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### Pirimiphos-methyl as a Protectant for High Moisture Stored Wheat<sup>1</sup>

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**ABSTRACT:** Throughout a 9-month period pirimiphos-methyl (*O*-[2-(diethylamino)-6-methyl-4-pyrimidinyl]*O,O*-dimethyl phosphorothioate) was more effective than malathion (*O,O*-dimethyl phosphorodithioate of diethyl mercaptosuccinate) in controlling insects on 14.6% moisture hard winter wheat stored in plywood bins. Pirimiphos-methyl persisted longer on the wheat than malathion did. Also in laboratory studies, malathion degraded faster than pirimiphos-methyl. The higher the grain moisture, in the laboratory studies the greater the breakdown of both insecticides.

Several laboratory and small-bin studies have indicated that pirimiphos-methyl (PM)-(*O*-[2-(diethylamino)-6-methyl-4-pyrimidinyl]*O,O*-dimethyl phosphorothioate) at a dosage of from 2.0 to 10.0 ppm is an effective insect protectant for stored wheat of normal moisture levels (LaHue, 1974, 1975, 1976a, b, 1977; Bengston et al., 1975; Coulen and Barres, 1975). However, malathion (*O,O*-dimethyl phosphorodithioate of diethyl mercaptosuccinate) degrades at high moisture (Kadoum and LaHue, 1979; Fam, 1974; Minnet et al., 1968; Strong and Sbur, 1960; Waters, 1959). Also, field studies have not been conducted with PM. We therefore tested PM and malathion against several stored-grain insects for their persistence in high moisture (14.6%) wheat. Laboratory studies were also conducted with wheat of different moisture contents.

#### Materials and Methods

Field tests were conducted with eight 1.5-metric ton lots of cleaned untreated, hard winter wheat (Chanute variety) containing small amounts foreign material and cracked kernels (<0.1% of weight). The wheat was held

<sup>1</sup> 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 the USDA.

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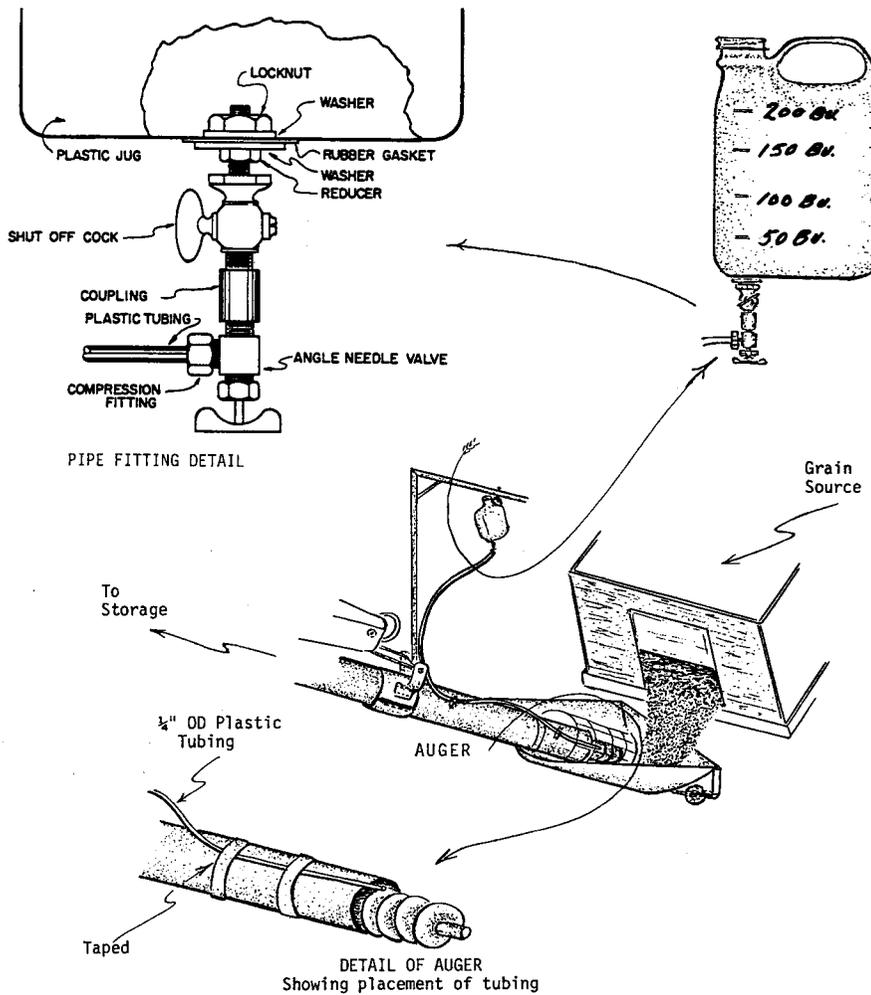


Fig. 1. Schematic drawing of the gravity feed system used to apply the grain protectants. The "pipe fitting detail" in the upper left hand corner taken from Quinlan et al., 1979.

at ca. 24.4°C, treated, and augered into eight 3.84 m<sup>3</sup> bins (2.4 × 1.2 × 1.2 m) constructed of 1.9-cm plywood. Moisture content of the wheat averaged 14.6%. The exterior of each bin was painted white. Bins were covered with a galvanized sheet-metal roof and exposed to the elements. Insecticides (added to water as emulsifiable concentrates) were applied the 1st week of June by the liquid gravity-feed system (Fig. 1) described by Quinlan et al. (1979). The rate was 704 ml/metric ton. Control wheat received a water treatment at the same rate.

The pirimiphos-methyl formulation, designated Actellic<sup>®</sup>, contained 597 g ai/liter. The malathion was the standard 57% product (labeled for grain use) and contained 597 g ai/liter.

Two weeks after the wheat was treated and placed in storage, each bin was infested with 1000 adults each of the rice weevil, *Sitophilus oryzae* (L.), red flour beetle, *Tribolium castaneum* (Herbst), lesser grain borer, *Rhyzopertha dominica* (F.) and sawtoothed grain beetle, *Oryzaephilus surinamensis* (L.). The insects were poured evenly through a funnel and hollow pipe that was raised gradually out of the center of the grain from the bottom of the bin to the grain surface.

Samples of 500 g of wheat were taken from the truck before treatment, samples from the bins (monthly after treatment) were removed from the center of the grain mass with a 1.52-m partitioned grain trier and placed in plastic-lined bags for transportation to the laboratory for examination. There the wheat was sifted with an 8-mesh screen (2380  $\mu$ ), and the numbers of living insects were determined. The 2 fractions were then recombined, and the moisture was determined with a Motomco<sup>®</sup> Model No. 919 moisture meter. Then the sample was divided with a Boerner<sup>®</sup> sampler and used for bioassay and residue analyses at 3-month intervals.

The bioassay test insects were adults (mixed sexes) of the rice weevil (14–21 days old) and lesser grain borer (7–14 days old), reared on hard red winter wheat of 12.5% moisture and red flour beetles (14–21 days old) reared in a medium of flour, cracked wheat and brewer's yeast (10:50:1.5 parts by volume, respectively). Fifty insects were placed on 150 g of the test wheat in a 473-ml jar fitted with an open ring, 40-mesh screen, and filter paper lid for one week. Then the adults were removed, the live and dead insects were counted, and the jars of wheat were retained for 53 days so we could determine mortality of the progeny. During the exposure and incubation, jars were stored in the insect rearing room at 26.7°C and 60% RH.

A 250 g portion of each sample was placed in a sealed jar and kept frozen (–17°C) until it could be extracted and analyzed for insecticidal residues.

Residues of pirimiphos-methyl and malathion were extracted from wheat and cleaned up for analysis in a Tracor<sup>®</sup> M.T. gas chromatograph by a procedure adapted by L. I. Davidson from that of Storrherr et al. (1964). For details of this method see Quinlan et al., 1979.

**LABORATORY STUDIES:** Laboratory studies were conducted with hard winter wheat (Chanute variety) that was screened and cleaned to remove broken kernels, dust, and foreign material. The wheat was weighed, and 975 g were placed in each 3785-ml wide mouth glass jar along with 25 g of cracked wheat.

The wheat was originally 10% moisture content and lots were tempered to 12, 14, and 16% moisture with distilled water. Each treatment was replicated twice.

Table 1. Total number of living insects found in 500-g samples of wheat treated with pirimiphos-methyl (PM) and malathion (MAL) or untreated (controls).

Insecticide	Amount (ppm)	Mean no. insects/1000 g wheat									
		June <sup>a</sup>	July <sup>b</sup>	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar
PM	7.8	0	0	0	0	0	0	0	0	0	0
MAL	10.4	0	0	0	0.7	4.7	103	188	106	84	64
Controls	0.0	0	0	36	147	383	567	462	5	2	2

<sup>a</sup> Immediately after treatment.

<sup>b</sup> One month after treatment.

The same emulsifiable concentrate used in the field tests were diluted with distilled water and applied at a rate of 1 ml/kg to deliver 7.5 ppm of active ingredient to the wheat in each jar. The applications were made with a 1-ml volumetric pipette to the inside walls of the glass jars above the grain level while the jars were turning on a 33-rpm turntable. Three air-tight lids were placed on the jars, and the jars were then shaken by hand for 30 sec. Next they were rolled on a horizontal roller for 30 sec and then placed on a mechanical tumbler for 15 minutes, which allowed the grain to mix end over end in the jars. The jars were closed and stored in the already mentioned insect rearing room. Each treatment was repeated 3 times. After the required aging period the sealed lids were removed, and the grain in each jar was divided into 10 samples (100 g) with an electric grain divider (Dean Gamet® "Precision Divider"). One 100-g sample was used for chemical analysis, and the remaining samples were returned to the jar. The chemical samples were placed in sealed jars and stored as before at  $-17^{\circ}\text{C}$  until the analysis could be conducted.

### Results and Discussion

**FIELD STUDIES:** In the field tests, the moisture of the grain at the beginning of the study was about the same in all bins, and it remained constant throughout the storage period in all treatments. The average moisture was ca. 14.6% before treatment and averaged ca. 14.4% when the study was terminated.

No live insects were found in any of the wheat samples before treatment, during the June and July sampling (immediately after treatment), or in the pirimiphos-methyl-treated wheat throughout the storage period (Table 1). Insects were found in the control wheat during August, and the number increased each month until December, but from January to March, only a few insects were found. In the malathion-treated wheat, the first insects were observed during September, and the numbers increased until December and then generally declined through March. However, the considerable frass and insect-damaged grain in the controls during October and increasing

Table 2. Bioassay mortality of rice weevil (RW), lesser grain borer (LGB), and red flour beetle (RFB) exposed to samples of wheat treated with pirimiphos-methyl (PM), malathion (MAL), or untreated (controls) (means—3 bins).

Treatment	% Mortality of insects at indicated time <sup>a</sup>																	
	RW						LGB						RFB					
	Before	0	1	2	3	6	Before	0	1	2	3	6	Before	0	1	2	3	6
PM	4.4	100	100	100	100	100	3.4	96	30	11	6	26	0	100	100	100	100	99
MAL	0.7	100	100	80	7	21	2.7	92	21	13	6	16	0.7	100	87	26	5	2
Controls	0.9	2	0	0	1	6	1	1	0	4	13	7	0	2	1	2	0	5

<sup>a</sup> Immediately after treatment; 1, 2, 3, and 6 refer to months posttreatment.

Table 3. F<sub>1</sub> progeny of rice weevil (RW), lesser grain borer (LGB), and red flour beetle (RFB) exposed to samples of wheat treated with pirimiphos-methyl (PM), malathion (MAL), or untreated (controls) (means—3 bins).

Insecticidal treatment	No. F <sub>1</sub> progeny of insects at indicated time <sup>a</sup>																	
	RW						LGB						RFB					
	Before	0	1	2	3	6	Before	0	1	2	3	6	Before	0	1	2	3	6
PM	1150 <sup>b</sup>	0	15 <sup>c</sup>	12 <sup>c</sup>	53 <sup>c</sup>	118 <sup>c</sup>	223 <sup>b</sup>	0	7 <sup>c</sup>	7 <sup>c</sup>	3 <sup>b</sup>	3 <sup>c</sup>	71 <sup>b</sup>	0	0	0	0	0
MAL	773 <sup>b</sup>	1 <sup>c</sup>	90 <sup>d</sup>	746 <sup>b</sup>	1040 <sup>b</sup>	752 <sup>b</sup>	216 <sup>b</sup>	1 <sup>c</sup>	7 <sup>b</sup>	10 <sup>b</sup>	13 <sup>b</sup>	18 <sup>b</sup>	81 <sup>b</sup>	0	0	0	0	51 <sup>b</sup>
Controls	1252 <sup>b</sup>	980 <sup>b</sup>	1440 <sup>b</sup>	1740 <sup>b</sup>	1150 <sup>b</sup>	848 <sup>b</sup>	175 <sup>b</sup>	222 <sup>b</sup>	290 <sup>b</sup>	381 <sup>b</sup>	125 <sup>b</sup>	94 <sup>b</sup>	77 <sup>b</sup>	34 <sup>b</sup>	81 <sup>b</sup>	86 <sup>b</sup>	180 <sup>b</sup>	300 <sup>b</sup>

<sup>a</sup> Immediately after treatment; 1, 2, 3, and 6 refer to months posttreatment.

<sup>b</sup> Majority of adults alive.

<sup>c</sup> All adults dead.

<sup>d</sup> Majority of adult insects dead.

Table 4. Residues of pirimiphos-methyl (PM) and malathion (MAL) on wheat at various intervals after treatment (means of 3 bins).

Treatment	Calculated dosage applied (ppm)	Residues (ppm) at indicated time							
		1 day	5 days	12 days	1 month	2 months	3 months	6 months	9 months
PM	7.8	3.2	3.7	—	3.2	2.9	3.2	2.6	3.0
MAL	10.4	8.6	4.1	2.6	2.1	2.0	1.6	0.9	0.5

through the remainder of the storage period, was not seen in the malathion-treated grain. It is not known why the insects in the control wheat decreased suddenly and remained low from January to March nor why the populations in the malathion-treated wheat dropped gradually during the same period.

The predominant species of insects found in the control and the malathion-treated wheat, were the lesser grain borer and the rice weevil; each comprised ca. 40% of the insect populations. The red flour beetle and the rusty grain beetle, *Cryptolestes ferrugineus* (Stephens), each comprised ca. 9.0% of the population in the controls and ca. 10% of the population in the malathion-treated grain. (The presence of the rusty grain beetle is interesting since none of that species was placed in the grain.) The sawtoothed grain beetle comprised ca. 2% of the insect population in the controls but was not found in malathion-treated wheat.

Tables 2 and 3 summarize results of the bioassays. High adult mortality (Table 2) was recorded in all the PM-treated grain: all the rice weevils were killed as were all the red flour beetles except during the last 6 months when mortality was 99%. The lesser grain borer adult was much harder to kill and some had survived at each interval. Grain treated with malathion produced lower mortalities, and again the lesser grain borer adults were harder to kill. Data for  $F_1$  progeny are reported in Table 3. Few were recovered from the PM-treated wheat and all emerged adults were dead. The malathion-treated grain had more developing progeny, and most of the progeny were alive. The controls, meanwhile, had a low level of mortality, the number of  $F_1$  progeny was consistently high.

The recovery of pirimiphos-methyl immediately after treatment (Table 4) averaged 41% of the amount applied; 9 months later, ca. 38% was recovered; in contrast, the immediate recovery of malathion averaged 82% of the amount applied; 9 months later 6% was recovered.

Average wheat temperatures taken at the center of each bin (Table 5) showed heating in the malathion-treated bins during the 4th month and in the control grain during the 2nd month. The malathion-treated grain continued to heat until the end of the study; the control grain cooled at the 6th month.

The field study was such that the insecticides applied to grain were tested

Table 5. Average temperature (°C) of wheat (center of bin) treated with pirimiphos-methyl (PM), malathion (MAL), untreated (controls).

Treatment	Interval after treatment (Mo.) <sup>a</sup>									
	0 <sup>b</sup>	1	2	3	4	5	6	7	8	9
PM	24.4	27.7	25.6	25.6	20.0	11.7	5.6	-1.1	5.0	7.2
MAL	25.6	27.7	25.0	25.6	23.9	21.7	25.6	4.4	19.4	21.1
Controls	28.9	27.7	28.3	32.8	24.4	15.6	5.6	1.3	5.0	7.2

<sup>a</sup> Months are 0 = June 1 through 9 = March 1.

<sup>b</sup> Immediately after treatment.

under severe conditions; together the high moisture of the grain, the long, warm storage period (June to September), and the heavy artificial infestation of the grain after treatment accounted for the very high populations in the untreated grain. Nevertheless, PM was stable in the moist wheat and thus effective in protecting it from insect attack.

The results of the laboratory tests are shown in Table 6. The residues of both malathion and PM dropped as the moisture content of the grain increased, but the malathion residues were lower than the residues of PM. As noted previously this rapid degradation of malathion has been reported by other workers and explains, at least in part, the lesser effectiveness of malathion in the field study.

#### Acknowledgments

Gailen D. White (retired) of this laboratory initiated the field studies and modified the gravity feed system after a commercial model. Residue analyses were conducted by Loren I. Davidson. Joseph L. Wilson of this laboratory performed the treating, sampling, insect examinations and bioassays. The auger detail Fig. 1 was drawn by Hobart P. Boles, Research Entomologist of this laboratory.

Table 6. Malathion and pirimiphos-methyl residues (ppm) at 4 moisture levels after insecticides applied to wheat at 7.5 ppm. Wheat was stored in sealed jars at 26.6°C (3 replicates).

Interval after treatment	Malathion residues at indicated moisture content (%)				Pirimiphos-methyl residues at indicated moisture content (%)			
	10	12	14	16	10	12	14	16
24 hours	6.2	6.1	5.7	5.0	5.2	4.9	4.7	5.1
6 weeks	6.4	4.9	2.0	0.4	4.8	4.6	4.7	2.9
3 months	4.6	4.2	1.1	0.2	4.3	4.2	4.3	1.8
6 months	2.8	2.6	0.3	0.1	4.1	3.9	4.0	1.2
9 months	2.7	2.5	0.3	<0.1	3.9	3.9	3.5	0.9

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