NOTE ON RESIDUES IN FLOUR RESULTING FROM MALATHION APPLICATION IN AN OPERATING MILL[1]

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For many years, various concentrations of pyrethrins plus piperonyl butoxide have been used for insect control in flour mills. However, pyrethrins recently have been in short supply, causing the milling industry to look for alternatives. The present study was conducted to determine what residues were found in mill streams and finished flour when malathion is sprayed around milling equipment during normal mill operation.

MATERIALS AND METHODS

The purifier floor was selected as the test site because an application of malathion in this area (116 by 45.5 ft) would produce the highest residues in the mill streams.

The material used was a 3% active ingredient oil-base spray that had been diluted from a standard 57% premium-grade malathion emulsifiable concentrate according to label instructions; 4 gal of the malathion solution was used to treat the test area. The spray was applied to the entire purifier floor including the areas around the bases of the purifiers and the junctures of the floor and wall from 8:30 to 8:47 a.m. while the mill was in full operation. The treatment was applied at 80 lb/in.^{2} with a De Vilbiss® compressed air sprayer with a standard nozzle.

Samples of tempered wheat, purifier suction flour, mainstream tailings, and patent flour were taken for analysis before, during, and after the application of malathion. The purifier suction flour represented 1.99% of the wheat milled and 2.69% of the total flour produced. The mainstream tailings flour represented 0.13% of the flour milled and 0.175% of the total flour produced. The mill was operating at 850 bu/hr.

As samples were collected, they were placed in double polyethylene bags to prevent loss of residue. When they arrived at the Manhattan laboratory *ca.* 2 hr after the last sample was bagged, they were placed in a freezer and held at 0°F. The following day, the samples were placed in metal cans, sealed, and shipped, packed in Dry Ice, to Dr. R. A. Simonaitis at the Stored-Product Insects Research and Development Laboratory at Savannah, GA, for the analysis of the residues. Table I presents the sampling schedule and residues found in the various mill streams by Dr. Simonaitis. The values represent the ppm malathion detected on two chromatography machines, the Micro-Tech®, which has a sensitivity of 0.1 ppm and the Hewlett-Packard®, which has a sensitivity of 0.06 ppm. The residue analyses were performed as follows: two hundred grams of the commodity was finely ground in a food homogenizer and then tumbled in a

[1]Mention of a pesticide or a commercial or proprietary product does not constitute a recommendation or endorsement by the U.S. Department of Agriculture.

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TABLE I
Malathion Residues in Mill Streams Before, During, and After Spraying the Purifier Floor of a Flour Mill

Sampling Schedule a.m.	Malathion in Purifier suction flour ppm	Malathion in Mainstream tailings ppm	Malathion in Patent flour ppm
8:20	< 0.06	<0.06	<0.20
8:30[a]	< 0.06	<0.06	
8:40	15.9	4.53	1.0
8:50	19.8	1.16	1.4
9:00	10.7	0.58	0.6
9:10	7.1		0.4
9:20	5.6		0.18
9:30	2.4	0.40	
9:50			<0.06
10:00	2.2	0.38	
10:20			<0.06

[a]Malathion spray applied 8:30–8:47 a.m.

mason jar for 1 hr at 10 rpm. Fifty grams was weighed into an Erlenmeyer flask with a ground glass stopper, and 150 ml of methylene chloride was added. The flasks were shaken on a wrist-action shaker for 2 hr. The solution was filtered rapidly through a fluted Whatman 2V filter paper into a vial, capped, and stored at −20°C before gas-chromatographic analysis. The analyses were performed on either a Hewlett-Packard Model 5750 or a Tracor Model 220 gas chromatograph, both equipped with a Tracor flame-photometric detector with a 526-μm phosphorus filter. The column conditions used were: column, 4 ft × 0.25-in. o.d. glass containing 5% OV 101 on 80–100-mesh Gas Chrom Q®; temperature, oven—220°C, injection port—270°C, detector—220°C, bypass valve—220°C; gas flow-rate, nitrogen—160 cm^3/min, hydrogen—200 cm^3/min, oxygen—40 cm^3/min; injection volume, 10 μl.

RESULTS AND DISCUSSION

No malathion residues were detected (less than 0.06 ppm) in any of the samples of tempered wheat. The highest residue, 19.8 ppm, was found in purifier suction flour sampled 20 min after the application began. There was 2.2 ppm present 1.5 hr later. The mainstream tailings contained 4.5 ppm 10 min after the treatment started; 80 min later, 0.38 ppm was found. The patent flour contained 1 ppm when it was sampled 10 min after the beginning of the application; 3 min after the spraying ceased, the flour contained 1.4 ppm; but 1 hr later, no residue could be detected (less than 0.06 ppm). The average residue in the patent flour during the 50-min sampling period was *ca.* 0.75 ppm.

Although a tolerance of 8 ppm has been established for wheat in storage, no malathion residue is allowed in flour consumed by humans. A tolerance for malathion in flour would have to be established before it could be used extensively in flour mills.

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