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Chlorpyrifos-methyl: Doses that Protect Hard Winter Wheat Against Attack by Stored-Grain Insects[1,2]

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ABSTRACT

Chlorpyrifos-methyl was applied to cleaned, hard winter wheat as a water emulsion at doses ranging from 1–10 ppm AI. The effectiveness of residues in samples withdrawn at intervals during a 12-mo storage showed that initial applications of 3 or more ppm AI controlled rice weevils, *Sitophilus oryzae* (L.), granary weevils, *S. granarius* (L.), and maize weevils, *S. zeamaiz* Motschulsky. In contrast, a dose as high as 8 ppm was required to kill ca. 90% of the lesser grain borers, *Rhyzopertha dominica* (F.), 83% of the confused flour beetles, *Tribolium confusum,* Jacquelin duVal, and 98% of the red flour beetles, *T. castaneum,* (Herbst) 12 mo after application.

Chlorpyrifos-methyl, a broad spectrum insecticide with an acute oral mammalian LD_{50} to rats > 1630 mg/kg, is being considered as a protectant for grain against insect attack. However, in a preliminary evaluation with applications of water emulsions to wheat and shelled corn (5, 10, and 20 ppm AI), the 5 ppm dose, although giving satisfactory initial kills, did not give the protection needed to control all test species for 6 mo. Therefore, studies were undertaken to determine the length of protection against attack of 6 species of laboratory-reared stored-grain insects provided by the application of doses of chlorpyrifos-methyl that ranged from 1–10 ppm.

MATERIALS AND METHODS. — Cleaned, hard winter wheat, 'Scout' variety, was tempered to 12.5% moisture by drying with forced air at 40.5°C immediately after it was removed from cold storage. The wheat was then allowed to equilibrate in covered drums for 35 days before 1000-g lots were placed in 3.8-liter large-mouth glass jars for treatment. Treatments consisted of emulsions prepared immediately before use by diluting the chlorpyrifos-methyl EC (0.24 kg/liter AI) with neutral distilled water so application of one ml deposited the calculated dose. Emulsions were kept in constant agitation at 25°C with a wrist action shaker during the applications to the wheat. Applications, handling of the treated wheat, and tests for toxicity were as described by LaHue (1977).

Four separate dilutions of the EC were prepared for each dose (4 treatment replications), and 4 subsamples from each replication were used in exposing each species of insect (thus, a total of 16 exposures/species/dose) to each interval of storage. The test species were rice weevil, *Sitophilus oryzae* (L.), granary weevil, *S. granarius* (L.), maize weevil, *S. zeamaiz* Motschulsky, confused flour beetle, *Tribolium confusum* Jacquelin duVal, red flour beetle, *T. castaneum* (Herbst), and lesser grain borer, *Rhyzopertha dominica* (F.).

Samples for residue analysis, taken at the beginning of each toxicity test exposure, were held at −20°C until analyzed. The method of residue analysis was adapted from Storrherr et al. (1964) and was described by LaHue (1977). All samples were analyzed in duplicate.

RESULTS AND DISCUSSION. — In laboratory studies, LD_{50} or LD_{95} values are used primarily in comparing the effectiveness of insecticides applied topically or

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[2] This paper reports the results of research only. Mention of a pesticide does not constitute a recommendation for use by the USDA nor does it imply registration under FIFRA as amended. Also, mention of a proprietary product does not constitute an endorsement by USDA.

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Table 1.—Mortality of *Sitophilus* exposed for 21 days to wheat treated with chlorpyrifos-methyl.

Dose (ppm)	Avg % mortality at indicated month posttreatment[a,b]			
	3	6	9	12
	Rice weevils			
1	96.0	81.2	69.3	7.5
2	100.0	100.0	100.0	85.3
3	100.0	100.0	100.0	100.0
Check	0	0.2	0.4	0.4
	Maize weevils			
1	97.0	81.0	55.7	4.2
2	100.0	100.0	66.7	55.5
3	100.0	100.0	100.0	100.0
4	100.0	100.0	100.0	100.0
5	100.0	100.0	100.0	100.0
Check	0	0	0	0
	Granary weevils			
1	100.0	89.4	88.1	4.7
2	100.0	100.0	99.0	64.0
3	100.0	100.0	100.0	100.0
Check	0	0	0	0

[a] 100% mortality was obtained for all doses when insects were exposed the 1st 2 mo of storage.
[b] 100% mortality of rice weevils and granary weevils was obtained with doses > 3 ppm; all maize weevils were killed by doses > 5 ppm at 12-mo posttreatment.

used as vapors or as residues on treated surfaces. Thus, data may reflect single exposures to the chemical, short periods of contact with a vapor, or a contact with a treated surface area. The control of insects confined to and feeding on treated grain should not be evaluated in such a manner; consequently, all mortality data have been summarized in tabular form.

All weevils were killed by doses of 1 ppm or greater in exposures made during the 1st 2 mo posttreatment. Table 1 reports avg percentage mortality of adult rice weevils, maize weevils, and granary weevils that resulted from the 21-day exposures to grain stored for as long as 12 mo. Maize weevils were slightly more tolerant of the residues than rice weevils and granary weevils during the last 6 mo of storage. For example, 6 maize weevils (of 400) survived the 21-day exposure to wheat treated with a 4 ppm dose and stored for 12 mo, and then when confined to untreated wheat after the exposure, produced 62 F_1 progeny.

Adult lesser grain borers were considerably more tolerant of the residue than the *Sitophilus* spp. However, all borers were killed by exposures to the insecticide 7 days posttreatment, but 6.7 and 15.1% of the adults survived on the grain with the 1 ppm dose after the grain had been stored 14 and 21 days. Mortality was high on grain treated with 4 ppm and stored for 6 mo and on grain treated with 7 ppm and stored for 9 mo (Table 2). A dose of 8 ppm was required to kill most of the exposed adults and prevent damage by progeny to the grain for 12 mo.

Adult confused flour beetles were the most tolerant of the 6 test species. Doses of 2–10 ppm inclusive produced 100% mortality 7 and 14 days posttreatment, but 4.0 and 29.8%, respectively, survived exposures to 1 ppm at these intervals. Also at 21 days posttreatment, 81.0 and 14.0% survived in grain treated with 1 and 2 ppm, respectively. A dose of 8 ppm caused 100% mortality of adults after 3 mo and a dose of 9 ppm caused 100% mortality after 9 mo (Table 2).

Adult red flour beetles were less tolerant of chlorpyrifos-methyl than confused flour beetles; a 3 ppm dose caused 100% mortality after 3 mo and a 5 ppm dose after 9 mo; also control was excellent at 12 mo posttreatment when the grain had been treated with 8 ppm (Table 2).

Table 3 reports residues found during the 12-mo study. The recoveries (determined by applying standard solutions to control samples) averaged 98.3± 8.0%, 95% confidence limits, for the mean. Analyses of unknowns were bracketed with analyses of standards.

Table 2.—Mortality of lesser grain borers and confused and red flour beetles, exposed for 21 days to wheat treated with chlorpyrifos-methyl.

Dose (ppm)	Avg % mortality at indicated months posttreatment					
	1	2	3	6	9	12
	Lesser grain borer					
1	85.9	77.0	18.8	18.4	10.2	13.7
2	95.1	70.0	78.0	50.5	20.3	22.6
3	100.0	97.0	93.0	73.3	35.6	34.3
4	100.0	100.0	99.0	96.0	63.0	39.5
5	100.0	100.0	100.0	96.0	67.0	50.3
6	100.0	100.0	100.0	100.0	76.4	67.9
7	100.0	100.0	100.0	100.0	88.1	80.1
8	100.0	100.0	100.0	100.0	90.8	90.3
9	100.0	100.0	100.0	100.0	99.0	95.1
10	100.0	100.0	100.0	100.0	100.0	98.8
Check	1.0	0.4	0	0	0	1.1
	Confused flour beetles					
1	12.9	7.0	3.4	2.0	0	0
2	67.0	34.0	38.5	6.0	0.9	0
3	94.2	82.2	49.5	16.0	20.4	0
4	100.0	84.0	69.0	32.7	18.5	1.9
5	100.0	85.5	79.0	41.0	36.0	6.2
6	100.0	97.0	84.2	58.4	47.0	12.7
7	100.0	100.0	92.2	81.7	90.6	68.7
8	100.0	100.0	100.0	85.5	85.1	83.1
9	100.0	100.0	100.0	100.0	100.0	85.5
10	100.0	100.0	100.0	100.0	100.0	89.3
Check	0	0	0	0	0	0
	Red flour beetles					
1	93.5	22.8	3.0	11.7	2.0	0
2	98.0	97.1	93.5	53.8	6.7	0
3	100.0	100.0	100.0	89.0	76.9	2.0
4	100.0	100.0	100.0	94.0	87.6	2.5
5	100.0	100.0	100.0	100.0	100.0	20.0
6	100.0	100.0	100.0	100.0	100.0	66.4
7	100.0	100.0	100.0	100.0	100.0	78.2
8	100.0	100.0	100.0	100.0	100.0	98.1
9	100.0	100.0	100.0	100.0	100.0	100.0
10	100.0	100.0	100.0	100.0	100.0	100.0
Check	0	0	0	0	0	0

Table 3.—Residues of chlorpyrifos-methyl on composite samples of wheat treated with increasing doses.

Dose (ppm)	Avg residues (ppm) for indicated posttreatment intervals								
	Days			Months					
	7	14	21	1	2	3	6	9	12
1	0.7	0.6	0.6	0.6	0.5	0.4	0.5	0.3	0.2
2	1.4	1.4	1.2	1.2	1.2	1.0	.9	.9	.5
3	2.5	2.3	2.0	2.0	2.0	1.7	1.4	1.2	.8
4	2.8	2.5	2.3	2.0	2.0	1.9	1.6	1.5	1.1
5	3.7	3.4	2.9	3.1	2.6	2.5	1.9	1.5	1.4
6	4.1	3.7	3.7	3.5	3.7	2.8	2.4	2.1	1.5
7	5.2	5.1	4.6	4.3	4.1	3.5	2.9	2.6	1.9
8	6.3	5.9	5.5	5.2	4.8	4.2	2.9	2.6	2.1
9	7.2	6.9	6.6	6.4	6.0	5.1	4.5	3.7	2.6
10	8.5	8.3	8.1	7.8	6.9	6.5	5.5	4.3	2.8

Since the relative effectiveness of the several doses of chlorpyrifos-methyl to 6 species of adult stored-product insects was established for a 12-mo period, the results may be used to determine the minimum effective doses required to kill the adults and to establish time intervals-effective dose (TIED) values for the species. For example, to obtain 100% mortality of rice weevils after 9 mo storage, an initial treatment of 2 ppm would be required (Table 1), but to obtain 100% mortality of confused flour beetles, a dose of 9 ppm would be necessary. The data also indicate the residue that is required on the grain for the effective control of the different insects. The order of tolerance to the killing action of the residues was confused flour > lesser grain borers > red flour beetles > *Sitophilus* spp.

REFERENCES CITED

LaHue, D. W. 1977. Pirimiphos-methyl: gradient of effective doses on hard winter wheat against attack of four species of adult insects. J. Econ. Entomol. 70: 295–7.

Storrherr, R. W., M. E. Getz, R. R. Watts, S. J. Friedman, F. Erwin, L. Guilffrida, and F. Ives. 1964. Identification and analysis of five organophosphorus pesticides: recoveries from crops fortified at different levels. J. Assoc. Off. Agric. Chem. 47: 1087–93.