BIFENTHRIN (178)

EXPLANATION

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 was provided to the 1995 and 1996 Meetings on the use of bifenthrin as a stored grain protectant. The 1996 JMPR recommended MRLs for wheat and milled commodities related to the use on stored grain.

The 28th Session of the CCPR (1996, ALINORM 97/24, para 79) noted that the CXLs for animal products might need to be changed if GAP for barley, wheat and maize is changed. The 29th Session (1997, ALINORM 97/24A, para 74) postponed discussion pending a review by the 1997 JMPR.

The 1996 JMPR listed the following information as desirable in connection with the use of bifenthrin as a grain protectant.

1. Validation of the analytical method for recoveries of bifenthrin residues from bread at the levels occurring in practice and at the LOD.

2. Information on the degree of extraction of bifenthrin residues from bread by the current procedure.

3. Information on national registrations and MRLs for bifenthrin covering its use on stored grain.

4. Information on the fate of bifenthrin during the simulated commercial malting of barley treated post-harvest (from 1995).

The Meeting received a report on the baking process used in the bread trials reviewed in 1996, information on the registration status of bifenthrin as a grain protectant and reports on a malting trial and on laboratory and silo scale grain storage trials.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Slaiding (1995) determined residues of bifenthrin and malathion in barley and malt by a GC-MS method with LODs of 0.01 mg/kg for bifenthrin and 0.02 mg/kg for malathion. The samples were soaked in water and then extracted with an acetone/methanol mixture. The extract was cleaned up by passage through small columns before analysis. Recoveries were 63-86% for bifenthrin and 65-81% for malathion.

The Australian Wheat Board laboratory analysed wheat samples (Table 1) by a method developed by Academy of Grain Technology. Wheat (50 g) was shaken for 15 minutes in a stoppered flask with hexane (50 ml). The mixture was allowed to stand for 42 hours and shaken for a further 15 minutes before filtration to provide a clear solution for GLC on a non-polar column with EC detection. The limit of detection was reported to be 0.01 mg/kg.

Agricultural Chemistry Branch samples (Table 1) were analysed for bifenthrin residues by a method for organochlorine residues method (Simpson, 1993). Wheat (5 g) was ground before extraction by shaking with methanol for 1 hour. Bifenthrin residues were partitioned into hexane after dilution of the methanol with water. The extract was dried before clean-up on a Florisil column. Bifenthrin residues were then determined by GLC with a Hall detector for chlorinated compounds. The limit of detection was 0.05 mg/kg and recoveries from spiked samples were 106% at 0.53 mg/kg and 101% at 0.27 mg/kg.

Maize and wheat samples in the South African trials (Table 3) were extracted with 10% acetone in hexane by shaking for 3 hours (Van der Linde, 1995). The extract was filtered through sodium sulphate and analysed by GLC with an ECD for bifenthrin and an FPD for malathion. Good recoveries were obtained from samples spiked at 0.2 and 1 mg/kg. Only a summary of the method was available.

USE PATTERN

The registered uses of bifenthrin on stored grain were recorded by the 1995 JMPR. Croatia and Romania issued temporary registrations for such uses in 1996 for 2 and 5 years respectively.

RESIDUES RESULTING FROM SUPERVISED TRIALS

In an Australian trial Bengston (1995) treated wheat (500 tonnes) in silo storage with a mixture of bifenthrin at 0.5 g ai/t and chlorpyrifos-methyl at 10 g ai/t and sampled the grain at intervals up to 6 months for residue analysis. The temperature of the stored grain fell from 24.8°C 1.5 months after treatment to 15°C at the end of the storage. The grain moisture level was in the range 9.3 to 9.8%.

Samples of treated grain were transferred to a freezer at -10°C 5-11 days after sampling, where they were held for 7-13 months before delivery to four laboratories for analysis. Samples analysed by Agricultural Chemistry were held at -20°C for approximately 10 weeks before analysis. No information was available on the conditions or duration of the storage of samples analysed by the Australian Wheat Board.

Table 1. Residues in wheat treated with bifenthrin + chlorpyrifos-methyl (0.5 + 10 g ai/t) and stored for 6 months (Bengston, 1995). Four laboratories[1] analysed the wheat.

Storage period, months	Residues, mg/kg				
	Bifenthrin		Chlorpyrifos-methyl		
	Lab AC	Lab AWB	Lab APG	Lab AWB	Lab NSW
0	0.56	0.5	8.9	9.9	6.3
1.5	0.32	0.4	7.1	7.7	-

Storage period, months	Residues, mg/kg				
	Bifenthrin		Chlorpyrifos-methyl		
	Lab AC	Lab AWB	Lab APG	Lab AWB	Lab NSW
3	0.43	0.4	7.0	7.7	6.1
4.5	0.37	-	7.1	7.9	5.6
6	0.43	0.4	6.4	6.6	5.2

[1]AC: Agricultural Chemistry Branch, Queensland Department of Primary Industries.
AWB: Australian Wheat Board, Victoria.
APG: Agricultural Production Group, Queensland Department of Primary Industries.
NSW: New South Wales Department of Agriculture.

De Baptista (1995) treated small lots (2 kg) of rice, wheat and maize with bifenthrin + piperonyl butoxide (0.4 + 2 mg/kg and 0.8 +4 mg/kg), in plastic bags. In each trial the treated grain was divided and stored at room temperature in 4 closed paper sacks, 500 g in each. At each sampling interval one sack of grain was taken for the determination of bifenthrin. Bifenthrin residues (Table 2) were considerably depleted after 60 days storage, whereas bifenthrin is generally persistent on wheat in bulk storage. Details of the GLC analytical method were not provided, but the LOD was 0.02 mg/kg and the analytical recoveries were 81±5%.

Table 2. Residues of bifenthrin in grain treated with bifenthrin and piperonyl butoxide and stored in closed paper sacks at room temperature in Brazil (De Baptista, 1995).

Grain	Treatment, mg/kg. bifenthrin + piperonyl butoxide	Storage interval, days	Bifenthrin, mg/kg
Rice (Araguaia)	b 0.4 + pb 2	0 15	0.3 0.4
Rice (Araguaia)	b 0.8 + pb 4	0 15	0.7 0.6
Rice (Araguaia)	b 0.4 + pb 2	0 15 30 60	0.4 0.4 0.2 0.1
Rice (Araguaia)	b 0.8 + pb 4	0 15 30 60	0.9 0.7 0.4 0.2
Wheat (BR23)	b 0.4 + pb 2	0 15 30 60	0.4 0.3 0.2 0.1
Wheat (BR23)	b 0.8 + pb 4	0 15 30 60	0.6 0.7 0.5 0.3
Wheat (BR23)	b 0.4 + pb 2	0 15	0.4 0.2
Wheat (BR23)	b 0.8 + pb 4	0 15	0.8 0.7
Maize (AG303)	b 0.4 + pb 2	0 15	0.5 0.4
Maize (AG303)	b 0.8 + pb 4	0 15	0.7 0.7

Grain	Treatment, mg/kg. bifenthrin + piperonyl butoxide	Storage interval, days	Bifenthrin, mg/kg
Maize (AG303)	b 0.4 + pb 2	0	0.4
		15	0.3
		30	0.2
		60	0.08
Maize (AG303)	b 0.8 + pb 4	0	0.8
		15	0.8
		30	0.6
		60	0.2

In a South African trail with stored grain Van der Linde (1995) treated maize and wheat grain in 20 kg lots with bifenthrin + malathion at 0.3 + 6.0 and 0.4 + 8.0 g ai/t and stored the grain in 20-l plastic containers with lids in ambient conditions for 20 weeks. The results are shown in Table 3. The residues on the grain were substantially lower than the target application rates. Milled samples were obtained by grinding the whole grain to a fine powder in a laboratory mill. Bifenthrin residues did not decrease during storage.

Table 3. Bifenthrin and malathion residues in stored maize and wheat after treatments of 20 kg lots and storage in 20 litre plastic bins (Van der Linde, 1995).

Grain	Storage interval, weeks	Treatment with bifenthrin 0.3 g ai/t + malathion 6.0 g ai/t				Treatment with bifenthrin 0.4 g ai/t + malathion 8.0 g ai/t			
		bifenthrin, mg/kg		malathion, mg/kg		bifenthrin, mg/kg		malathion, mg/kg	
		grain	milled	grain	milled	grain	milled	grain	milled
Maize	0	0.16	0.11	2.5	1.6	0.19	0.11	3.2	2.7
	4	0.12	0.14	1.7	1.0	0.16	0.12	1.9	1.0
	12	0.10	0.12	1.4	1.1	0.13	0.14	2.6	1.4
	20	0.14	0.14	1.7	1.3	0.19	0.16	2.8	1.6
Wheat	0	0.23	0.13	4.0	3.7	0.27	0.18	5.5	4.4
	4	0.22	0.20	3.9	4.7	0.24	0.26	4.0	4.6
	12	0.18	0.17	3.8	3.4	0.24	0.30	4.3	6.4
	20	0.20	0.38	3.4	4.2	0.27	0.27	4.3	6.0

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Full details of the Chorleywood baking process used in the baking trials reported in 1996 were provided at that time. The report (Collins, 1976) has now been made available. Essential features of the process are intense mechanical working of the dough within a few minutes, addition of ascorbic acid at 75 mg/kg, inclusion of fat at 0.7%, extra water at 3.5% and additional yeast.

Baxter (1995) provided a more detailed report of the barley malting trials reviewed by the 1995 JMPR. The trials were pilot scale (50 kg), whereas the 1995 JMPR had understood them to be with 350 g lots.

When barley was treated at 0.6 + 12 g ai/t with bifenthrin + malathion (twice the recommended rate) and malted, bifenthrin levels were considerably reduced and residues of

malathion effectively disappeared. Residue levels in the barley before screening and cleaning were not reported. The study was defective because the barley samples were stored for approximately 100 days at ambient temperature before malting began and no samples were taken at that time. The results are shown below.

Sample	Nominal treatment	
	Bifenthrin	Malathion
	0.60 g/t	12 g/t
	Residues, mg/kg	
Screened and cleaned barley[1]	0.31	4.6
Derooted malt[2]	0.026	<0.02

[1]Barley sampled 23-31 March 1994
[2]Malt prepared from barley stored at room temperature until 6 July 1994

APPRAISAL

Information was provided to the 1995 and 1996 Meetings on the use of bifenthrin as a stored grain protectant. The 1996 Meeting recommended MRLs for wheat and milled commodities related to this use. It was suggested at the CCPR that the CXLs for animal products might be affected by revised GAP for cereals.

The 1996 JMPR listed the following information as desirable in connection with the use of bifenthrin as a grain protectant.

Validation of the analytical method for recoveries of bifenthrin residues from bread at the levels occurring in practice and at the LOD.

Information on the degree of extraction of bifenthrin residues from bread by the current procedure.

Information on national registrations and MRLs for bifenthrin covering its use on stored grain.

Information on the fate of bifenthrin during the commercial malting of barley treated with it post-harvest. The studies should simulate the commercial process. (From 1995 JMPR).

No additional information was available on the analytical method for bifenthrin residues in bread.

Croatia and Romania have issued temporary registrations for the treatment of stored grain with bifenthrin.

The results of a barley malting trial were made available. It suggested that bifenthrin residues decreased substantially during the malting process but the study was defective because no barley samples were taken for analysis at the time the malting commenced.

The Meeting received additional data from trials with bifenthrin on stored grain. Bifenthrin residues are generally persistent during storage.

The recommendations of the 1992 JMPR for MRLs for bifenthrin in cattle fat, kidney, liver, meat and milk were based on the assumption that levels of bifenthrin in the diet of cows were unlikely to exceed 2 ppm, on the basis of a feeding study in which bifenthrin was fed at 5 ppm for 28 days. The 1995 Meeting recommended MRLs of 0.5 mg/kg in wheat and 2 mg/kg in wheat bran because of the post-harvest use on wheat. Because these levels do not exceed the level of 2 ppm in the feed on which the 1992 recommendations were based no change to the draft MRLs for cattle commodities is needed.

In a study of metabolism in laying hens (1992 JMPR) bifenthrin constituted 51.5% of the total residue in abdominal fat of 1.0 mg/kg produced by a feeding level of 40 ppm. In a feeding study on laying hens also reported in 1992 birds were fed for 28 days at 0.25 ppm. Total residues at the limit of detection of 0.01 mg/kg were detected only in fat, suggesting that the residue of bifenthrin in fat was 0.005 mg/kg. Bifenthrin residues in the eggs were 0.002-0.004 mg/kg.

The current CXLs for bifenthrin in chicken fat and the fat of chicken meat are 0.05* mg/kg, which should be adequate for a feed level of 2.5 ppm bifenthrin if the proportionality between levels in the feed and fat is the same as in the trial. The current CXL for bifenthrin in eggs is 0.01* mg/kg, which should be adequate for a feed level of 0.6-1.2 ppm bifenthrin.

The draft MRL for bifenthrin in wheat (to cover post-harvest use) is 0.5 mg/kg, and the STMR is 0.255 mg/kg. The current CXLs for chicken eggs, fat, meat and edible offal should be adequate for chickens consuming bifenthrin-treated wheat, which can be a major part of a poultry diet.

The 1996 Meeting estimated maximum residue and STMR levels for bifenthrin in bran produced from post-harvest-treated wheat of 2 and 0.89 mg/kg respectively. Bran may constitute 50% of the poultry diet; at this level the CXLs for chicken fat, meat and offal should be adequate. The CXL for eggs is probably adequate. It is at the LOD and its adequacy depends on assumptions about the proportion of bran in the poultry diet.

The Meeting made no recommendations to change the existing CXLs or draft MRLs for bifenthrin.

The Meeting noted that the data submitted had not provided the information which the 1995 and 1996 Joint Meetings needed to recommend MRLs for stored grains, except wheat, and their products.

Future submissions of data should meet the requirements of the *FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed.* The studies should be valid and supply the information listed in the 1995 and 1996 Evaluations.

FURTHER WORK OR INFORMATION

Desirable

1. Validation of the analytical method for recoveries of bifenthrin residues from bread at the levels occurring in practice and at the LOD.

2. Information on the degree of extraction of bifenthrin residues from bread by the current procedure.

3. Information on national registrations and MRLs for bifenthrin covering its use on stored grain.

4. Valid studies on the fate of bifenthrin during the malting of barley treated with it post-harvest. The studies should simulate the commercial process.

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