Field Tests with Pirimiphos-Methyl as a Protectant for Farmers Stock Peanuts[1]

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ABSTRACT

Farmers stock peanuts were treated with pirimiphosmethyl (0- [2- (diethylamino) -6-methyl-4-pyrimidinyl 0,0-dimethyl phosphorothioate) applied at rates of 10, 20, and 30 ppm as a protectant against stored-product insects and stored in metal bins (4.5 metric tons) for 1 year. Similar peanuts were treated with malathion (diethyl mercaptosuccinate S-ester with 0, 0-dimethyl phosphorodithioate) applied at a rate of 52.1 ppm as a standard for comparison. Although residues of pirimiphos-methyl decreased ca. 63% during the year (half of the decrease during the first 4 months), rates of 20 and 30 ppm gave excellent protection for 1 year, and a rate of 10 ppm gave protection for about 6 months. Malathion was relatively ineffective, either because it degraded so rapidly the first 2 months or because malathion-resistant strains of insects were present.

Of the 16 species of stored-product insects found in the peanuts, red flour beetles, **Tribolium castaneum** (Herbst), and almond moths, **Cadra cautella** (Walker), were the predominant species.

Additional index words: pirimiphos-methyl, protectant, peanuts, insecticide residues, insect damage, stored-product insects, malathion-resistant insects.

Peanuts are subject to damage and contamination by stored-product insects from the time they are harvested until they are utilized or consumed. Nevertheless the warehouses used for peanut storage are often of such design and construction that adequate insect control is difficult or impossible. Thus, for many years, insecticides have been applied as protectants to farmers stock peanuts going into storage. Malathion (diethyl mercaptosuccinate *S*-ester with *O,O*-dimethyl phosphorodithioate) gained wide acceptance for this use after experiments in 1960-61 showed its effectiveness (6). However, insect resistance to malathion was soon discovered, and in recent years it became widespread and severe (9, 10, 12, 13). The occurrence of this resistance has made it necessary to develop new and improved protectants to prevent peanut losses due to insects.

In simulated field tests, pirimiphos-methyl (*O*-[2-(diethylamino) -6-methyl -4-pyrimidinyl] *O,O*-dimethyl phosphorothioate) showed promise as a protectant for farmers stock peanuts (7). This compound is an organophosphorus insecticide that has both vapor and contact toxicity, it is effective against a broad spectrum of insect species including strains that are resistant to malathion (11), and it has low mammalian toxicity (acute oral LD_{50} to rats at 2050 mg/kg) and so has the potential of being safe for use with peanuts. For these reasons, the efficacy demonstrated in the simulated field tests led to field testing of pirimiphos-methyl. The research reported here was conducted at the Peanut Insect Investigations Research Laboratory, ARS, USDA 3/which was located at the Coastal Plain Experiment Station, Tifton, Ga., during 1972-73.

Materials and Methods

The cylindrical, 4.5-ton (metric) capacity metal bins equipped with thermocouples and aeration systems described by Redlinger and Womack (8) were used in the study. Approximately 90 tons (metric) of 'Florunner' segregation I, farmers stock peanuts, were acquired at harvest and loaded into the bins with a portable belt conveyor. There they were treated with insecticide sprays from a flat spray nozzle (Spraying Systems No. 5001) attached to the top of the conveyor. The sprayer consisted of an electrically driven gear pump equipped with an adjustable pressure regulator calibrated to deliver the required amount of insecticide on the peanuts as they free fell into the bins.

Either pirimiphos-methyl or malathion was applied. The pirimiphos-methyl sprays were prepared from a 60% emulsifiable concentrate diluted in water to produce calculated deposits of 10, 20, or 30 ppm on the peanuts when applied at a rate of 1.39 liters/metric ton (5 gal/15 tons). The malathion spray, a standard treatment for comparison, was prepared from an emulsifiable concentrate containing 57% premium-grade malathion diluted in water to give a calculated deposit of 52.1 ppm. Each insecticide and rate and the untreated controls were replicated 4 times (4 bins) in a randomized complete block experimental design.

A thermocouple system was used to record the temperature of the peanuts each month for the first 7 months and then at 9 and 12 months. Conditions in the storage bins were maintained with an aeration system, controlled by a thermostat and a humidistat wired in series and set to operate at 13°C and 65% RH.

Insects from laboratory-reared cultures were used. About 250 2-wk-old adults of the red flour beetle (**Tribolium castaneum** (Herbst)) and the merchant grain beetle (**Oryzaephilus mercator** (Fauvel)) were released during loading operation. Also ca. 1000 eggs each of the Indian meal moth (**Plodia interpunctella** (Hubner)) and the almond moth (**Cadra cautella** (Walker)) were scattered uniformly over the surface of the peanuts after each bin was filled and the contents were leveled.

Biological efficacy of the treatments was evaluated by determining the number of live and dead insects of each species in samples collected from each bin and by assess-

[1]This paper reports the results of research only. Mention of a pesticide in this paper does not constitute a recommendation for use by the U.S. Department of Agriculture nor does it imply registration under FIFRA as amended. Also, mention of a commercial or a proprietary product in this paper does not constitute an endorsement for use by the U.S. Department of Agriculture.

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ing the percentage of insect damage to loose-shelled and to inshell kernels in the samples. The persistence of insecticide residues was monitored by chemical analysis of samples of peanuts from each bin. Sampling was done within 9 days post treatment for residue analysis and then at 2, 4, 6, 9, and 12 months for residue analysis and for the biological assessments. Ports for obtaining peanut samples horizontally across the bins were located as follows: surface (0 to 10 cm deep), 30 cm below the surface, 1/2 the distance between surface and floor, and 30 cm above the floor. A 3-m (10-ft) peanut trier was used to collect about 2.5 kg of peanuts from each port. This trier was inserted 4 to 6 times at different angles so that each sample was representative of the full cross section of the bin. In addition, a full depth vertical sample was obtained by inserting the trier down through the top of the peanuts.

Each 2.5-kg sample was reduced with a peanut divider into a 325-g subsample for residue analysis and a 1000-g subsample for the biological assessments. The remaining material was shelled, and the moisture content of the shelled nuts was determined with a Steinlite model PT-2 moisture meter.

The 325-g subsamples for residue analyses were sealed in 1-liter glass jars and stored in a freezer at ca. -18°C until analyzed. A Hewlett-Packard series 5750 gas chromatograph equipped with a Tracor flame photometric detector in the phosphorous mode (interference filter 526 nm), a HP 7670A automatic sampler, and a HP 3370B electronic integrator were used for the residue analysis. The glass column used was 122 cm long, 6 mm O.D., and 4mm I.D.; it was packed with 8% OV 101 (methyl silicone) and 2% HI-EFF 8AP (cyclohexanedimenthanol adipate) on 60/80-mesh Gas Chrom Q. The gas flow rates were: nitrogen (carrier), 35 cc/min; hydrogen, 20 cc/min; and oxygen, 40 cc/min. The temperatures were: column oven, 240°C; injection port, 320°C; and detector, 200°C. The detector was operated at 750 V. The retention time at these conditions was 3.25 min for pirimiphos-methyl and 5.44 min for malathion. Sub-samples were prepared for extraction by grinding in a Waring Blendor and tumbling the ground peanuts in the 1-liter glass jars for 2 h. Then 40 g of ground peanuts were weighed into a 250-ml Erlenmeyer flask, 120 ml of reagent grade acetone were added, and the flask was shaken on a wrist-action shaker for 3 h. The solution was filtered through Whatman 2v filter paper into 2ml glass vials; each vial was fitted with a rubber septum and sealed with an aluminum cap; and the sample vials were placed on the automatic sampler turntable where 10 μliters from each vial were automatically injected into the gas chromatograph for analysis. Every third vial contained an analytical standard of known concentration. Quantitation was accomplished by comparison of the integration counts obtained for the standards with those obtained for the samples.

The 1000-g subsamples of farmers stock peanuts to be used for biological assessments were handled and examined as described by Redlinger (7).

Lots used to assess insect-damaged kernels were either examined immediately after collection or stabilized for later examination by storing in moistureproof bags in a freezer ca. -18°C. The examination for insect damage of loose- and in-shell kernels was conducted as described by Redlinger (7).

Data for the number of live insects and damaged kernels from treatments were evaluated statistically by analysis of variance, and the means were compared using Duncan's (2) new multiple range test. Conclusions presented are based on the results of these analyses.

Results and Discussion

TEMPERATURE

There was little variation of peanut temperatures between replicates or type of treatments and it is therefore possible to compare results from both treated and control bins. Tabular data of temperatures have been omitted. Peanut temperatures, as recorded, ranged from 1° to 7°C above the average ambient temperature during the 12-month period. Apparently the aeration system, which operated intermittently for a total of 287 hours from November to January, had little or no overall influence on the temperature of the peanuts. In fact, the greatest decrease (10°C) occurred in October, prior to aeration. All other temperature changes were gradual and ranged from 1° to 5°C per month. The average temperature of the peanuts decreased from 29°C in September when the test was started to a low of 13°C in February. Then there was a gradual increase. By June the average temperature was 29°C. In September, when the test was terminated, the temperature of the peanuts was 27°C.

PEANUT MOISTURE

At the beginning of storage, the moisture content of the peanuts ranged from 8 to 10% (a mean of 8.5%) for all bins. During the first 2 months of storage, it decreased rapidly to an average 6.6%. This rapid decrease occurred during the period the low temperature was recorded (October). During the remainder of the storage, the moisture content ranged from 5.9 to 6.7%, a mean of 6.3%. Since there were only minor variations in moisture content between replicates, treatments, or sampling locations within bins, moisture content of the peanuts was considered a minor factor in the overall performance of the insecticides.

RESIDUES

Chemical analysis of the farmers stock peanuts for residues of pirimiphos-methyl at ca. 3 days after treatment showed that the amounts applied were near the intended levels (Table 1). However, the initial residue of malathion was lower than expected though it appeared adequate to protect the peanuts. This difference may reflect the fact that malathion was applied first and that sampling for residues of malathion was not done

Table 1. Residues of insecticides found on farmers stock peanuts at various times after treatment (average of 5 samples from each of 4 replicates).

Sampling period (months after treatment)	Residue (ppm) ±SE from application of			
	Pirimiphos-methyl			Malathion
	10 ppm	20 ppm	30 ppm	52 ppm
0.1	9.3±0.17	20.7±0.85	34.7±4.20	1/35.8±6.40
2	3.0±0.29	5.8±0.55	10.9±0.52	10.1±1.97
4	4.8±0.62	10.5±0.80	16.6±1.01	11.3±1.94
6	4.0±0.22	10.5±0.34	13.2±0.57	11.6±1.89
9	4.0±0.31	9.9±1.10	13.6±2.27	4.5±1.35
12	2.9±0.29	11.0±2.61	9.5±1.38	3.0±0.45

1/ Initial samples for malathion residue analyses were collected 9 days after the peanuts were treated.

until 9 days after treatment. Also, malathion generally degrades very rapidly during the first month after application to peanuts (7).

The residue of both insecticides decreased sharply after 2 months of storage, at this time when the temperature and the moisture content of the peanuts dropped. Nevertheless, these conditions probably did not cause the decreased residues, especially since somewhat higher residues were found at subsequent sampling periods.

As the test progressed, residues of each insecticide, though they fluctuated somewhat between sampling periods, showed a general trend of degradation. After the 12 months of storage, the malathion residues had decreased to 3.0 ppm about 6% of the initial deposit, which compares favorably with results obtained in simulated field tests (7). The situation with pirimiphos-methyl was quite different though, the greatest decrease did occur during the first few months. For example, at the 4-month sampling period, residues were 4.8, 10.5, and 16.6 ppm, respectively, for the 3 rates, showing that 48 to 52% of the initial deposit of each dosage applied was still present on the peanuts. Even at 9 months there was little or no additional degradation of residues. After the full 12 months of storage, average residues found on the peanuts were 2.9, 11.0 and 9.5 ppm, respectively, for the 10, 20 and 30 ppm dosage rates.

Table 2. Number of insects found per 1000-g sample of farmers stock peanuts at each sampling period after treatment (average of 5 samples from each of 4 replicates).

Sampling period (months after treatment)	Insects (no.) from bins treated with 1/				
	Pirimiphos-methyl			Malathion	Control
	10 ppm	20 ppm	30 ppm	52 ppm	
	Alive				
2	0.6^{a}	0.1^{a}	0^{a}	449.1^{b}	198.2^{c}
4	3.0^{a}	0.2^{a}	0^{a}	155.5^{b}	29.0^{c}
6	0.7^{a}	0^{a}	0.1^{a}	123.4^{b}	36.9^{b}
9	44.0^{a}	0.1^{a}	0.7^{a}	132.1^{b}	148.5^{b}
12	20.0^{a}	1.8^{a}	6.3^{a}	211.5^{b}	201.6^{b}
	Dead				
2	8.2^{b}	1.7^{a}	7.1^{b}	41.6^{c}	13.9^{bc}
4	7.7^{bc}	1.4^{a}	6.1^{ab}	56.2^{d}	17.0^{c}
6	7.9^{b}	2.1^{a}	4.5^{ab}	48.2^{d}	17.4^{c}
9	29.1^{cd}	7.2^{a}	10.6^{abc}	24.5^{bcd}	31.4^{d}
12	29.5^{abc}	15.6^{ab}	27.6^{bc}	117.1^{d}	66.0^{c}

1/ Means in the same line followed by different letters are significantly different at the 1% level of probability using Duncan's new multiple range test.

INSECT INFESTATION

Sixteen species of stored-product insects were found in the peanuts. As expected, the four species released at the time of storage were among the most abundant. Almond and Indian meal moths were predominant during the first 6 months, and the red flour beetle was predominant at the 9- and 12- month sampling periods. The other species present, in order of decreasing abundance, were: corn sap beetle, *Carpophilus dimidiatus* (F); long-headed flour beetle, *Latheticus oryzae* Waterhouse; hairy fungus beetle, *Typhaea stercorea* (L.); cigarette beetle, *Lasioderma serricorne* (F.); flat grain beetle, *Cryptolestes pusillus* (Schonherr); foreign grain beetle, *Ahasverus advena* (Waltl); cadelle, *Tenebroides mauritanicus* (L.); dersmestids (species not determined); coffee bean weevil, *Araecerus fasciculatus* (De Geer); lesser grain borer, *Rhyzopertha dominica* (F.); maize weevil, *Sitophilus zeamaiz* Motschulsky; and two-banded fungus beetle, *Alphitophagus bifasciatus* (Say).

The buildup of insect populations in both the untreated peanuts and in the malathion-treated peanuts was rapid and generally continuous throughout the storage period (Table 2), though the population did decline slightly during the winter months. Moreover, the relatively large numbers of insects (especially almond and Indian meal moths and red flour beetles) found in the malathion-treated peanuts indicated that the populations in these peanuts included native insects that unlike the susceptible laboratory strains, were somewhat resistant to malathion. These 3 species have been shown by researchers to have considerable resistance to malathion (9, 10, 12, 13). Another indication of the presence of resistant insects was provided by the comparison of the ratio-of live to dead insects found in the malathion-treated with the ratio in the control peanuts. Since these ratios were ca. the same at any particular sampling period, most of the dead insects in the malathion-treated peanuts seemed to result from natural mortality. However, the situation was somewhat obscured by the development of large numbers of parasites and predators (*Bracon hebetor* Say, *Xylocoris flavipes* (Reuter), mites, psocids, and spiders) in the untreated peanuts. The numbers observed in the control bins were not tabulated and only a small percentage of those observed were collected in the samples of peanuts. Such populations were unable to become established in any treated peanuts, but they apparently effected some control of insects in the untreated peanuts and may have affected the ratio of live to dead insects there. One may hypothesize that malathion was therefore actually detrimental, because parasites and predators in malathion-treated peanuts were unable to develop as they did in the untreated peanuts. During the first 4 months after treatment the number of live insects in the control bins were significantly different from the malathion bins at the 1% level and at 6 months at the 5% level. The red flour beetle was the predominant insect at the 9- and 12-month sampling and perhaps predators were unable to hold them in check. The importance of

Xylocoris flavipes (Reuter) and *Bracon hebetor* Say in suppressing stored-product insects has been demonstrated by several researchers (1, 3, 4, 5).

Data for the number of live insects were evaluated statisticially by analysis of variance, and means were compared using Duncan's new multiple range test. The maximum F ratio test showed variance was heterogeneous so data were transformed [log (x+1)] before running analysis. However, data presented in Table 2 are means of actual numbers of insects.

Each of the pirimiphos-methyl treatments provided a high degree of protection against insect infestation of the peanuts. Even the 10 ppm treatment, which was less effective after 9 months was significantly more effective during the 12 months than the malathion standard. Relatively few live or dead insects were found in peanuts treated with either 20 or 30 ppm pirimiphos-methyl. Both rates were about equally effective and were highly effective as a protectant throughout the entire storage period. Indeed, the low numbers of total insects indicate that the pirimiphos-methyl treatments were repellent to the insects; a "free-choice" repellency noted by Redlinger (7) in the simulated field tests. The ratio of numbers of insects alive to numbers dead in the peanuts treated with pirimiphos-methyl shows that a high percentage of the insects found were killed by the treatments. Then, if these peanuts were invaded by malathion-resistant insects, as were those treated with malathion, the insects had little or no cross resistance to pirimiphos-methyl. Zettler (11) too found that malathion-resistant strains of the Indian meal moth showed no cross-resistance to pirimiphos-methyl and in recent studies (unpublished data) found no cross-resistance in malathion-resistant strains of the red flour beetle. These 2 species were among the 3 species of insects that were most abundant in the malathion-treated peanuts. Therefore, one may conclude that pirimiphos-methyl applied at a rate of 20 ppm to farmers stock peanuts gave adequate protection against infestation by both susceptible and malathion-resistant insects.

INSECT DAMAGE

Insect damage to loose-shelled kernels increased gradually during the 12-month storage period regardless of the treatment (Table 3), and the percentage of damage corresponded to the total number of insects found. The data were subjected to an analysis of variance and significant differences were determined by Duncan's new multiple range test. There was a high significant difference (1% level) between damaged kernels from pirimiphos-methyl-treated peanuts and the standard malathion treatment. However, no difference was shown between the 3 pirimiphos-methyl rates of application. These data further demonstrated the level of insect control afforded by the pirimiphos-methyl treatments.

There was much less insect damage to inshell kernels (Table 3) than to loose-shelled kernels, but it followed the same trends: Peanuts treated with pirimiphos-methyl had 1.4% or less damaged kernels after 12 months; while the malathion-treated peanuts and the untreated control had 7.6 and 8.7% damaged kernels, respectively. However, these findings also emphasize dramatically the importance of the peanut shell in protecting kernels against insects. For example, 41.5% of the loose-shelled and only 8.7% of the inshell kernels in samples from the control bins were damaged by insects. Plainly the producer and the warehouseman should minimize damage to the peanut pods during harvesting, handling, and storage.

Table 3. Insect damage to peanut kernels at various times during storage after pirimiphos-methyl or malathion was applied as protectants to farmers stock peanuts (average of 3 samples from each of 4 replicates).

Sampling period (months after treatment)	Insect-damaged kernels (%) after treatment with 1/				
	Pirimiphos-methyl			Malathion	Control
	10 ppm	20 ppm	30 ppm	52 ppm	
	Loose-shelled kernels				
2	4.7[a]	4.0[a]	3.9[a]	16.2[b]	9.2[ab]
4	6.0[a]	4.9[a]	5.1[a]	32.0[b]	16.8[c]
6	6.4[a]	5.8[a]	5.6[a]	34.0[b]	17.4[a]
9	10.8[a]	9.1[a]	7.5[a]	40.7[b]	31.1[b]
12	16.9[a]	14.5[a]	9.7[a]	43.4[b]	41.5[b]
	Inshell kernels				
2	0.6[a]	0.5[a]	0.5[a]	2.4[b]	2.1[b]
4	0.5[a]	0.6[a]	0.7[a]	3.4[b]	2.3[b]
6	0.8[a]	0.7[a]	0.7[a]	5.2[b]	3.1[b]
9	1.0[a]	0.9[a]	0.6[a]	5.9[b]	5.3[b]
12	1.4[a]	1.1[a]	0.9[a]	7.6[b]	8.7[b]

1/ Means in the same line followed by different letters are significantly different at the 1% level using Duncan's new multiple range test.

Summary

Pirimiphos-menthyl applied as a spray at rates of 20 and 30 ppm gave excellent protection against infestation by several species of stored-product insects to farmers stock peanuts during a 1-year storage period. A dose of 10 ppm provided good protection for about 6 months. When applied at the recommended dosage, malathion was relatively ineffective as a protectant, either because it degraded so rapidly or because some of the insect species were resistant to malathion.

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