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Project 87-S8 - Tree and Crop Research
Fumigation, Sealing and Concealed Damage Studies

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Interpretive Summary: Under objective 1, fumigation tests are being conducted using a constant dosage of 30 g/1000 ft^3, derived from the equivalent of 150 aluminum phosphide pellets/1000 ft^3. This dosage is applied to 0-24 hours old navel orangeworm (NOW) eggs that are placed amongst inshell almonds in 28 L fumigation chambers.

Three chambers were prepared as stated and held at 70°F and three at 90°F. At the end of predetermined time periods, a chamber at each temperature was aerated and mortality of NOW eggs was determined. At 70°F, mortality after 48 hours was 46.4%, at 72 and 96 hours, mortality was 100%. At 90°F, mortality after 24 hours was 96.3% and for 48 and 72 hours, 100%. Fumigation tests at 70°F for 72 hours and at 90°F for 48 were then replicated 3 times each, resulting in 100% mortality of NOW eggs. The ending phosphine mean concentration for 72 hours at 70°F was 200 ppm. For 90°F, the ending mean concentration after 48 hours was 75 ppm.

Since it seems apparent that the rate of insect mortality from phosphine is more dependent on time and temperature than on dosage, ending concentrations in which mortality is known, should be easily applied to any commercial fumigation condition. Studies to verify the above findings as well as studies at 60° and 80°F will be completed in 1988.

Under objective 2, two organic bromide depletion tests have been completed. Almond nutmeats in shipping cartons without liners and nutmeats as bulk (no packaging material) were fumigated using one of the new methyl bromide

schedules. Each was fumigated in triplicate at 1 LB/1000 ft^3 (16 g/m^3) for 4 hours at 80°F. The nuts were aerated and stored at 80°F. Residue analyses were conducted at predetermined intervals and continued until the nuts reached 1 ppb (0.001 ppm). Nutmeats treated in the carton took 19 days to reach 1 ppb while those treated as bulk took 12 days.

Experimental Procedure for Objective 1: Only the NOW egg stage is being tested since earlier studies have shown this stage is the most resistant to phosphine. A minimum of 400 eggs are being used per fumigation replication. Screen vials containing the eggs are placed amongst the almonds which are contained within the fumigation chambers. The almond load is approximately 53% within the 28L fiberglass chambers. Air circulation is used throughout the exposure and aeration periods. Temperatures are controlled at ± 2.0°F.

In previous studies we attempted to develop concentration mortality data on the naked egg stage (no commodity) using phosphine gas from preanalyzed mixtures in gas cylinders rather than reacting aluminum phospide. The theory was to develop CT products which could be applied to any commercial fumigation condition. However, we found that from a practical standpoint, CT products are not applicable, mainly because the rate of insect mortality from phosphine is more dependent on time and temperature than on concentration. As a consequence, phosphine is now being generated in the normal way from aluminum phosphide pellets with the presence of almonds. Equivalent weights of broken-up pellets to achieve a dosage of 150 pellets per 1000 ft (30g/1000 ft^3) is placed in the chamber load. This is a recommended commercial dosage. Phosphine concentration readings are then taken after the first 2 hours and then after approximately each 24 hour period and at the end of the exposure period. Ultimately, the end result of our present study will be a phosphine concentration obtained at the end of the fumigation exposure period for fumigation temperatures of 60.0, 70.0, 80.0 and 90°F which results in 100% mortality of NOW eggs and should be easily applied to any commercial situation.

Experimental Procedure for Objective 2: All methyl bromide (MB) fumigations will be conducted in 1 ft^3 (28L) fiberglass chambers. Metal open-construction baskets, wooden bins, cardboard cartons and plastic liners will be used so as to simulate bulk and packaged conditions where almonds are fumigated commercially. All fumigations will be replicated 3 times using each of the new MB schedules and one of the old MB schedules.

New Schedules

Temperature °F	Time hours	MB dosage lbs/1000^3 Meats	Inshell
50 (10°C)	12	1.0 (16g/m^3)	1.5 (24g/m^3)
60 (15.6°C)	8	1.0 (16g/m^3)	1.5 (24g/m^3)
80 (26.7°C)	4	1.0 (16g/m^3)	1.5 (24g/m^3)

	Old Schedule	
60 (10°C)	24	3.5 (56g/m^3)

Forced aeration will be applied to the nuts for the same length of time as the fumigation period. Following aeration, the nuts will be placed in temperature-controlled storage rooms at the same temperature as the fumigation and held for residue analysis.

Results for Objective 1: Tables 1 and 2 show the NOW egg mortality results which were obtained at different exposure times with temperatures of 70 and 90°F. At 70°F (Table 1) 100% mortality was achieved after 72 hours exposure and the phosphine concentration was only 15 ppm. At 90°F (Table 2), 100% mortality was obtained after 48 hours and the final phosphine concentration was 153 ppm. When replicated tests were conducted at these dosages, times and temperatures, there was considerable variation in the phosphine concentration at the end of the exposure periods. This variation may be an effect of the almond load, i.e., difference in sorption and desorption rates as influenced by moisture content and possibly by the ratio of nuts with and without shells. Another more apparent cause for this variation may possibly be explained by the differences in pellet particle size used from test to test after pellets are broken up to achieve a certain weight for dosage which may affect the reaction rate during exposure. All of these possible causes will be investigated. More tests will be required to obtain a final reliable phosphine concentration reading at temperatures of 60, 70, 80 and 90°F which can be utilized commercially.

Results for Objective 2: Organic bromide residues in almond meats which were treated as bulk or in packaging cartons at 1 lb/1000 ft^3 for 4 hours at 80°F and then stored at 80°F are shown in Fig. 1. It is obvious that the carton material retains methyl bromide to some extent. As shown, it takes more than 5 days longer for the nuts stored in cartons to reach 1 ppb (0.001 ppm) than did those treated and stored as bulk at the same temperature. Organic bromide studies are presently being conducted on meats fumigated and stored in wooden bins.

Table 1. Phosphine gas concentrations during fumigation and mortality results of navel orangeworm (NOW) eggs, 0-24 h old, treated in a 53% load of inshell almonds with 30g/1000 ft^3 aluminum phosphide (AlP) for 48, or 72 h at 90°F (21°C).

No. of hours treated	Concentration (ppm)						No. of eggs treated	Corrected % mortality[2/]
	2 h[1/]	19.5 h	24 h	48 h	72 h	96 h		
48 h	231.9	582.0	474.0	196.8	-	-	498	46.4
72 h	220.4	437.5	297.4	71.0	15.0	-	455	100.0
96 h	231.9	625.0	537.2	341.0	204.2	126.1	438	100.0
Untreated	-	-	-	-	-	-	436	15.0

[1/] Gas sampling times following AlP introduction.

[2/] Mortality results of treated NOW eggs are corrected for natural mortality that occurred in untreated eggs.

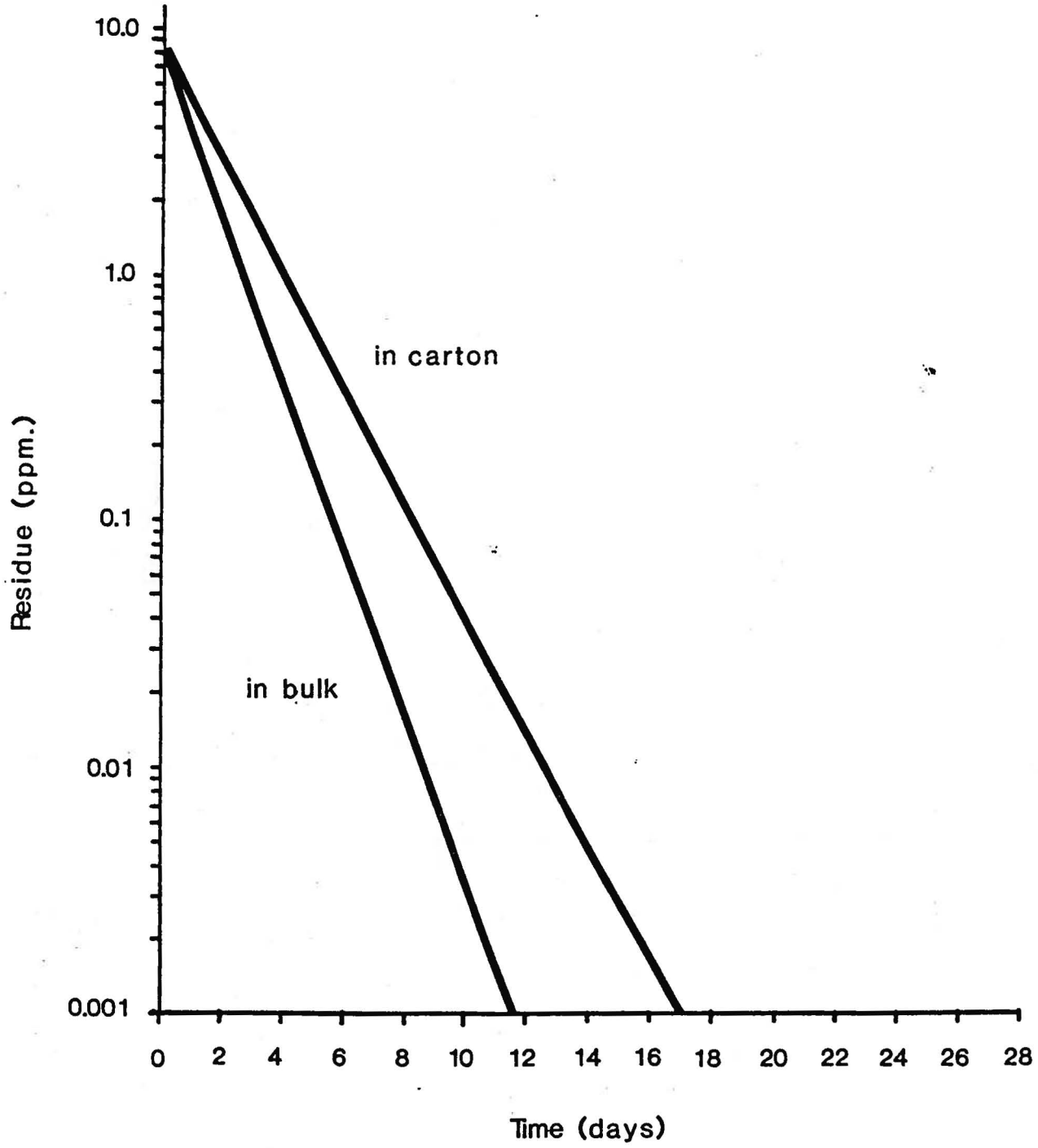
Table 2. Phosphine gas concentrations during fumigation and mortality results of navel orangeworm (NOW) eggs, 0-24 h old, treated in a 53% load of inshell almonds with 30g/1000 ft^3 aluminum phosphide (AlP) for 24, 48, or 72 h at 90°F (32°C).

No. of hours treated	Concentration (ppm)					No. of eggs treated	Corrected % mortality[2/]
	2 h[1/]	19.5 h	24 h	48 h	72 h		
24 h	181.2	417.8	314.7	-	-	436	96.3
48 h	179.4	446.3	332.0	153.4	-	418	100.0
72 h	252.5	325.5	190.0	19.2	12.1	428	100.0
Untreated	-	-	-	-	-	453	16.0

[1/] Gas sampling times following AlP introduction.

[2/] Mortality results of treated NOW eggs are corrected for natural mortality that occurred in untreated eggs.

Fig. 1. Desorption rates of residual methyl bromide from nonpareil almond meats fumigated in bulk or in carton for 4 hours at 80°F with 1 lb./1000 ft.^{3} CH_3BR and stored at 80°F.



Phosphine/Chamber Sealing Material Tests:

Permeability of Materials

Test materials

1. Gaco Western cured neoprene sheet (company sample)
2. Gacoflex neoprene N-110 black
3. Gaco Western UA-6500 white
4. Gacoflex P-5000 white

Fumigation test cells

Fumigations conducted in three aluminum cells, each having an average volume of 1447.9 ml. Cells contructed such that top half (avg. vol. 716.3 ml) is removable from bottom half (avg. vol. 731.6 ml) so that sealing material can be placed between halves to test for permeability to phosphine. Each cell-half fixed with needle port for gas analysis and inflow and outflow valves for PH_3 introduction and aeration.

Test conditions

PH_3 dosage: 300 ppm

Exposure period: 24 h

Avg. exp. temp.: 21°C

Test procedure

Three cells were used to triplicate sealing material permeability tests. Sealing material was cut to fit cell and laid on wire screen (for support) placed on bottom half of cell. Top half of cell was then placed onto bottom half of cell with sealing material secured between the two halves. Cell halves were tightened with a torque wrench to 72 inch pounds. To avoid pressurizing the cell upon introduction of PH_3 gas, a volume of air equal to the volume of 300 ppm PH_3 gas was withdrawn from the upper cell-half, then 300 ppm PH_3 gas was immediately introduced into the upper cell-half. After

PH_3 introduction, gas samples were taken from both upper and lower cell halves for gas analysis to determine starting concentration and to detect any instant penetration of PH_3 through the sealing material. Gas analysis was performed using gas chromatography, flame photometric detector. No PH_3 was ever detected in bottom cell halves at start time of tests. Cells were placed in fume hood for 24 h period; average temperature was 21°C. At the end of 24 hours, gas samples were taken from both cell halves for gas analysis to calculate sorption and to measure the amount of PH_3, if any, that penetrated through the sealing material. Cells were then aerated completely in preparation for next test.

1. Gaco Western cured neoprene sheet (company sample)

Rep	Cell	ppm PH_3 Start	ppm PH_3 24 h	% sorption	ppm PH_3 penetration
1	#1 Top	372.9	344.2	7.6	0.46
	Bottom	NPD	0.46		
2	#2 Top	359.4	331.8	7.6	0.38
	Bottom	NPD	0.38		
3	#3 Top	304.6	293.8	3.4	0.38
	Bottom	NPD	0.38		
$\bar{x}$ ± SD	Top	345.6 ± 36.2	323.3 ± 26.3	6.2 ± 2.4	0.41 ± 0.05
	Bottom	NPD	0.41 ± 0.05		

NPD = No peak detected

2. Gacoflex neoprene N-110 black

Rep	Cell	ppm PH_3 Start	ppm PH_3 24 h	% sorption	ppm PH_3 penetration
1	#1 Top	332.4	324.9	2.2	0.01
	Bottom	NPD	0.01		
2	#2 Top	373.5	337.0	9.8	0.03
	Bottom	NPD	0.03		
3	#3 Top	316.0	295.5	6.5	0.04
	Bottom	NPD	0.04		
$\bar{x}$ ± SD	Top	340.6 ± 29.6	319.1 ± 21.3	6.2 ± 3.8	0.03 ± 0.02
	Bottom	NPD	0.03 ± 0.02		

NPD = No peak detected

3. Gaco Western UA-6500 white

Rep	Cell	ppm PH_3 Start	ppm PH_3 24 h	% sorption	ppm PH_3 penetration
1	#1 Top	338.1	294.4	10.7	8.3
	Bottom	NPD	8.3		
2	#2 Top	322.6	286.6	9.2	7.0
	Bottom	NPD	7.0		
3	#3 Top	327.4	300.6	6.0	7.5
	Bottom	NPD	7.5		
$\bar{x}$ ± SD	Top	329.4 ± 7.9	293.9 ± 7.0	8.6 ± 2.4	7.6 ± 0.6
	Bottom	NPD	7.6 ± 0.6		

NPD = No peak detected

4. Gacoflex P-5000 white

Rep	Cell	ppm PH_3 Start	24 h	% sorption	ppm PH_3 penetration
1	#1 Top	355.3	334.9	5.7	0.21
	Bottom	NPD	0.21		
2	#2 Top	369.1	346.2	6.0	0.76
	Bottom	NPD	0.76		
3	#3 Top	318.6	310.0	2.5	0.58
	Bottom	NPD	0.58		
$\bar{x}$ ± SD	Top	347.7 ± 26.1	330.4 ± 18.5	4.7 ± 1.9	0.52 ± 0.28
	Bottom	NPD	0.52 ± 0.28		

NPD = No peak detected.