PROPOXUR (075)

EXPLANATION

Propoxur was evaluated by the JMPR in 1973, 1977, 1981, 1983 and 1991. At the 1994 CCPR, several delegations expressed the opinion that the MRLs recommended by the 1991 JMPR for head lettuce and potatoes were based on very old data. The manufacturer stated that new data on potatoes would be available for the 1996 JMPR and that additional studies were scheduled for lettuce. New data from supervised trials on these commodities were provided to the Meeting.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Samples from the newly submitted supervised trials were analysed by HPLC with on-line reaction and fluorometric detection. The method determines both propoxur and its metabolite 2-hydroxy-propoxur (2-OH-propoxur). The homogenized sample is extracted with dichloromethane and the dichloromethane evaporated. The residual material is transferred to an Extrelut cartridge with 20 ml of saturated aqueous sodium chloride solution and 2 x 25 ml of dichloromethane. The cartridge is eluted with a further 50 ml of dichloromethane and the eluate evaporated to dryness. The dry residue is dissolved in 2 ml of methanol for analysis. A reverse-phase HPLC system is used with an acetonitrile/water gradient. The column eluate is passed successively through an on-line hydrolysis reactor to produce methylamine and a derivatization reactor to form a fluorophore with *o*-phthalaldehyde and 2-mercaptoethanol. Fluorometric detection is at 340 nm excitation and 455 nm emission.

The mean recoveries from potatoes were 86% for propoxur and 84% for 2-OH-propoxur at fortification levels of 0.02-0.2 mg/kg, and from lettuce 95% for propoxur and 93% for 2-OH-propoxur at 0.04-1.0 mg/kg. The limits of determination were 0.02 mg/kg for potatoes and 0.04 mg/kg for lettuce (Blass, 1990).

In the older trials samples were analysed by a colorimetric method (lettuce in the 1960s) or by GLC (lettuce and potatoes in the 1970s). The colorimetric method is based on measurement of the reaction product of *o*-isopropoxyphenol and aminoantipyrine at 490 nm. In the GLC method the compound determined is methyl *N*-methylcarbamate which is formed in the injection port by transesterification of propoxur with methanol.

USE PATTERN

Information on use patterns was provided by the governments of Germany, The Netherlands and Poland, and the manufacturer. The uses on potatoes and lettuce are given in Table 1.

Table 1. Registered uses of propoxur on lettuce and potatoes. All spray applications. The figures in parentheses should not be compared with the application rates in the supervised trials: they are included only for reference.

Crop	Country	Form.	Application			PHI, days
			kg ai/ha	kg ai/hl	No.	
Lettuce	Germany	SL	0.18-0.24	0.03-0.04	2	7
	Italy	WP	(0.20-0.50)	0.025-0.05	1	10
	Italy	WP	(0.2-0.3)	0.025-0.038	2	10
	Netherlands	WP	0.20-0.40	0.05	1	14/21[1]
Lettuce	Netherlands	WP	0.15-0.30	0.037	2	14/21[1]
(glasshouse)	Netherlands	EC	0.40-0.60		2	14/21[1]
Potatoes	Brazil	EC	0.48-0.60	(0.048-0.075)	7	14
	Germany	SL	0.24	0.04	1	14
	Germany	SL	0.18	0.03	2	14
	Italy	EC	(0.08-0.20)	0.01-0.02	2	10
	Italy	WP	(0.38-0.53)	0.047-0.066	1	10
	Italy	WP	(0.20-0.50)	0.025-0.063	2	10
	Kuwait	EC	0.30-0.75	(0.03-0.13	1	
	Kuwait	WP	0.50-0.75	(0.05-0.13)	1	
	Poland	WP	0.30-0.50	(0.075-0.30)	2	7
	Rumania	WP	0.50	(0.063-0.083)	4	
	Saudi Arabia	EC	(0.18-0.42)	0.03-0.07	2	
	Saudi Arabia	WP	(0.30-0.45)	0.05-0.075	1	
	Netherlands	WP	0.50		2	14
	United Arab Republic	EC	0.30-0.75	(0.03-0.13)	1	
	Uruguay	EC	(0.20-0.40)	0.05	4	14

[1] 14 days from March to September and 21 days from October to February

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data on residues in lettuce and potatoes were submitted to the Meeting by the manufacturer and the government of Poland (Tables 2 and 3).

Table 2. Residues of propoxur and its metabolite 2-OH-propoxur in potatoes.

Country Year	Application				PHI, days	Residues, mg/kg		Reference
	Form.	No	kg ai/ha	kg ai/hl		Propoxur	2-OH-propoxur	
Germany 1994 (6 locations)	SL	1	0.24	0.04	14 -15	≤0.02 (6)	<0.02 (6)	3
Poland 1994	EC	1	0.5		3 7	<0.02 ≤0.02		4

Country Year	Application				PHI, days	Residues, mg/kg		Reference
	Form.	No	kg ai/ha	kg ai/hl		Propoxur	2-OH-propoxur	
					14	<0.02		
Poland 1993	EC	2	0.24	0.04	23	<0.01		4

Table 3. Residues of propoxur and 2-OH-propoxur in lettuce under field conditions.

Country, Year	Application				PHI, days	Residues, mg/kg		Ref.
	Form.	No	kg ai/ha	kg ai/hl		Propoxur	2-OH-propoxur	
Lettuce	WP	2	0.24	0.04	0	2.9, 3.8, 5.1, 5.7	<0.04 (4)	2
Germany					3	0.34, 0.54, 0.67, 0.73	<0.04 (4)	
(4 locations)					4	0.32, 0.36, 0.52, 0.62	<0.04 (4)	
1991 (June)					7	0.05, 0.07, 0.10, 0.13	<0.04 (4)	
					14	<0.04 (4)	<0.04 (4)	

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The governments of The Netherlands and Poland reported monitoring data for propoxur on several crops. The result are given in Tables 4 and 5.

Table 4. Monitoring data on propoxur in crops in Poland, 1994.

Commodity	No. of samples analysed	No. of samples with detectable residues[1]	Detection frequency, %	Residues, mg/kg
Apples	121	0		
Cabbage, white	114	2	1.8	0.1, 0.3
Carrot	18	0		
Cauliflower	30	0		
Celery	23	0		
Cherry, sour	41	0		
Currants, black	58	0		
Currants, red, white	20	0		
Onion, Bulb	21	0		
Parsley	30	0		
Potatoes	88	0		
Tomato (glasshouse)	167	0		

[1] The LOD was not reported

Table 5. Monitoring data on propoxur in crops in The Netherlands, 1991-1994.

Commodity	No. of samples analysed	No. of samples with detectable residues[1]	Detection frequency, %	Mean residues,[2] mg/kg

Commodity	No. of samples analysed	No. of samples with detectable residues[1]	Detection frequency, %	Mean residues,[2] mg/kg
Apples	87	2	2.3	<0.05
Bananas	3	1	33.3	<0.05
Celery	29	2	6.9	<0.05
Cucumbers	644	2	0.3	<0.05
Currants	620	13	2.1	<0.05
Egg plant	8	1	12.5	<0.05
Endive	104	4	3.8	<0.05
Leek	10	4	40.0	<0.05
Lettuce	2845	9	0.3	<0.05
Plums	536	2	0.4	<0.05
Raspberries	267	4	1.5	0.10
Strawberries	3343	21	0.6	<0.05
Sweet peppers	1129	7	0.6	<0.05
Wheat	185	7	3.8	<0.05

[1] LOD = 0.05 mg/kg
[2] For samples with residues below the LOD, half of the LOD is taken for calculation of the mean residues

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Crop	MRL, mg/kg	Remarks
Australia	Potatoes	10	
Austria	Beet, sugar	1	
	Fruit	1	
	Hop	50	
	Other food of animal origin	0.05	
	Other plant commodities	0.5	
	Vegetables	1	
Belgium	All plant commodities	N.D.	<0.05 mg/kg
	Eggs	0.5	
	Fruit	3	
	Meat	0.05*	
	Milk	0.05*	
	Milk products	0.05*	
	Poultry meat	0.5	
	Vegetable	3	
Brazil (Propoxur and its metabolite expressed as propoxur)	Apple	3	
	Eggplant	3	
	Broccoli	3	
	Cabbage	3	

Country	Crop	MRL, mg/kg	Remarks
	Cabbage, white	3	
	Cacao	0.03	
	Cauliflower	3	
	Citrus fruit	3	
	Cotton seed	0.03	
	Cucurbits	3	
	Garlic	3	
	Grassland	5	
	Meat	0.05	
	Milk	0.05	
	Onion	3	
	Peach	3	
	Peanut	0.03	temporary
	Pepper, cayenne	3	
	Pepper, sweet	3	
	Plum	3	
	Potatoes	0.5	
	Poultry	0.03	temporary
	Soya	0.03	temporary
Chile	Apple	3	
Propoxur and its metabolites	Cereals	0.5	
express as propoxur	Cherry	3	
	Meat	N.D.	<0.05 mg/kg
	Milk	N.D.	<0.05 mg/kg
	Peach	3	
	Pear	3	
	Plum	3	
	Potatoes	0.5	
	Rice husked	0.1	
European Community	Fruit	3	
	Vegetables	3	
Finland	All plant commodities	3	
Germany	Fruit	3	
	Hop	50	
	Other plant commodities	0.05	
	Vegetables	3	
Greece	All plant commodities	3	
Italy	Fruit	3	
	Potatoes	3	
	Tobacco	3	
	Vegetables	3	
Japan	Cereals except rice	0.5	

Country	Crop	MRL, mg/kg	Remarks
	Fruit	1	
	Potatoes	0.5	
	Rice	1	
	Vegetables	2	
Kenya	Apple	3	temporary
	Blackberry	3	temporary
	Cherry	3	temporary
	Currant, red	3	temporary
	Gooseberry	3	temporary
	Legume animal feed, green	5	temporary
	Meat	0.05*	temporary
	Milk	0.05*	temporary
	Other vegetables	3	temporary
	Peach	3	temporary
	Pear	3	temporary
	Plum	3	temporary
	Rice husked	0.1	temporary
	Root vegetables	0.5	temporary
	Strawberry	3	temporary
Luxembourg	Fruit	3	
	Other plant commodities	0.05	
	Vegetables	3	
Malaysia	Apple	3	
	Cereals grain	0.5	
	Cherry	3	
	Rice Milled	0.1	
	Strawberry	3	
Netherlands	Eggs	0.5	
	Fruit	3	
	Meat	0.05*	
	Chicken meat	0.5	
	Milk	0.05*	
	Potatoes	0.5	
	Rice	0.1	
	Vegetables	3	
	Other food commodities	N.D.	<0.05 mg/kg
Poland	Strawberry	0.2	
	Fruit, other	3	
	Potatoes	0.1	
	Vegetables, other	3	
	Rape seed	0.5	
Portugal	Fruit	3	

Country	Crop	MRL, mg/kg	Remarks
	Potatoes	0.05	
	Vegetables	3	
South Africa	Grape	0.05	
	Grape	0.05	export tolerance
Spain	Cereals	0.05	
	Food dry	0.05	
	Forage crops straw	1	
	Fruit	3	
	Hop	0.05	
	Nuts	3	
	Oil plants seed	0.05	
	Potatoes	0.05	
	Pulses	0.05	
	Spices	0.05	
	Stimulant plants	0.05	
	Sugar plants	0.05	
	Tea	0.05	
	Tobacco	0.05	
	Vegetables	3	
Taiwan	Banana	1	
	Banana without peel	0.1	
	Papaya	1	
	Papaya without peel	0.1	
	Pineapple	1	
	Pineapple without peel	0.1	
	Rice	0.1	
Turkey	Apple	2	
	Milk	0.05	
	Pear	2	
Uruguay	Apple	3	
	Pear	3	

* at the lower limit of determination

APPRAISAL

Propoxur was evaluated by the JMPR in 1973, 1977, 1981, 1983 and 1991. At the 1994 CCPR, several delegations expressed the opinion that the MRLs recommended by the 1991 JMPR for head lettuce and potatoes were based on very old data. The manufacturer stated that new data on potatoes would be available for the 1996 JMPR and that additional studies were scheduled for lettuce. New data from supervised trials on these commodities were provided to the Meeting, together with information on GAP, analytical methods and monitoring surveys.

Analytical methods

Analyses in the new supervised trials were by HPLC with on-line derivatization and fluorometric detection. This method allows the determination of both propoxur and its metabolite 2-hydroxy-propoxur (2-OH-propoxur).

Recoveries from lettuce and potatoes were 86-95% for propoxur and 84-93% for 2-OH-propoxur and the limits of determination were 0.02 mg/kg in potatoes and 0.04 mg/kg in lettuce.

The method was considered suitable for use in supervised trials and for enforcement.

Field trials data

The Meeting evaluated newly submitted data from supervised trials on lettuce and potatoes and re-evaluated data reviewed by earlier Meetings in the light of current GAP. The Meeting agreed not to estimate STMRs for these commodities until a periodic review was undertaken, since CXLs exist for many commodities and metabolic studies were not submitted to the Meeting.

Lettuce. Six supervised trials in The Netherlands in 1963 and 1971 under glass were reported in the 1973 JMPR monograph. The residues were 0.9-20.2 mg/kg at 0-13 days and 0.5-0.8 mg/kg at 14-17 days from single applications of 0.6-0.9 kg ai/ha. The details of the trials were not clear and the treatments did not match current glasshouse GAP in The Netherlands (0.15-0.30 kg ai/ha for WP and 0.40-0.60 kg ai/ha for EC, PHI 14 days from March to September and 21 days from October to February, 2 applications).

Eight supervised field trials carried out in Germany during 1961-1964 were reported in the 1991 JMPR monograph. Samples were analysed by a very old colorimetric method and details of 4 trials (0.3-0.75 kg ai/ha, one application, 0-18 days PHI) were not clear. In the other 4 trials the conditions (0.15 or 0.6 kg ai/ha, one application, 0-8 days PHI) were not comparable with GAP in Germany (0.18-0.24 kg ai/ha, 0.03-0.04 kg ai/hl, 2 applications, 7 days PHI) or The Netherlands (0.20-0.40 kg ai/ha, one application, 14 or 21 days PHI). The trials data could not be used for evaluation.

In three field trials in 1975, reported in 1991, the conditions (0.24 kg ai/ha, 0.04 kg ai/hl, 3 applications, 0-9 days PHI) were comparable to German GAP except in the number of applications and the residues were 0.01, 0.2 and 0.3 mg/kg at 7 days PHI. The Meeting considered that the effect of the number of applications would not be significant because propoxur residues were observed to decrease rapidly during the first 7 days after application.

In 1991, supervised trials were carried out at four locations in Germany according to German GAP with the current HPLC analytical method. The residues of propoxur were 0.05, 0.07, 0.10 and 0.13 mg/kg, and of 2-hydroxy-propoxur <0.04 mg/kg in each trial.

The Meeting used the three 1975 and four 1991 German trials to estimate maximum residue levels. The residues were 0.01, 0.05, 0.07, 0.10, 0.13, 0.2 and 0.3 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg for propoxur in head lettuce to replace the existing CXL of 3 mg/kg.

Potatoes. Seven supervised trials in Germany in 1971-1975 were reported in the 1991 monograph. Although most of these trials were at higher dose rates or shorter PHIs than required by GAP in Germany and The Netherlands, all of the residues were below the limit of determination of 0.1 mg/kg.

In 1994, supervised trials were carried out at six locations in Germany according to German GAP (0.24 kg ai/ha, 1 application, 14 days PHI). Analyses by the new HPLC analytical method showed propoxur and 2-hydroxy-propoxur residues to be below the limit of determination (0.02 mg/kg).

The government of Poland provided data on three supervised trials to the Meeting. The trials were in 1993 and 1994 and according to GAP (0.3-0.5 kg ai/ha, 2 applications, 7 days PHI). The residues were below the limit of determination (0.01 or 0.02 mg/kg). The Meeting concluded that adequate data on propoxur residues in potatoes determined by a modern method of analysis with an LOD of 0.02 mg/kg were now available.

The Meeting estimated a maximum residue level of 0.02* mg/kg for propoxur in potatoes to replace the existing CXL (0.1* mg/kg).

Monitoring data

In monitoring in Poland for propoxur in 1994, 731 samples of apples, white cabbages, carrots, cauliflowers, celery, sour cherries, black, red and white currants, bulb onions, parsley, potatoes and tomato (glasshouse) were analysed. No residues were found in any samples except two of white cabbage at 0.1 and 0.3 mg/kg, but information on the LOD was not available. The detection frequency was 0.3% for all samples and 1.8% for white cabbage. The residues found in white cabbage were below the national MRL (vegetables: 3 mg/kg).

Comprehensive monitoring was carried out in The Netherlands from 1991 to 1994 on 9810 samples of apples, bananas, celery, cucumbers, currants, egg plant, endive, leeks, lettuce, plums, raspberries, strawberries, sweet peppers and wheat. The overall detection frequency was 0.81% and the detection frequency for individual commodities ranged from 0.3% for cucumbers and lettuce to 40% for leeks. The mean residues in all crops were below the national MRL (fruit and vegetables 3 mg/kg, other food commodities <0.05 mg/kg).

RECOMMENDATIONS

The Meeting estimated the maximum residue levels shown below, which are recommended for use as MRLs.

Definition of the residue for compliance with MRLs: propoxur

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New	Previous	
VL 0482	Lettuce, Head	0.5	3	7
VR 0589	Potato	0.02*	0.1*	7-14

REFERENCES

(all unpublished)

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