

## BIFENTHRIN (178)

### EXPLANATION

Bifenthrin was first evaluated at the 1992 JMPR and MRLs were estimated for cereals, fruits, potatoes, hops, animal feeds and animal commodities. MRLs of 0.05\* mg/kg were recommended for barley, maize and wheat to cover field application. Information has now been made available on the use of bifenthrin as a grain protectant on stored grain.

At the 26th (1994) Session of the CCPR (ALINORM 95/24, para 295) the Delegations of Germany and France considered that the available data on dry hops were inadequate for the proposed MRL. Additional explanatory notes on bifenthrin residue trials on hops have now been made available to the Meeting. Details of recent supervised trials on hops in the UK have also been supplied.

Information on registered uses of bifenthrin in the UK was made available to the Meeting.

### METHODS OF RESIDUE ANALYSIS

Burden (1994) described a residue analytical method for bifenthrin and malathion in cereal grains and milling and baking products. The method relies on acetone extraction followed by solvent partition and column chromatography for clean-up and GLC with an ECD for quantitative analysis. The method was capable of determining bifenthrin and malathion simultaneously. The LOD for both compounds was stated to be 0.01 mg/kg, but no recovery data were available at this level on the cereal substrates. No information was available on the extraction efficiency of acetone for aged bifenthrin residues.

Analytical recoveries of added bifenthrin were grain 93-107% (15 samples), bran 92 and 99%, white flour 109%, wholemeal flour 104%, white bread 96% and wholemeal bread 101%. Recoveries were determined at concentrations of 0.1-0.4 mg/kg in grain, 0.3 and 20 mg/kg in bran, and 0.3 mg/kg in white flour, wholemeal flour, white bread and wholemeal bread.

Kennedy (1994) used a similar method for the analysis of hops. Analytical recoveries of 67-113% were achieved from spiked samples (11) over the concentration range 0.01 mg/kg to 10 mg/kg. The limit of determination was 0.01 mg/kg.

### USE PATTERN

Bifenthrin is a pyrethroid insecticide particularly effective against Bostricid beetles (*Prostephanus truncatus* and *Rhyzopertha dominica*), but will also control *Sitophilus spp*, *Oryzaephilus spp*, and other insect pests. Proposed and registered uses of bifenthrin as a grain protectant are listed in Table 1. Registered uses of bifenthrin in the UK are listed in Table 2.

Table 1. Uses of bifenthrin as a protectant for stored grain.

Country	Form.	Application rate			
		bifenthrin, g/l	malathion, g/l	bifenthrin, g ai/t	malathion, g ai/t
Belgium	EC	20	400	0.3	6
Belgium	UL	7.5	150	0.3	6
Brazil <sup>1</sup>	EC	25 g/l bifenthrin + 125 g/l piperonyl butoxide		0.4	
France <sup>1</sup>	EC	20	400	0.3	6
France <sup>1</sup>	UL	7.5	150	0.3	6
Morocco <sup>1</sup>	EC	20	400	0.3	6
Morocco <sup>1</sup>	UL	7.5	150	0.3	6
Poland <sup>1</sup>	EC	20	400	0.3-0.4	6-8
Poland <sup>1</sup>	UL	7.5	150	0.3-0.4	6-8
UK <sup>1</sup>	EC	20	400	0.3	6
UK <sup>1</sup>	UL	7.5	150	0.3	6

<sup>1</sup> Proposed registration

The label and proposed labels also carry instructions for the treatment of grain stores 3-4 weeks before filling. The formulation is applied at the rate of 2.5 g bifenthrin per 100 m<sup>2</sup> of surfaces within the store.

Table 2. Registered uses of bifenthrin in the UK. All EC foliar applications.

Crop	Application			PHI, days
	Rate per applic., kg ai/ha	Spray conc, kg ai/hl	No.	
Apple	0.05	0.01	2	14
Barley, winter	0.0062	0.0015-0.003	2	60
Broccoli	0.0075	0.0012-0.0024	2	1
Brussels sprouts	0.0075	0.0012-0.0024	2	1
Cabbage	0.0075	0.0012-0.0024	2	1
Calabrese	0.0075	0.0012-0.0024	2	1
Cauliflower	0.0075	0.0012-0.0024	2	1
Hops	0.014-0.09	0.004	5	10
Linseed	0.0075	0.0018-0.0036	2	115
Oats, winter	0.0062	0.0015-0.003	2	60
Pear	0.05	0.01	2	14
Peas	0.0075	0.0018-0.0036	2	3
Rape, winter oilseed	0.0075	0.0018-0.0036	2	150
Rye	0.0062	0.0015-0.003	2	60
Strawberries	0.024-0.04	0.004	2	14
Triticale	0.0062	0.0015-0.003	2	60
Wheat, durum	0.0062	0.0015-0.003	2	60
Wheat, winter	0.0062	0.0015-0.003	2	60

In the USA bifenthrin is used on hops to control hop aphid and two-spotted spider mite. It is applied as a high-volume spray up to 3 times at a concentration of 0.0033 kg ai/hl and a rate of 0.1 kg ai/ha. The PHI is 14 days.

## RESIDUES RESULTING FROM SUPERVISED TRIALS

Kennedy (1994) reported on the bifenthrin residues in dried hops resulting from the application of bifenthrin in supervised field trials in the UK. The data are summarised in Table 3. Harrison and Owen (1994) used plot sizes of 5 or 6 rows of 15 m of hops in the trials. The hops were treated 5 times to run-off in high-volume applications. Two of the trials were in Kent and three in the West Midlands.

Table 3. Residues of bifenthrin in dried hops from high-volume foliar application of 5 x 0.004 kg ai/l EC in supervised trials in the UK in 1993 (Kennedy 1994).

Variety	Day	Residues, mg/kg	Ref.
Challenger	0	5.5 c1.4	FCC 0693
	7	5.1 c0.10	
Target	0	4.6 c<0.01	FCC 0693
	7	2.6 c<0.01	
Target	0	6.2 c0.03	FCC 0693
	7	5.5 c0.05	
Challenger	0	6.8 c1.1	FCC 0693
	7	4.5 c0.65	
Yeoman	0	5.3 c0.02	FCC 0693
	7	4.1 c0.01	

c: control sample

Residue data from supervised trials on hops in Germany were recorded in the 1992 Residue Evaluations. Additional explanatory notes have now been provided and the same data are presented in more detail in Table 4.

Table 4. Residues of bifenthrin in dried hops from EC foliar application of EC in supervised trials in Germany. Data were previously summarized in Table 5 of the 1992 bifenthrin residue evaluation. Underlined residues are from treatments according to UK GAP.

Year (variety)	Application			Day	Residues, mg/kg	Ref.
	kg ai/ha	kg ai/hl	No.			
1984 (Tettnanger Früh-hopfen)	1×0.09 +4×0.15	0.003	5	0	3.6	008334 73/44
				3	2.1	
				5	0.5	
				7	1.2	
				10	<u>1.9</u>	
1984 (Northern Brewer)	1×0.09 +4×0.15	0.003	5	0	4.8	008335 73/44
				3	3.1	
				5	2.5	
				7	1.8	
				10	<u>1.9</u>	
1984 (Spalter)	1×0.09 +4×0.15	0.003	5	0	6.4	008336 73/44
				3	7.2	
				5	4.1	
				7	4.9	
				10	<u>2.9</u>	
1984 (Hersbrucker Spät)	1×0.09 +4×0.15	0.003	5	0	0.7	008337 73/44
				3	0.3	
				5	2.0	
				7	0.3	
				10	<u>0.7</u>	
1985 (Spalter)	1×0.075 +3×0.13	0.0025	4	0	2.2	008419 73/52
				3	4.0	
				5	2.5	
				7	1.4	
				10	<u>0.9</u>	
1985 (Hersbrucker Spät)	1×0.075 +3×0.13	0.0025	4	0	2.8	008420 73/52
				3	2.7	
				5	1.5	
				7	1.1	
				10	<u>4.2</u>	

Year (variety)	Application			Day	Residues, mg/kg	Ref.					
	kg ai/ha	kg ai/hl	No.								
1985 (Northern Brewer)	1×0.075 +3×0.13	0.0025	4	0	8.2	008421 73/52					
				3	1.5						
				5	9.0						
				7	8.1						
				10	<u>0.1</u>						
1985 (Tettninger Früh-hopfen)	1×0.075 +3×0.13	0.0025	4	0	2.3	008422 73/52					
				3	2.5						
				5	1.6						
				7	1.5						
				10	<u>1.8</u>						
1987 (Golden Brewer)	2×0.075 +3×0.13	0.0025	5	0	g2.8 c0.13	DOW 02084 73/72B					
				3	g1.7 c0.16						
				5	g1.6 c0.13						
				7	g0.94 c0.09						
				10	g1.6 c0.55						
				7	1.6 c0.90						
				10	<u>2.7</u> c1.2						
				1987 (Golden Brewer)	2×0.075 +3×0.13		0.0038	5	0	g3.1 c0.61	DOW 02085 73/72B
									3	g1.9 c0.57	
									5	g1.4 c0.49	
7	g0.90 c0.59										
10	g1.6 c0.89										
7	1.6 c0.60										
10	<u>2.5</u> c1.3										
1987 (Northern Brewer)	2×0.075 +3×0.13	0.0025	5	0	g1.2	DOW 02086 73/72B					
				3	g0.92						
				5	g0.50						
				7	g0.92						
				10	g0.28						
				7	2.1						
				10	<u>1.0</u>						
1987 (Northern Brewer)	2×0.075 +3×0.13	0.0038	5	0	g1.4	DOW 02087 73/72B					
				3	g0.59						
				5	g1.3						
				7	g0.79						
				10	g1.1						
				7	2.3						
				10	<u>1.9</u>						

g: green hops. c: control sample

Haubruege *et al.* (1994) in Belgium treated grain (50 kg batches) by spraying and mixing in a cement mixer. Grain was then stored in mini-silos in a room where the temperature and humidity were not strictly controlled, but were measured. Temperatures were in the range 14.4 to 19.8°C, with grain moistures between 13.1% and 16.0%. Grain samples (200 g) were withdrawn at intervals and held at -18°C until analysis.

Binns *et al.* (1994) treated wheat (batches of 50 kg) with bifenthrin or a bifenthrin/malathion mixture in a cement mixer and then stored the treated grain at 20°C at ambient humidity in the UK. After various storage intervals samples (1.2 kg) were withdrawn for chemical analysis and at the 1-month and 3-month intervals samples of 3 kg were sent to a milling and baking laboratory.

Harrison and Derbyshire (1994) in the UK applied bifenthrin and malathion to barley grain, variety Marinka, moving down a chute into a grain store. The grain moisture was in the range 13% to 15%.

Table 5. Residues of bifenthrin and malathion resulting from supervised trials on stored grain after post-harvest applications. All 1992.

Country	Grain weight, temp	Form	Treatment, g ai/t	Storage time	Residues, mg/kg		Reference
					Bifenthrin	Malathion	
<b>BARLEY</b>							
UK	1 tonne	EC	b 0.4 + m 8.0	1 day	0.30	4.6	AK/2020/FM
				1 month	0.32	3.3	
				3 months	0.31	2.0	
				6 months	0.28	0.78	
				12 months	0.26	0.25	
UK	25-50 kg	EC	b 0.3 + m 6.0	1 day	0.22	3.0	AK/2020/FM
				1 month	0.22	2.4	
				3 months	0.29	1.9	
				6 months	0.24	0.42	
				12 months	0.19	0.20	
<b>WHEAT</b>							
Belgium	50 kg 15-20°C	UL	b 0.3 + m 6.0	1 day	0.18	4.4	11
				1 month	0.17	4.8	
				3 months	0.22	2.1	
				6 months	0.14	0.86	
				12 months	0.16	0.69	
Belgium	50 kg 15-20°C	EC	b 0.3 + m 6.0	1 day	0.18	3.8	11
				1 month	0.19	3.6	
				3 months	0.14	1.9	
				6 months	0.15	0.35	
				12 months	0.15	0.62	
Belgium	50 kg 15-20°C	UL	b 0.3 + m 6.0	1 day	0.23	5.5	11
				1 month	0.23	2.3	
				3 months	0.24	2.1	
				6 months	0.14	1.1	
				12 months	0.19	0.79	
Belgium	50 kg 15-20°C	EC	b 0.3 + m 6.0	1 day	0.23	6.3	11
				1 month	0.16	4.2	

Country	Grain weight, temp	Form	Treatment, g ai/t	Storage time	Residues, mg/kg		Reference
					Bifenthrin	Malathion	
				3 months 6 months 12 months	<0.01 <sup>1</sup> 0.25 0.12	0.12 <sup>1</sup> 0.83 0.50	
France (Us)	25-50 kg	UL	b 0.3 + m 6.0	1 day 1 month 3 months 6 months 12 months	0.23 0.22 0.23 0.22 0.26	3.5 2.2 1.9 1.2 1.0	73/89-1012 CSL
France (Us)	25-50 kg	EC	b 0.3 + m 6.0	1 day 1 month 3 months 6 months 12 months	0.24 0.20 0.21 0.21 0.19	3.6 2.4 1.8 0.9 0.29	73/89-1012 CSL
UK	25 kg 20°C	EC	b 0.5	1 day 1 month 3 months 6 months 12 months	0.37 0.39 0.38 0.39 0.40		AB09
UK	25 kg 20°C	EC	b 0.3 + m 6.0	1 day 1 month 3 months 6 months 12 months	0.25 0.26 0.24 0.27 0.22	3.8 2.5 1.8 1.2 0.53	AB09
UK	25 kg 20°C	UL	b 0.3 + m 6.0	1 day 1 month 3 months 6 months 12 months	0.26 0.23 0.25 0.28 0.24	3.5 2.4 2 1.4 0.82	AB09

b: bifenthrin m: malathion

<sup>1</sup> sample possibly interchanged with control sample

## NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following MRL had been established.

Country	MRL, mg/kg	Commodity
Belgium	0.5	Stored grain

## FATE OF RESIDUES IN STORAGE AND PROCESSING

Baxter (1995), in a laboratory-scale experiment, reported on the fate of bifenthrin and malathion during the malting of treated barley. Barley (batches of 350 g) treated at twice the recommended rate with bifenthrin and malathion was malted and the malts analysed for residues. Residues of bifenthrin in the barley and malt were 0.31 mg/kg and 0.026 mg/kg respectively; residues of malathion in the barley and malt were 4.6 mg/kg and not detected (<0.02 mg/kg) respectively. Information on the treatment rates, storage intervals before malting and the residue levels on the barley were not provided.

## APPRAISAL

Bifenthrin was first evaluated at the 1992 JMPR and MRLs of 0.05\* mg/kg were recommended for barley, maize and wheat to cover field applications. Information has now been made available on the use of bifenthrin as a grain protectant on stored grain.

At the 26th Session of the CCPR (ALINORM 95/24, 1994, para 295) the delegations of Germany and France considered that the available data on dry hops were inadequate for the proposed MRL. Additional explanatory notes on bifenthrin residue trials on hops have now been made available to the Meeting. Details of recent supervised trials on hops in the UK have also been supplied.

The residue analytical method for bifenthrin in cereal grains and milling and baking products relies on acetone extraction followed by solvent partition and column chromatography for clean-up and GLC with ECD for quantitative analysis. Good analytical recoveries were achieved for bifenthrin on grain (0.1-0.4 mg/kg), bran (0.3 and 20 mg/kg) and white flour, wholemeal flour and white bread (all 0.3 mg/kg).

A similar method was used for the analysis of hops where analytical recoveries of 67-113% were achieved for 11 spiked samples over the concentration range 0.01 mg/kg to 10 mg/kg. The limit of determination was 0.01 mg/kg.

In the UK registered use bifenthrin may be applied 5 times to hops at a spray concentration of 0.004 kg ai/hl. The hops may be harvested 10 days after the final application. The hops in the UK supervised trials were harvested 7 days after the final application, which was not strictly within GAP. However, the Meeting regarded the results as being consistent with the data from the German trials and as providing additional support.

The Meeting was informed that a registration for the use of bifenthrin on hops would not be pursued for the time being in Germany. The Meeting noted that the German residues data for dried hops, where bifenthrin spray concentrations were 0.0025-0.0038 kg ai/hl and the hops were harvested 10 days after the final application, were within UK GAP and re-evaluated the data according to UK GAP. Residues on dried hops at 10 days PHI in the 12 trials were 0.1, 0.7, 0.9, 1.0, 1.8, 1.9, 1.9, 1.9, 2.5, 2.7, 2.9 and 4.2 mg/kg.

The US use pattern on hops was reported in the 1992 JMPR Residue Evaluations as pending. The US trial data on hops reported in the 1992 Evaluations were evaluated against US GAP for hops reported to the current Meeting (3 applications, 0.1 kg ai/ha, spray concentration 0.0033 kg ai/hl and 14 days PHI). Residues in dried hops in the eight US trials were 0.5, 0.5, 0.7, 0.9, 1, 5, 5 and 5 mg/kg. Residues in dried hops from the same trials harvested 28 days after the final application also ranged up to 5 mg/kg.

Bifenthrin residues in dried hops from the total of twenty trials in the USA and Germany were 0.1, 0.5 (2), 0.7 (2), 0.9 (2), 1.0 (2), 1.8, 1.9 (3), 2.5, 2.7, 2.9, 4.2, and 5 (3) mg/kg. With a number of residues from supervised trials at 5 mg/kg, it is likely that in commercial practice residues in excess of 5 mg/kg will occur. The Meeting agreed that the data supported the current recommendation of 10 mg/kg for bifenthrin in dried hops.

Bifenthrin is effective as a grain protectant. It is registered in combination with malathion for use on stored grain in Belgium, and is proposed for registration in Brazil, France, Morocco, Poland and the UK. Bifenthrin is to be used at 0.3-0.4 g ai/t in combination with malathion at 6-8 g ai/t.

Storage experiments on barley in the UK and on wheat in Belgium, France and the UK showed that bifenthrin residues are stable for 12 months on stored grain at 20°C. Other pyrethroid grain



protectants have shown similar persistence. The levels of bifenthrin on the grain at the beginning and end of storage will essentially be the same.

The Meeting was reluctant to proceed with a recommendation for cereals until a number of points had been clarified. Information is needed on the efficiency of extraction of aged residues, national MRLs covering use on stored grains, and the fate of bifenthrin residues during commercial milling, baking and malting. Information should also be provided when proposed registrations become official or when new registrations for grain protectant uses are obtained.

## **FURTHER WORK OR INFORMATION**

### Desirable

1. Information on the efficiency of extraction by acetone of aged bifenthrin residues on stored grain. Acetone extraction is the first step in the analytical method.
2. Information on national MRLs for bifenthrin relating to uses on stored grains.
3. Information on the fate of bifenthrin during the commercial milling of wheat treated with it post-harvest. The studies should simulate commercial practices, including the effects of commercial cleaning.
4. Information on the fate of bifenthrin during the baking of bread.
5. Information on the fate of bifenthrin during the commercial malting of barley treated with it post-harvest. The studies should simulate the commercial process.

## **REFERENCES**

Baxter, E.D. 1995. Pesticide trial MP 177: grain protectant. BRF International, UK. Unpublished.

Benwell, L. 1994. Bifenthrin and malathion: the determination of residues in grain and milling/baking products. Report 73/103-1012. Hazleton Europe, UK. Unpublished.

Binns, T.J., 6Buckland, A. and Tamsett, S. 1994. An assessment of the residues of 3 pesticide formulations applied to wheat. Report AB09. Central Science Laboratory, UK. Unpublished.

Burden, A.N. 1994. Bifenthrin and malathion: the determination of residues in grain and milling/baking products. Report 73/89-1012. Hazleton Europe, UK. Unpublished.

Harrison, C. and Derbyshire, R. 1994. Study to generate crop samples for residue analysis following treatment with a formulated bifenthrin/malathion product (ECH 92465). Final report on project AK/2020/FM. Agrisearch, UK. Unpublished.

Harrison, C. and Owen, J. 1994. Programme to generate crop samples for residue analysis following treatment of hops with Talstar 10 EC. Final report on project AK/2242/FM. Agrisearch UK Ltd. Unpublished.

Haubrugge, E., Letellier, C. and Gaspar, C. 1994. Long-term effects of a bifenthrin/malathion insecticide: small bin tests. Report no. 11. Faculty of Agricultural Sciences, Belgium. Unpublished.

Kennedy, E.M. 1994. The determination of concentrations of bifenthrin in hops. Study no. FCC 0693. Restec Laboratories Limited. Unpublished.