

**HEXYTHIAZOX (176)****EXPLANATION**

Hexythiazox was evaluated by the JMPR in 1991 and 1994. The 28th Session of the CCPR (ALINORM 97/24, 1996, para 75) was informed that data for hexythiazox on hops would become available for a future evaluation and hexythiazox was scheduled for a residue evaluation in 1998.

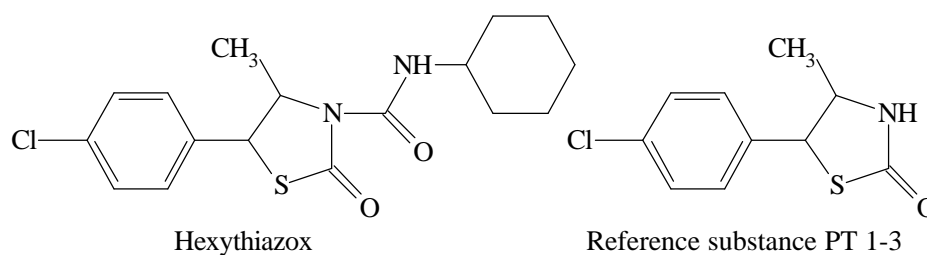
Information on methods of analysis, registered uses, supervised residue trials on hops, processing studies on hops and national MRLs was made available by two basic manufacturers. New information on the use pattern and national MRLs was provided by The Netherlands, Poland and Germany. Summary sheets for hexythiazox residue trials on apples and black currants were provided by Poland; the trials have already been evaluated by the 1994 JMPR.

**METHODS OF RESIDUE ANALYSIS**

Tilting (1994) described analytical method 343 used for residue analysis in the German supervised trials and processing trials on hops. The method was tested and validated for a range of substrates including soil, beer, brewing wastes, yeast, apple, strawberry, green hops and dry hops.

Residues were extracted from solid substrates with methanol. Beer was mixed with calcium hydroxide and acetone before filtration through Celite. The sample extract was diluted with sodium chloride solution and the residues were extracted into hexane and dichloromethane. Column clean-up was effected on a Florisil-charcoal column. The residue from the evaporated eluate was taken up in hexane + acetone (9+1) for GLC with an NPD where the injection port temperature was 300°C. Hexythiazox and metabolites containing the 5-(4-chlorophenyl)-4-methylthiazolidin-2-one moiety are converted to PT 1-3 in the injector.

Comparison with PT 1-3 standards showed that on injection into the hot injector port the mean conversion of hexythiazox to PT 1-3 was 99% (CV 6.6%).



Recoveries from spiked samples established that the method was acceptable with the following LODs: beer, brewing wastes and fruit 0.05 mg/kg, green hops 0.1 mg/kg, and dried hops 0.5 mg/kg.

The 1991 JMPR reported that hexythiazox is the main residue in crops and its metabolites are present in negligible amounts. The residues determined by this “total residue” method may therefore be accepted as equivalent to residues of hexythiazox, the current definition of the residue.

Tilting (1995) validated method 343/1 (method 343 with an added GPC clean-up) for dried hops (LOD 0.5 mg/kg). Hexythiazox metabolites PT 1-3, PT 1-8 (the 4-hydroxycyclohexyl) and PT 1-9 (4-oxocyclohexyl metabolite) were determined by the method with satisfactory recoveries at 2

mg/kg on dry hops. The structures of the metabolites are shown in Figure 1 of the 1991 JMPR Residue Evaluations (page 462).

Williams (1996), in examining the specificity of the hexythiazox method, tested the GLC behaviour in method 343/1 of 26 pesticides registered in the USA for use on hops and found that only one, triforine, had potential interference. As triforine is removed by the Florisil-charcoal column it would not interfere.

Fomenko (1997) tested the behaviour of the hexythiazox metabolites PT 1-2 (5-(4-chlorophenyl)-4-methyl-2-oxothiazolidine-3-carboxamide), PT 1-4 (3-hydroxycyclohexyl metabolite) and PT 1-8 through the US FDA multi-residue methods. The compounds were eluted through DB-1, DB-17 and DB-225 columns with a good response on an ECD, but not on other detectors. The compounds were not recoverable from the Florisil column clean-up required before GLC analysis with an ECD, so the metabolites cannot be determined by the current FDA multi-residue methods.

Mckinney and Tomkinson (1995) described the analytical method (ENC-8/98) used in the US hexythiazox trials on hops. Hops samples were extracted with methanol-water and an aliquot of the extract was evaporated before the addition of dilute hydrochloric acid and sodium chloride followed by partition of the residues into dichloromethane. The residue was hydrolysed in 0.1M NaOH at 60°C for 30 minutes. After further partitioning, the residue was further cleaned up on a small C-18 column and a Celite activated charcoal column for determination by HPLC. An LOD of 0.1 mg/kg was achieved for dry hops. The mean and range of recoveries in 10 tests on hops were 76% and 62-87% respectively.

The hops from the Japanese trials were analysed by method RD-8808 (Soeda *et al*, 1988). Hops were extracted with a methanol-water mixture and the filtered extract was diluted with water and sodium chloride before extraction with hexane. The hexane layer was washed with a mixture of sodium chloride and sodium hydroxide mixture and the residues were extracted from the hexane into acetonitrile. Clean-up was effected on a C-18 Sep-pak and a Florisil column. Residues were then determined by HPLC on a reversed phase system with UV detection at 225 nm. An LOD of 0.2 mg/kg was achieved.

### Stability of pesticide residues in stored analytical samples






The stability of hexythiazox residues in hops was tested by analysis of a sample stored for 3 days (15.0, 13.1 mg/kg) and 40 days (15.6, 11.4 mg/kg) at -30°C (Nippon Soda, 1994). The test provides some evidence of storage stability for 30-40 days.

The 1991 residue evaluation provides information on the stability of hexythiazox residues in various samples during frozen storage up to 6 months. No information was available on the stability of hexythiazox in hops during storage for 15 months.

Tilting (1998) tested the stability of hexythiazox residues in hops stored at -20°C and beer refrigerated at 4°C for 2 years. Residues were stable for the duration of the study in hops, but decreased by approximately half in beer (Table 1).

Table 1. Storage stability of hexythiazox in hops and beer (Tilting, 1998). Duplicate samples were analysed at each time and results are expressed as mg hexythiazox per kg sample.

Days	Green hops	Dried hops	Days	Spent hops	Beer
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Days	Green hops		Dried hops		Days	Spent hops		Beer	
0	4.5	4.3	5.5	5.0	0	3.6	3.1	0.27	0.28
13	4.1	3.8	4.0	4.1	14	4.4	5.0	0.45	0.48
31	3.9	4.1	5.4	8.1 	30	4.4	4.2	0.37	0.37
74	4.5	4.6	4.3	3.9	71	3.8	3.1	0.37	0.33
119-120	3.3	3.2	3.8	3.2	134	3.7	4.1	0.52	0.49
178	4.0	3.9	3.2	3.0	180	3.8	3.7	0.34	0.36
361	3.6	3.5	3.6	3.6	365	3.3	<0.05 	0.27	0.24
538	7.2 	5.5	<0.05 	<0.05 	538	4.5	4.9	0.43	0.32
741	4.9	4.7	4.3	5.0	755	5.2	5.6	0.19	0.21
					799			0.15	0.22

 aberrant result.

## USE PATTERN

Hexythiazox is an acaricide used on fruit, vegetables and hops. The Meeting was provided with additional information on registered uses (Table 2).

Table 2. Registered uses of hexythiazox.

Crop	Country	Form	Application				PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	Number	
Apples	Netherlands	WP	foliar spray	0.060	0.004	2	28
Apples	USA	WP	foliar spray	0.21		1	GS <u>3</u> /
Aubergines	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Beans	Netherlands	WP	foliar spray	0.004-0.016	0.002	1	3
Blackberries	Netherlands	WP	foliar spray	0.050-0.060	0.005	1	-
Cherries	Netherlands	WP	foliar spray	0.060	0.004	2	28
Courgette	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Courgette	Netherlands	WP	foliar spray	0.020-0.040	0.005	1	3
Cucumber	Germany <u>5</u> /	WP	foliar spray	0.03-0.06		2	3
Cucumber	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Cucumber	Poland	EC	high vol	0.01-0.06	0.003	g	3
Cucumber	Poland	EC WP	high vol	0.1-0.2	0.01		3
Currants	Poland	EC	high vol	0.04	0.004-0.005		14
Currants red black	Netherlands	WP	foliar spray	0.050-0.060	0.005	1	14
Eggplant	Poland	EC	high vol	0.01-0.06	0.003	g	3
Eggplant	Poland	EC WP	high vol	0.1-0.2	0.01		3
Filbert	Poland	EC WP	high vol	0.04-0.075	0.004-0.015		30
Fruit trees	Poland	EC WP	high vol	0.04-0.075	0.004-0.015		30
Gherkins	Netherlands	WP	foliar spray	0.020-0.040	0.005	1	3
Gherkins	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Gooseberries	Netherlands	WP	foliar spray	0.050-0.060	0.005	1	14
Grapes	Germany <u>5</u> /	WP	foliar spray		0.004	1	28
Hops	Germany <u>5</u> /	WP	foliar spray	<u>1</u> /	0.003	2	28
Hops	Germany <u>5</u> /	WP	foliar mist LV	<u>2</u> /	0.0045	2	28
Hops	Japan <u>5</u> /	WP	spray	0.20-0.30	0.0033-0.005	2	7
Hops	USA <u>6</u> /	WP	foliar spray	0.21-0.28		1	GS <u>4</u> /
Melons	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Okra	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Pears	Netherlands	WP	foliar spray	0.060	0.004	2	28
Pears	USA	WP	foliar spray	0.21-0.42		1	28
Peppers	Poland	EC	high vol	0.01-0.06	0.003	g	3
Peppers	Poland	EC WP	high vol	0.1-0.2	0.01		3
Peppers, chilli	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Peppers, sweet	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3

Crop	Country	Form	Application				PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	Number	
Plums	Germany <u>5</u> /	WP	foliar spray		0.004	1	28
Plums	Netherlands	WP	foliar spray	0.060	0.004	2	28
Pome fruits	Germany <u>5</u> /	WP	foliar spray		0.004	1	28
Raspberries	Netherlands	WP	foliar spray	0.050-0.060	0.005	1	14
Strawberry	Germany <u>5</u> /	WP	foliar spray		0.004	1	28
Strawberry	Netherlands	WP	foliar spray	0.025-0.050	0.005	1	3
Strawberry	Netherlands	WP	foliar spray	0.030-0.060	0.005	1 g	3
Strawberry	Poland	EC	high vol	0.04	0.004-0.005		14
Tomato	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3
Tomato	Poland	EC	high vol	0.01-0.06	0.003	g	3
Tomato	Poland	EC WP	high vol	0.1-0.2	0.01		3
Winter squash	Netherlands	WP	foliar spray	0.025-0.075	0.005	1 g	3

g: glasshouse use

1/ spray volume 1000-5000 l/ha. Max application rate 0.15 kg ai/ha.

2/ spray volume 700-3300 l/ha. Max application rate 0.15 kg ai/ha.

3/ growth stage - pink bud

4/ growth stage - before burr (cone formation)

5/ label provided

6/ proposed GAP

## RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised residue trials on hops were carried out in Germany, Japan and the USA. The results are shown in Table 3.

Where residues were not detected, results are recorded in the tables as below the limit of determination (LOD), e.g. <0.1 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOD, to 1 significant figure. Although all trials included control plots, no control results are recorded in the tables because residues in control samples did not exceed the LOD. Residues are recorded uncorrected for recovery. Recovery values were mainly in the range of 60-110%.

Hexythiazox was applied by mistblower to plots of approximately 390 m<sup>2</sup> in the hops trials in Germany in 1992. Samples of 0.5-1 kg were stored for about 470 days (1992 trials) and 180 days (1993 trials) before analysis. A mistblower was also used in the trials in 1993 where plot sizes were 27-36 m<sup>2</sup>. In trials D07/84/93, D07/83/93 and D07/82/93 light rainfall occurred within 45 minutes to 6½ hours after application, which may have reduced residue levels.

Hexythiazox was applied to hops in 50-146 m<sup>2</sup> plots by mist backpack or handgun sprayer in the US trials. Hop cones in the US trials were collected at normal maturity and were dried for 3.5 to 16 hours at 32-65°C, which is normal commercial practice. Samples (1.5 - 3 kg) were stored for 260 days before analysis.

In the Japanese trials the hop flowers were dried by hot air drier just after harvest at 50°C for 1 hour and then at 60°C for 7 hours.

Table 3. Hexythiazox residues in hops resulting from supervised trials in Germany, Japan and the USA. Double-underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Country, Year (Variety)	Application				PHI, days	Commodity	Hexythiazox, mg/kg	Ref				
	Form	kg ai/ha	kg ai/hl	no.								
Germany 1992 (Perle)	WP	0.16	0.0045	1	0	hop catkins	2.8	D07/85/92				
					21	hop catkins	0.95					
					28	hop catkins	0.48					
									35	hop catkins	0.14	
									28	kiln-dried hops	<u>0.93</u>	D07/85/92
									28	beer	<0.05	
									28	spent hops	0.15	
				28	spent hops (dry weight)	0.65						
Germany 1992 (Perle)	WP	0.16	0.0045	1	0	hop catkins	3.3	D07/86/92				
					21	hop catkins	1.6					
					28	hop catkins	0.96					
									35	hop catkins	0.35	
									28	kiln-dried hops	<u>1.5</u>	D07/86/92
									28	beer	<0.05	
									28	spent hops	0.18	
				28	spent hops (dry weight)	0.61						
Germany 1992 (Perle)	WP	0.16	0.0045	1	0	hop catkins	3.6	D07/87/92				
					21	hop catkins	0.93					
					28	hop catkins	0.53					
									35	hop catkins	0.47	
									28	kiln-dried hops	<u>1.3</u>	D07/87/92
									28	beer	<0.05	
									28	spent hops	0.18	
				28	spent hops (dry weight)	0.53						
Germany 1992 (Perle)	WP	0.16	0.0045	1	0	hop catkins	3.1	D07/88/92				
					21	hop catkins	1.1					
					28	hop catkins	0.23					
									35	hop catkins	0.34	
									28	kiln-dried hops	<u>0.88</u>	D07/88/92
									28	beer	<0.05	
									28	spent hops	0.11	
				28	spent hops (dry weight)	0.32						
Germany 1993 (Perle)	WP	0.22	0.0045	1	0	leaves and hop catkins	5.2	D07/82/93				
					21	hop catkins	0.11					
					27	hop catkins	0.18					
					35	hop catkins	<0.1					
					27	kiln-dried hops	<u>0.79</u>					
Germany 1993 (Perle)	WP	0.17	0.0045	1	0	leaves and hop catkins	5.8	D07/83/93				
					21	hop catkins	<0.1					
					27	hop catkins	0.12					
					35	hop catkins	0.12					
					27	kiln-dried hops	<u>0.71</u>					
Germany 1993 (Northern Brewer)	WP	0.19	0.0045	1	0	leaves and hop catkins	5.5	D07/84/93				
					21	hop catkins	<0.1					
					27	hop catkins	0.1					
					35	hop catkins	0.13					
					27	kiln-dried hops	<u>0.64</u>					
Germany 1993 (Northern Brewer)	WP	0.16	0.0045	1	0	leaves and hop catkins	6.4	D07/85/93				
					20	hop catkins	0.27					
					27	hop catkins	0.29					
					34	hop catkins	0.13					
					27	kiln-dried hops	<u>0.79</u>					

Country, Year (Variety)	Application Form	Application			PHI, days	Commodity	Hexythiazox, mg/kg	Ref
		kg ai/ha	kg ai/hl	no.				
Germany 1993 (Magnum)	WP	0.18	0.0045	1	0	leaves and hop catkins	5.8	D07/86/93
					20	hop catkins	0.23	
					27	hop catkins	0.13	
					34	hop catkins	<0.1	
					27	kiln-dried hops	<u>0.61</u>	
Japan (Iwate) 1985 (Shinshyu- wase)	WP	0.35	0.005	2	7	dry hops	<u>14</u>	#94/10371
					14	dry hops	14	
					21	dry hops	5.4	
Japan (Yamagata) 1985 (Kirin 2-gou)	WP	0.25	0.005	2	7	dry hops	<u>16</u>	#94/10371
					14	dry hops	6.6	
					21	dry hops	12	
USA 1995 (Galena)	(ID) WP	0.29	0.030	1	59	dry hops	0.1 <0.1	AA950601
USA 1995 (Galena)	(ID) WP	0.29	0.030	1	45	dry hops	0.34 0.36	AA950601
USA 1995 (Nugget)	(OR) WP	0.29	0.037	1	59	dry hops	0.21 0.20	AA950601
USA 1995 (Nugget)	(OR) WP	0.27	0.037	1	44	dry hops	1.3 0.99	AA950601
USA 1995 (Galena)	(WA) WP	0.25	0.018	1	60	dry hops	1.2 1.9	AA950601
USA 1995 (Galena)	(WA) WP	0.28	0.020	1	45	dry hops	3.5 3.9	AA950601

1/ growth stage: pre-bloom to bloom. Note: hops growth stages are to the wire, lateral shoots, flower formation, burrs and cones.

2/ growth stage: bloom

3/ growth stage: early bloom

4/ growth stage: early burr

5/ growth stage: burr.

## FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received information on the fate of hexythiazox residues during the drying of hops and brewing of beer.

Beck and Tilting (1994a) described the standardised procedures for drying hops and brewing beer. The green hops are dried at approximately 65°C for 7 hours with good air circulation, during which time the moisture content of green hops is reduced from approximately 80% to about 10% (equivalent to a concentration factor of 3.3). The beer production process is shown in Figure 1. The residue data for the dried hops, beer and spent hops are included in the supervised trial results in Table 3.

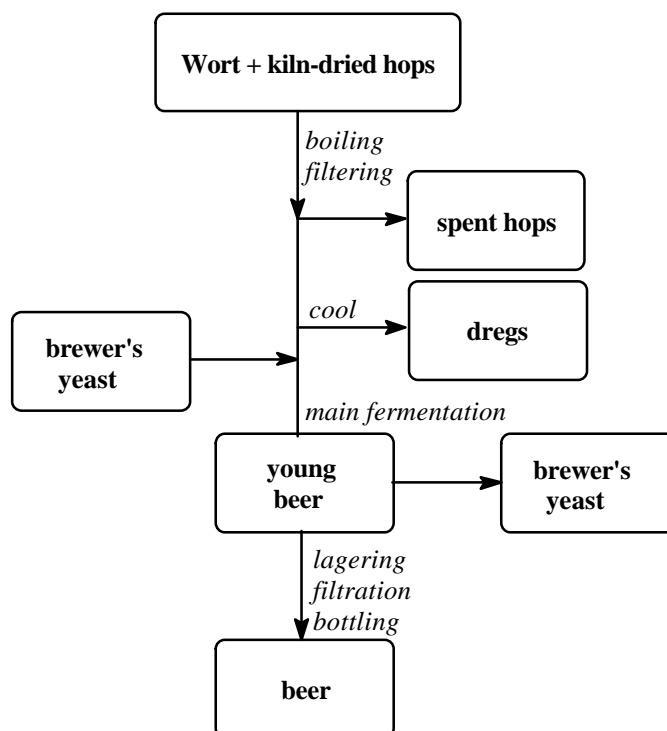


Figure 1. Beer production from wort and kiln-dried hops (Beck and Tilting, 1994a).

Table 4. Residue data and processing factors for hexythiazox in hops. Trial details are shown in Table 3.

Trial	Hexythiazox residues, mg/kg				Processing factors		
	green hops	dry hops	beer	spent hops (dry)	green hops → dry hops	dry hops → beer	dry hops → spent hops
D07/85/92	0.48	0.93	<0.05	0.65	1.9	0 (<0.054)	0.70
D07/86/92	0.96	1.5	<0.05	0.61	1.6	0 (<0.033)	0.41
D07/87/92	0.53	1.3	<0.05	0.53	2.5	0 (<0.038)	0.41
D07/88/92	0.23	0.88	<0.05	0.32	3.8	0 (<0.057)	0.36
D07/82/93	0.181	0.792			4.4		
D07/83/93	0.123	0.708			5.8		
D07/84/93	0.103	0.637			6.2		
D07/85/93	0.288	0.792			2.8		
D07/86/93	0.126	0.614			4.9		
					mean 3.7	0 (<0.046)	0.47
					median 3.8	0 (<0.046)	0.41

The mean processing factor for dry hops is 3.7 (range 1.6-6.2), which is quite close to the value calculated on the assumption of a moisture decrease from 80% to 10% and no loss of hexythiazox. Hexythiazox residues were not detected in beer in the four processing trials, which suggests that very little if any hexythiazox reaches the beer. Approximately half of the hexythiazox on the dry hops before brewing remains on the dry spent hops.

## NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was informed of the following national MRLs for hexythiazox.

Country	MRL, mg/kg	Commodity
Belgium	0.05	apple, pear
France	0.5	beans, pome fruits, wine grapes
	0.2	citrus, maize, other vegetables, peach, plum, other vegetables
Germany	3	hops
	0.5	grapes
	0.2	pome fruits, stone fruits
	0.05	other foods of vegetable origin
Italy	1	apple, kiwifruit, peach
	0.5	cucumber, eggplant, pear, strawberry, sweet pepper
	0.05	soybean
Japan	35	tea
	30	hops
	2	fruit, other vegetables
	0.5	bean, pea, potato
	0.2	sugar beet
Netherlands	0.2	beans (legume vegetables), bilberries, blueberries, cranberries, currants (red, black and white), elderberries, gooseberries, grapes, raspberries
	0.1	fruiting vegetables (aubergines, courgettes, cucumbers, gherkins, melons, okra, peppers, pumpkins, tomatoes, watermelons), strawberries
	0.05	pome fruit
	0.02*	other small fruit
	0* (0.02)	other food commodities
New Zealand	0.5	peach
	0.2	mandarin
Poland	0.5	fruits, vegetables
Spain	1	citrus
	0.5	other fruit, sweet pepper
	0.1	cucurbits
	0.05	other food and feedstuffs of plant origin
Switzerland	0.5	fruit
Taiwan	2	citrus (peel), lemon (peel), grapefruit (peel), orange (peel)
	1	guava, kaki plum, strawberry, table grapes, wax apple, wine grapes
	0.5	apple, citrus (without peel), grapefruit (without peel), lemon (without peel), loquat, orange (without peel), peach, pear, plum
USA	0.3	pear
	0.02	apple

## APPRAISAL

Hexythiazox was first evaluated in 1991 and again in 1994. It was scheduled for evaluation by the 1998 JMPR because the 28th Session of the CCPR (1996) was informed that data on hops would be made available (ALINORM 97/24, para 75). The Meeting received information on methods of analysis, registered uses, supervised residue trials and processing studies on hops.

The analytical method used in the supervised trials and processing studies in Germany relies on solvent partition and column chromatography for clean-up. In the GLC determination the residue is injected into a hot injector (300°C) where hexythiazox and its metabolites are converted to 5-(4-chlorophenyl)-4-methylthiazolidin-2-one (PT 1-3) which becomes the analyte. The LODs were 0.05 mg/kg in beer and brewing wastes, 0.1 mg/kg in green hops, and 0.5 mg/kg in dry hops.



The 1991 JMPR reported that hexythiazox is the main residue in crops and its metabolites are present in negligible amounts. The residues determined by this “total residue” method may therefore be accepted as equivalent to residues of hexythiazox, the current definition of the residue.

The method was shown to be free of interference from 26 pesticides registered for use on hops in the USA.

Hexythiazox was hydrolysed with alkali to convert it to PT 1-3 in the analytical method used in the US trials on hops. An LOD of 0.1 mg/kg was achieved for dry hops.

A similar extraction and clean-up was used in the Japanese trials but there was no hydrolysis step and the hexythiazox was determined by HPLC as the parent compound. An LOD of 0.2 mg/kg was achieved.

Hexythiazox residues on green and dry hops were stable during a two-year study of freezer storage stability, but the residues in beer declined by approximately half during refrigerator storage at 4°C for two years.

Hexythiazox is not registered for use on hops in the USA so the US trials could not be evaluated.

GAP for hops in Japan permits 2 applications at 0.0033-0.005 kg ai/hl or 0.2-0.3 kg ai/ha with harvest 7 days after the final application. In two Japanese trials in 1985 where the trial conditions were close to GAP the residues in dry hops were 14 and 16 mg/kg 7 days after the second application.

Hexythiazox is registered for use on hops in Germany where it may be applied at spray concentrations of 0.003-0.0045 kg ai/hl with an application rate not exceeding 0.15 kg ai/ha. Two applications are permitted and a 28 days PHI is specified. Hexythiazox was applied to hops in 9 trials in Germany in 1992 and 1993 following the conditions of German GAP. Only one application was made instead of the possible 2, but the average half-life of the hexythiazox residues in the 1992 trials was 10 days, which suggests that an earlier application would contribute no more than 20% of the final residue and probably much less because the first application would be at an earlier growth stage. The residues in dry hops from the 9 German trials according to GAP in rank order (median underlined) were 0.61, 0.64, 0.71, 0.79, 0.79, 0.88, 0.93, 1.3 and 1.5 mg/kg.

The residues in the Japanese trials (14 and 16 mg/kg) could not be combined with those in the German trials because they appeared to be from separate populations. The Meeting considered that 2 trials were insufficient to support an MRL, so the estimated maximum residue level was based on the highest data population with sufficient support, in this case the 9 German trials.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.88-1.5 mg/kg for hexythiazox in dry hops.

In 4 trials where beer was brewed from dry hops containing hexythiazox residues of 0.88-1.5 mg/kg, no residues (<0.05 mg/kg) were detected in the beer. The level of hexythiazox residues in dry spent hops was approximately half that in the initial dry hops. From 57% to 86% of the hexythiazox residues in the dry hops was accounted for in the spent hops and dregs from the process. Calculation showed that if all the remaining hexythiazox was in the beer, i.e. no losses occurred during boiling and fermentation, the level in the beer would be less than 0.0009 mg/l. The Meeting concluded that the STMR for hexythiazox residues in beer should be 0 mg/kg.

## RECOMMENDATIONS

On the basis of data from supervised trials the Meeting estimated the maximum residue levels and STMRs listed below. The maximum residue level for hops is recommended for use as an MRL.

Definition of the residue for compliance with MRLs and for the estimation dietary intake: hexythiazox.

Commodity		MRL, mg/kg		STMR, mg/kg
CCN	Name	New	Previous	
DH 1100	Hops, dry	2	-	0.79
	Beer			0

## DIETARY RISK ASSESSMENT

A maximum residue level for hexythiazox in hops has been recommended for use as an MRL. A processing study has resulted in an estimated STMR level of 0 mg/kg for hexythiazox residues in beer. All the other in values used for the intake estimation are previously established CXLs.

Estimated daily intakes for hexythiazox for the 5 GEMS/Food regional diets are in the range of 0 to 5% of the ADI. The Meeting concluded that the intake of residues of hexythiazox resulting from its uses that have been considered by the JMPR is unlikely to present a public health concern.

## REFERENCES

- Beck, J. and Tilting, N. 1994a. Study on residue behavior of BAS 9075 1 A (Synonym: BAS 9075 1 I) in hops and hops processed fractions, following application under field conditions in Germany, 1992. Study DE/AR/01/92. Report 3699. Includes trials D07/85/92, D07/86/92, D07/87/92, D07/88/92. Document #BASF 94/10325. BASF, Germany. Unpublished.
- Beck, J. and Tilting, N. 1994b. Study on residue behavior of BAS 9075 1 A (Synonym: BAS 9075 1 I) in hops following application under field conditions in Germany, 1993. Study DE/AR/01/93. Report 3948. Includes trials D07/82/93, D07/83/93, D07/84/93, D07/85/93, D07/86/93. Document #BASF 94/10324. BASF, Germany. Unpublished.
- Carringer, S.J. 1996. Magnitude of the residue of hexythiazox in hops raw agricultural commodity. AASI study AA950601. American Agricultural Services Inc., USA. EN-CAS project 95-0002. EN-CAS Analytical Laboratories, USA. Unpublished.
- Fomenko, J. 1997. Evaluation of hexythiazox metabolites through the FDA multiresidue methods. Maxim study A008.083. Maxim Technologies, Inc., USA. BASF study 97062. Document #97/5299. BASF, USA. Unpublished.
- McKinney, F.R. and Tomkinson. 1996. Analytical report. Magnitude of the residue of hexythiazox in hops raw agricultural commodities. AASI study AA950601. American Agricultural Services Inc., USA. EN-CAS project 95-0002. EN-CAS Analytical Laboratories, USA. Unpublished.
- Nippon Soda Co., Ltd. 1994. Residue results of hexythiazox on hop in Japan. Report RD-9427. Nippon Soda Co., Ltd. Document #BASF 94/10371. Unpublished.
- Soeda, Y., Gomyo, T., Tokieda, M. and Kobayashi, S. 1988. Analytical method for hexythiazox in hop by high-performance liquid chromatography. Report EC-119. Nippon Soda, Co., Ltd. Unpublished.
- Tilting, N. 1994. Determination of hexythiazox and its metabolites in soil, apples, strawberries (green plant and berries), hops (green and dried), beer, yeast and brewing wastes by GC with NPD. Report 3944. Method 343. Document #BASF 94/10226. BASF, Germany. Unpublished.
- Tilting, N. 1995. Validation of BASF method 343/1 for the determination of BAS 9075 I (hexythiazox) in dried hops. Study 30796. Document #BASF 95/11149. BASF, Germany. Unpublished.
- Tilting, N. 1998. Storage stability of hexythiazox residues in green hops, dried hops, spent hops and beer. Study DE/1/LST02/94. BASF. Report 98/10695. Unpublished.
- Williams, M. 1996. Specificity test for BASF analytical method 343/1 "Determination of hexythiazox and its metabolites in dried hops by GC with NPD." Horizon

report HL10138. Horizon Laboratories, USA. BASF  
study 95101. BASF report A9554. Document #BASF

96/5006. BASF, USA. Unpublished.