH 69
United States	EC	foliar	10	47 (min) 19 (air)	7		20	7	

EC formulations containing 15 g ai/L, 25 g ai/L, 27.5 g ai/L, 50 g ai/L or 100 g ai/L deltamethrin

OD formulations containing 10 g ai/L deltamethrin co-formulated with 10 g ai/L thiacloprid

WG formulations containing 250 g ai/kg deltamethrin

EW formulations containing 15 g ai/L, 25 g ai/L or 50 g ai/L deltamethrin
GS PHI is specified in terms of a growth stage (see Remarks column)

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information from USA and Canada on supervised field trials involving foliar treatments of deltamethrin on canola (oilseed rape).

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as "c=nn mg/kg" in the Reference and Comments columns. Residue data are recorded unadjusted for recovery.

Results from replicated field plots are presented as individual values, with levels of deltamethrin and its α-R- and trans– isomers reported separately. Individual isomer residues <LOQ have been taken as the LOQ value for the purposes of calculating the total residue.

In the trials, where multiple analyses are conducted on a single sample, the average value is reported and where duplicate samples have been analysed, both the individual results have been reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot, and the highest value has been used in calculations of MRLs and STMRs.

When residues were not detected they are shown as ND but for estimation, LOQ values have been used. Residues and application rates have been reported as provided in the study reports, although the results from trials used for the estimation of maximum residue levels (underlined) have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal.

When multiple applications were made to a crop, the treatment regimes were not always identical from one application to the next. In most trials, the actual treatment rates were within 10% of the listed 'target' application rates, but if not, the actual treatment rates are listed.

Oilseeds

Rape seed

Results from supervised trials from USA (1999) and Canada (1999, 2000) on canola (rape seed) were provided to the Meeting. In these trials, 80-336 square metre plots were treated with 1 or 2 foliar applications (6-8 days apart) of deltamethrin (EC formulations) and in the Canadian trials, additional plots were treated with 2 foliar applications of an SC formulation. In all trials, the last applications were made about 7-14 days before crop maturity. In the USA trials, applications were made by tractor-mounted or backpack sprayers with 4-8 nozzle booms to apply about 100 litres spray mix/ha.

Duplicate seed samples (min 0.5 kg) were harvested by hand or combine harvester, cleaned and frozen within 4 hours of sampling and stored frozen for up to 4 months (Canadian trials) or 12 months (USA trials) before analysis for cis-deltamethrin, α-R-deltamethrin, and trans-deltamethrin using method XM-10 (Canadian trials) or method BP/01/88 (USA trials).

Table 5 Residues in rape seed from supervised trials on canola in North America involving 1-2 foliar applications of deltamethrin (EC or SC formulations).

RAPE SEED Country, year Location (Variety)	Application no	Application g ai/ha	DAT	Deltamethrin residues (mg/kg) *Cis*-isomer	α-*R*-isomer	*Trans*-isomer	Total	Reference & Comments
USA, Canada	2	10	7					
USA, 1999 Pikeville NC (InVigor 2373 LL)	1	10	7	< 0.02 (2)	< 0.02 (2)	< 0.02 (2)	< 0.06 (2)	B002998 R02-01-C (EC)
USA, 1999 Pikeville NC (InVigor 2373 LL)	2	10	4 7 14 17 21	< 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.02 (2)	< 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.02 (2	< 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.02 (2) < 0.02 (2	< 0.06 (2) < 0.06 (2) < 0.06 (2) < 0.06 (2) < 0.06 (2)	B002998 R02-01-B (EC)
USA, 1999 Northwood ND (Arrow)	1	10	7	< 0.02 (2)	< 0.02 (2)	< 0.02 (2)	< 0.06 (2)	B002998 R05-01-C (EC)
USA, 1999 Northwood ND (Arrow)	2	10	7 14	0.04 (2) < 0.02	< 0.02 (2) < 0.02 (2)	< 0.02 (2) < 0.02 (2)	0.08 (2) < 0.06 (2)	B002998 R05-01-B (EC)
USA, 1999 New Rockford ND (Interstate RR Quest)	1	10	7	< 0.02 (2)	< 0.02 (2)	< 0.02 (2)	< 0.06 (2)	B002998 R07-01-C (EC)
USA, 1999 New Rockford ND (Interstate RR Quest)	2	10	7 14	0.02, < 0.02 < 0.02 (2)	< 0.02 (2) < 0.02 (2)	< 0.02 (2) < 0.02 (2)	0.06, < 0.06 < 0.06 (2)	B002998 R07-01-B (EC)
USA, 1999 Grangeville ID (Eagle)	1	10	7	< 0.02 (2)	< 0.02 (2)	< 0.02 (2)	< 0.06 (2)	B002998 R11-01-C (EC)
USA, 1999 Grangeville ID (Eagle)	2	10	7 14	< 0.02 (2) < 0.02 (2)	< 0.02 (2) < 0.02 (2)	< 0.02 (2) < 0.02 (2)	< 0.06 (2) < 0.06 (2)	B002998 R07-01-B (EC)
USA, 1999 Ashton, ID (Chinook)	1	10	7	< 0.02 (2)	< 0.02 (2)	< 0.02 (2)	< 0.06 (2)	B002998 R11-02-C (EC)
USA, 1999 Ashton, ID (Chinook)	2	10	7 14	< 0.02 (2) < 0.02, 0.02	< 0.02 (2) < 0.02 (2)	< 0.02 (2) < 0.02 (2)	< 0.06 (2) < 0.06, 0.06	B002998 R11-02-B (EC)
USA, 1999 Dayton, ID (Springfield)	1	10	7	0.02, 0.06 [a]	< 0.02, 0.026[b]	< 0.02, 0.028[c]	0.06, 0.12	B002998 R11-03-C (EC)
USA, 1999 Dayton, ID (Springfield)	2	10	7 14	0.04 (2) 0.04 (2)	< 0.02 (2) 0.02, 0.03	< 0.02 (2) < 0.02 (2)	0.08 (2) 0.08, 0.09	B002998 R11-03-B (EC)
Canada, 1999 Yorkton, SK (Independence)	1	10	7	< 0.015 (2)	ND (2)	< 0.015 (2)	< 0.045 (2)	B002857 Site 1-T1 (EC)
Canada, 1999 Yorkton, SK (Independence)	2	10	7 15	< 0.015 (2) < 0.015 (2)	ND (2) ND (2)	< 0.015 (2) ND (2)	< 0.045 (2) < 0.045 (2)	B002857 Site 1-T2 (EC)
Canada, 1999 Yorkton, SK (Independence)	2	10	7 15	0.034, 0.036 0.035, 0.039	ND (2) ND (2)	ND (2) ND, < 0.015	0.064, 0.066 0.065, 0.069	B002857 Site 1-T2 (FL)
Canada, 1999 Balcarres, SK (Exceed)	1	10	6	ND (2)	ND (2)	ND (2)	< 0.045 (2)	B002857 Site 2-T1 (EC)
Canada, 1999 Balcarres, SK (Exceed)	2	10	6 13	< 0.015 (2) < 0.015 (2)	ND (2) ND (2)	ND (2) ND (2))	< 0.045 (2) < 0.045 (2)	B002857 Site 2-T2 (EC)
Canada, 1999 Balcarres, SK (Exceed)	2	10	6 13	0.025, 0.021 0.015, 0.018	ND (2) ND (2)	ND (2) ND (2)	0.055, 0.051 0.045, 0.048	B002857 Site 2-T2 (FL)

RAPE SEED Country, year Location (Variety)	Application		DAT	Deltamethrin residues (mg/kg)				Reference & Comments
	no	g ai/ha		*Cis*-isomer	α-*R*-isomer	*Trans*-isomer	Total	
Canada, 1999 Fort Qu'Appelle, SK (Quest)	1	10	6	< 0.015 (2)	ND (2)	< 0.015 (2)	< 0.045 (2)	B002857 Site 3-T1 (EC)
Canada, 1999 Fort Qu'Appelle, SK (Quest)	2	10	6 14	< 0.015 (2) < 0.015 (2)	ND (2) ND (2)	< 0.015 (2) ND (2)	< 0.045 (2) < 0.045 (2)	B002857 Site 3-T2 (EC)
Canada, 1999 Fort Qu'Appelle, SK (Quest)	2	10	6 14	0.029, 0.031 0.017, 0.024	ND (2) ND (2)	ND (2) ND (2)	0.06, 0.062 0.048, 0.055	B002857 Site 3-T2 (FL)
Canada, 1999 Dilke, SK (Quest)	1	10	6	< 0.015 (2)	ND (2)	ND, < 0.015	< 0.045 (2)	B002857 Site 4-T1 (EC)
Canada, 1999 Dilke, SK (Quest)	2	10	6 13	< 0.015 (2) < 0.015 (2)	ND (2) ND (2)	ND (2) ND (2)	< 0.045 (2) < 0.045 (2)	B002857 Site 4-T2 (EC)
Canada, 1999 Dilke, SK (Quest)	2	10	6 13	0.025, 0.025 0.017, 0.021	ND (2) ND (2)	ND (2) ND (2)	0.056 (2) 0.048, 0.052	B002857 Site 4-T2 (FL)
Canada, 1999 Fort Whyte, MB (Hyola 401)	1	10	7	ND, < 0.015	ND (2)	ND (2)	< 0.045 (2)	B002857 Site 5-T1 (EC)
Canada, 1999 Fort Whyte, MB (Hyola 401)	2	10	7 12	< 0.015 (2) < 0.015 (2)	ND (2) ND (2)	ND (2) ND (2)	< 0.045 (2) < 0.045 (2)	B002857 Site 5-T2 (EC)
Canada, 1999 Fort Whyte, MB (Hyola 401)	2	10	7 12	0.031, 0.032 0.018, 0.019	ND (2) ND (2)	ND (2) ND (2)	0.062, 0.063 0.049, 0.05	B002857 Site 5-T2 (FL)
Canada, 2000 Balgonie, SK (Invigor 2573	1	10	8	< 0.015 (2)	< 0.012 (2)	< 0.019 (2)	< 0.045 (2)	B003360 Site 1-T1 (EC)
Canada, 2000 Balgonie, SK (Invigor 2573	2	10	8 13	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 1-T2 (EC)
Canada, 2000 Balgonie, SK (Invigor 2573)	2	10	8 13	< 0.015 (2) < 0.015, 0.02	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045(2) < 0.045, 0.051	B003360 Site 1-T2 (FL)
Canada, 2000 Balcarres SK (Invigor 2573)	1	10	10	< 0.015 (2)	< 0.012 (2)	< 0.019 (2)	< 0.045 (2)	B003360 Site 2-T1 (EC)
Canada, 2000 Balcarres SK (Invigor 2573)	2	10	10 14	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 2-T2 (EC)
Canada, 2000 Balcarres SK (Invigor 2573)	2	10	10 14	0.026, 0.03 0.016, 0.022	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	0.057, 0.061 0.047, 0.053	B003360 Site 2-T2 (FL)
Canada, 2000 Indian Head, SK (Liberty Link))	1	10	6	< 0.015 (2)	< 0.012 (2)	< 0.019 (2)	< 0.045 (2)	B003360 Site 3-T1 (EC)
Canada, 2000 Indian Head, SK (Liberty Link))	2	10	6 12	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 3-T2 (EC)
Canada, 2000 Indian Head, SK (Liberty Link)	2	10	6 12	0.019, 0.021 < 0.015, 0.016	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	0.05, 0.052 < 0.045, 0.046	B003360 Site 3-T2 (FL)
Canada, 2000 Fort Qu'Appelle, SK (Quest)	1	10	6	< 0.015 (2)	< 0.012 (2)	< 0.019 (2)	< 0.045 (2)	B003360 Site 4-T1 (EC)
Canada, 2000 Fort Qu'Appelle, SK (Quest)	2	10	6 12	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 4-T2 (EC)

RAPE SEED Country, year Location (Variety)	Application no	Application g ai/ha	DAT	Deltamethrin residues (mg/kg) *Cis*-isomer	Deltamethrin residues (mg/kg) α-*R*-isomer	Deltamethrin residues (mg/kg) *Trans*-isomer	Deltamethrin residues (mg/kg) Total	Reference & Comments
Canada, 2000 Fort Qu'Appelle, SK (Quest)	2	10	6 12	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 4-T2 (FL)
Canada, 2000 Minto, MB (Canterra 1867)	1	10	7	< 0.015 (2)	< 0.012 (2)	< 0.019 (2)	< 0.045 (2)	B003360 Site 5-T1 (EC)
Canada, 2000 Minto, MB (Canterra 1867)	2	10	7 13	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 5-T2 (EC)
Canada, 2000 Minto, MB (Canterra 1867)	2	10	7 13	0.04, 0.043 < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	0.071, 0.074 < 0.045 (2)	B003360 Site 5-T2 (FL)
Canada, 2000 Boissevain, MB (Quest)	1	10	7	< 0.015 (2)	< 0.012 (2)	< 0.019 (2)	< 0.045 (2)	B003360 Site 6-T1 (EC)
Canada, 2000 Boissevain, MB (Quest)	2	10	7 12	< 0.015 (2) < 0.015 (2)	< 0.012 (2) < 0.012 (2	< 0.019 (2) < 0.019 (2)	< 0.045 (2) < 0.045 (2)	B003360 Site 6-T2 (EC)
Canada, 2000 Boissevain, MB (Quest)	2	10	7 12	0.043, 0.044 0.031, 0.039	< 0.012 (2) < 0.012 (2)	< 0.019 (2) < 0.019 (2)	0.074, 0.075 0.062, 0.07	B003360 Site 6-T2 (FL)

[a] Analysed 3 times (0.081 mg/kg, 0.058 mg/kg, 0.048 mg/kg). Reported value is mean of all three results

[b] Analysed 3 times (0.028 mg/kg, 0.024 mg/kg, < 0.02 mg/kg). Reported value is mean of first two results

[c] Analysed 3 times (0.028 mg/kg, < 0.02 mg/kg, < 0.02 mg/kg). Reported value is highest value

FATE OF RESIDUES IN STORAGE AND PROCESSING

Processing

Rape seed

A processing study with rape seed was reviewed by the 2002 JMPR. In this study, deltamethrin was applied to oilseed rape as two foliar sprays at 30 g ai/ha at Indian Head, Saskatchewan, Canada with harvest 7 days after the second application [Ref: B002972]. For processing, the seeds were cleaned using aspiration to remove fine particles followed by sieving in a two-screen cleaner to separate small and large foreign particles. The whole seed was flaked using a flaking roll (setting 4–5 mm) and the flakes heated to 82–99 °C and pressed to liberate the crude oil. Additional oil was extracted from the presscake using hexane. The miscella (crude oil and hexane) was separated into hexane and crude oil and the residual hexane removed by heating at 72–90 °C. The crude oil was treated with phosphoric acid prior to refining. The longest interval between sampling and analysis, during which the samples were stored frozen, was 266 days. Residues of deltamethrin and its isomers were determined using method BP/01/98. Recoveries of deltamethrin and isomers in the various fractions are tabulated below.

Table 6 Procedural recoveries from the determination of deltamethrin, trans- and α-*R*-deltamethrin in rape seed and its processed commodities [JMPR 2002 Evaluations: Deltamethrin – Table 120, p 312]

Matrix	Fortification level	Recovery (%) Deltamethrin	Recovery (%) *trans*-isomer	Recovery (%) α-*R*-isomer
Seed	0.02	80, 76	75, 80	77, 68
Meal	0.02	112	104	114
Oil	0.05	88	83	71

Table 7 Residues of deltamethrin in rape seed and its processed commodities [JMPR 2002 Evaluations: Deltamethrin – Table 121, p 312]

Matrix	Residue (mg/kg)		
	Deltamethrin	*trans*-isomer	α-*R*-isomer
Seed (field)	0.025, 0.028, 0.025	< 0.02 (3)	< 0.02 (3)
Seed (processing)	0.027, 0.031, 0.025	< 0.02 (3)	< 0.02 (3)
Meal	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)
Refined oil	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)

The 2002 JMPR concluded that lack of significant residues precludes the estimation of processing factors for meal and refined oil except to note that they are less than 1.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Deltamethrin feeding studies in dairy cows, pigs, laying hens and chickens have been reviewed and reported by the 2002 JMPR [JMPR 2002 Evaluations: Deltamethrin – pp 312-315]. Conclusions reached by the 2002 Meeting are presented below.

When dairy cows were dosed with deltamethrin for 28 days at the equivalent of 2 and 10 ppm in the diet, residues in milk reached a plateau by day 4. Deltamethrin residues in the fat were higher than in other tissues. Transfer factors (residue level in tissue ÷ residue level in feed) for each tissue and milk for the two dosing levels (2 and 10 ppm respectively, single animals) were: fat, 0.023, 0.027; muscle, < 0.015, < 0.003; kidney, residues not reported due to analytical problems; liver, < 0.015, < 0.003; milk 28 days, 0.008, 0.0035.

When lactating dairy cows were administered a 1:1 mixture of deltamethrin and tralomethrin for 28 days at the equivalent of 2, 6 and 20 ppm in the diet, tralomethrin is rapidly converted to deltamethrin and the JMPR 2002 concluded that the study could be used to provide information on likely residues on exposure to deltamethrin at 2, 6 and 20 ppm in the feed. Residues in the fat were higher than in other tissues. Transfer factors (residue level in tissue ÷ residue level in feed) for each tissue and milk for the three dosing levels (2, 6 and 20 ppm respectively) were: fat, 0.006, 0.003, 0.001, mean 0.003; muscle, < 0.005, < 0.002, < 0.0005, mean < 0.0025; kidney, < 0.005, < 0.002, < 0.0005, mean < 0.0025; liver, < 0.005, < 0.002, < 0.0005, mean < 0.0025; milk 28 days, < 0.005, < 0.002, < 0.0005, mean < 0.0025, milk fat 28 days, 0.02, 0.005, 0.001, mean 0.009.

When pigs were fed deltamethrin in the diet for 130–141 days at 0.67 ppm, residues in the fat were higher than in other tissues. Transfer factors (residue level in tissue ÷ residue level in feed) for each tissue (fat, muscle, liver and kidney) were all < 0.04.

When laying hens and chickens were fed deltamethrin in the diet for up to 70 days in the case of chickens and for 20 weeks in the case of laying hens, residues were below the LOQ of the analytical methods for tissues and eggs.

When laying hens were dosed with a 1:1 mixture of deltamethrin and tralomethrin for 28 days at the equivalent of 2, 6 and 20 ppm in the diet, residues were below the LOQ of the analytical methods in the 2 ppm dose group. Residues in fat were substantially higher than residues in other tissues and eggs. Residue levels in muscle and liver were below the LOQ of the analytical methods for all the dosing groups. Transfer factors based on highest residues for fat were < 0.05, 0.04 and 0.03 respectively for the 2, 6 and 20 ppm feeding levels (< 0.05, 0.02, 0.02 if means are used). Transfer factors (based on highest and mean residue) for muscle and liver were < 0.01, < 0.003 and < 0.001 respectively for the 2, 6 and 20 ppm feeding levels. Residues in eggs reached a plateau by day 10 in the highest dose group. Residues in eggs were generally below the LOQ (0.01 mg/kg) for the other dose groups. The transfer factors (based on highest and mean residue) for eggs were < 0.0075, < 0.003 (at 7 days) and 0.002 (at 21 days) respectively for the 2, 6 and 20 ppm feeding levels.

APPRAISAL

Deltamethrin, a non-systemic synthetic pyrethroid insecticide was reviewed by JMPR several times between 1980 and 1992 and full periodic reviews were conducted for toxicology in 2000 and for residues in 2002. Residues from the veterinary uses of deltamethrin were evaluated by JECFA in 1999 and 2003.

The 2000 JMPR established an ADI of 0–0.01 mg/kg bw/day and an acute RfD of 0.05 mg/kg bw for deltamethrin and the residue definition established by the 2002 JMPR for plant and animal commodities, for both compliance with MRLs and for dietary intake assessment is the *sum of deltamethrin and its α-R- and trans– isomers*. The 2002 JMPR also concluded that the residue is fat soluble but that residues in milk should be measured on whole milk.

Specifications for deltamethrin technical material and relevant formulations have been established by the JMPS, most recently in January 2015, and published on the AGP-FAO Specifications webpage.

Deltamethrin was scheduled by the 47th Session of the CCPR for the evaluation of additional uses by the 2016 JMPR. The meeting received new GAP information and residue data on rape seed (canola).

Methods of analysis

The Meeting received information on the analytical methods (XM-10 and BP/01/88) used for the determination of deltamethrin residues in rape seed. These methods are similar to the those reviewed by the 2002 JMPR, involving hexane or hexane/acetone extraction, clean-up of reduced extracts by gel permeation chromatography (GPC) or GPC plus alumina column and residue determination by gas chromatography with electron capture detection (ECD). The LOQs of these methods in rape seed, oil and meal ranged from 0.01 to 0.05 mg/kg for deltamethrin, α-R-deltamethrin and trans-deltamethrin.

Stability of pesticide residues in stored analytical samples

In 2002, JMPR concluded that deltamethrin and its *trans*- and α-*R*- isomers are stable in various stored frozen substrates including grain and soya bean seed for at least 9 months, and 13-38 months in cotton seed, with no significant isomerisation occurring during frozen storage. Based on this information, the Meeting concluded that deltamethrin was stable in rape seed samples stored for the periods associated with the supervised field trials (up to 12 months).

Results of supervised residue trials on crops

The Meeting received information from USA and Canada on supervised field trials involving foliar treatments of deltamethrin to oilseed rape. The Meeting also noted that trials on rape conducted in Europe had been reviewed by the 2002 JMPR.

For estimating maximum residue levels and calculating STMRs and HRs, mean residue values have been used where duplicate samples have been analysed, LOQ values have been used when residues were not detected and the highest values have been used from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules.

Oilseeds

Rape seed

The critical GAPs for oilseed rape (canola) are in Australia (13.75 g ai/ha, PHI 7 days) and in Canada (10 g ai/ha, PHI 7 days).

In 16 independent field trials on canola conducted in North America, matching the GAP in Canada, deltamethrin residues in rape seed were: < 0.05 (4), < 0.06 (3), < 0.07 (3), < 0.09 (4), <0.11 and 0.14 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.07 mg/kg for deltamethrin in rape seed.

Fate of residues in processing

The Meeting noted that the 2002 JMPR had reviewed a processing study for rape seed and had concluded that the lack of significant residues the processed fractions precluded the estimation of processing factors for rape seed meal and refined oil, except to note they are less than 1.

Residues in animal commodities

Farm animal dietary burden

The Meeting recalculated the livestock dietary burden using the more recent diets listed in the FAO Manual Appendix IX (OECD Feedstuff Table) for the uses considered by the 2002 JMPR and a conservative estimated STMR-P of 0.07 mg/kg for rape seed meal.

	Animal dietary burden, deltamethrin, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	2.68[A]	2.68[C]	1.46	1.35	2.21	1.58	1.81	1.81
Dairy cattle	1.44	1.28	1.43	1.27	2.12[B]	1.61	1.62	1.62[D]
Poultry – broiler	1.72[E]	1.72[F]	1.16	1.16	1.15	1.15	0.9	0.9
Poultry – layer	1.72[G]	1.72[H]	1.28	1.17	1.15	1.15	1.35	1.36

[A] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

[B] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[C] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues

[D] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

[E] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues

[F] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues

[G] Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs

[H] Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs

The revised maximum dietary burdens are 2.7 ppm for beef cattle (2002 JMPR = 7.0 ppm) and 2.1 ppm for dairy cattle (2002 JMPR = 6.3 ppm). The mean dietary burdens are 2.7 ppm for beef cattle (2002 JMPR = 5.9 ppm) and 1.6 ppm for dairy cattle ((2002 JMPR = 5.8 ppm).

For poultry, the maximum and mean dietary burdens for broilers and layers are all 1.7 ppm (2002 JMPR = 2.7 ppm).

Maximum residue levels – animal commodities

Since the recalculated livestock dietary burdens for cattle and poultry do not exceed those estimated by the 2002 JMPR, the Meeting agreed that the current maximum residue levels for animal commodities accommodate the additional use on rape seed (canola).

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for dietary exposure assessments.

Definition of the residue (plant and animal commodities) for compliance with the MRL and for the estimation of dietary intake: *sum of deltamethrin and its α-R- and trans– isomers*

The residue is fat soluble.

	Commodity	MRL (mg/kg)		STMR	HR
CCN	Name	New	Prev		
SO 0495	Rape seed	0.2		0.07	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDIs) for deltamethrin were calculated for the food commodities for which STMRs or HRs have been estimated and for which consumption data were available. The results are shown in Annex 3.

The International Estimated Daily Intakes of deltamethrin for the 17 GEMS/Food cluster diets, based on estimated STMRs were 0–50% of the maximum ADI of 0.01 mg/kg bw (Annex 3). The Meeting concluded that the long-term dietary exposure to residues of deltamethrin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term dietary exposure

The International Estimated Short-term Intakes (IESTIs) for deltamethrin were calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available (Annex 4).

For deltamethrin the IESTI varied from 0–0% of the ARfD (0.05 mg/kg bw) and the Meeting concluded that the short-term dietary exposure to residues of deltamethrin, from uses considered by the Meeting, is unlikely to present a public health concern.

REFERENCES

Reference	Author(s)	Year	Title	Edition No
JMPR 2002	Anon	2002	Pesticide residues in food – 2002 Evaluations. Part I. Residues. Vol 1. Deltamethrin (135), pp 167-357. FAO Plant Production and Protection Paper 175/2, 2003. Published.	
B002857	Crofts, J.	2000	Determination of insecticide residues in canola following multiple applications of deltamethrin. Xenos Laboratories Inc., Ottawa, Canada. Bayer CropScience, Report No.: B002857 amended 2000-05-31. GLP/GEP: yes, unpublished	M-238595-02-1
B002972	Cole, M. G.; Netzband, D. J.	2000	Deltamethrin Derived Residues in Canola Seed and Processed Canola Commodities Following Two Applications of DECIS(R) 5 EC at Exaggerated Rates and the Shortest Proposed PHI, USA and Canada, 1999. Aventis CropScience USA LP, RTP, NC, USA. Bayer CropScience, Report No.: B002972, EPA MRID No.: 45222804. Date: 2000-09-07. GLP/GEP: yes, unpublished	M-238631-01-1
B002998	Cole, M. G.	2000	At Harvest Deltamethrin Derived Residues on Canola Following One or Two Applications of DECIS® 5EC at the Maximum Proposed Rate and the Shortest Proposed PHI, USA, 1998. Aventis CropScience USA LP, RTP, NC, USA. Bayer CropScience, Report No.: B002998. Report includes Trial Nos.:BP99R006. EPA MRID No.: 45222803. Date: 2000-09-07. GLP/GEP: yes, unpublished	M-238655-01-1
B003360	Beriault, J. N.; Belyk, M.	2001	Determination of Insecticide Residues in Canola Following Multiple Applications of Deltamethrin. Aventis CropScience Canada Co., Canada. Bayer CropScience, Report No.: B003360, Report includes Trial Nos.: 00AC08. Date: 2001-07-17. GLP/GEP: yes, unpublished	M-240400-01-1