

# Persistence of grain protectants in maize

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## Abstract

This paper reports on the persistence of organophosphates and synthetic pyrethroids in maize over a storage period of 36 weeks.

## Introduction

In recent years, a wide range of new grain protectants has been identified to manage grain insects that have developed resistance to malathion. An effective organophosphate (op) is usually combined with a synergised synthetic pyrethroid (sp) to ensure efficiency against a wide range of stored product insects in the multi-resistant strains of *Rhyzopertha dominica* (Bengston 1985). Australia has developed many of these op-sp combinations for stored grain. Some of these were evaluated in Malaysia for their efficacy against maize insects and their persistence under storage conditions. This paper reports on the persistence of organophosphates and synthetic pyrethroids in maize over a storage period of 36 weeks.

## Methods

Maize grain on a moving conveyor belt were treated with aqueous mixtures using a motorised sprayer. The admixture treatments were: a. fenitrothion (12 mg/kg) + fenvalerate (0.5 mg/kg) + piperonyl butoxide (pbo) (8.0 mg/kg); b. deltamethrin (1.0 mg/kg) + pbo (8.0 mg/kg); c. pirimiphos-methyl (6.0 mg/kg) + permethrin (1.0 mg/kg) + pbo (8.0 mg/kg); d. chlorpyrifos-methyl (10.0 mg/kg) + bioresmethrin (1.0 mg/kg) + pbo (8.0 mg/kg). Treated grain was sampled for storage in fibre-glass tanks. From these, subsamples were taken for chemical assay over a storage period of 0.6, 12, 18, 24, 30 and 36 weeks after treatment (WAT). For chemical assay, 40 g of grain were extracted with 100 mL methanol in a reagent bottle for 3 days (2). An aliquot of the filtered methanol extract was injected into a gas-liquid chromatograph (glc) equipped with an alkali flame ionisation detector (afid) to determine organophosphate deposits and residues. For fenvalerate, permethrin and deltamethrin analysis, an aliquot of the filtered methanol extract was cleaned up with a Sep-Pak Florisil cartridge for analysis with a glc equipped with an electron-capture detector (ecd). Bioresmethrin was estimated by direct injection into a high-performance liquid chromatograph (hplc) equipped with a UV-detector. Since pbo acts only as a synergist and its activ-

ity does not vary over a range of concentrations, levels of piperonyl butoxide in maize were not determined.

## Results and Discussion

Tables 1, 2, 3 and 4 demonstrate that, in real situations, applied dosages determined by chemical assay could be different from the target dosages. Contributing factors may include chemical volatility, bound residues, spray calibrations, loss of recovery of active ingredients and presence of dusts. Table 1 showed that fenvalerate was overdosed by 6 times. Data showed that

**Table 1.** Deposits and residues of grain protectants on maize after treatment of fenitrothion (12 mg/kg) + fenvalerate (0.5 mg/kg) + piperonyl butoxide (8.0 mg/kg).

WAT	Deposit/residue (mg/kg)	
	Fenvalerate <sup>a</sup>	Fenitrothion <sup>b</sup>
0	3.0	7.4
6	2.7	4.4
12	2.5	2.5
18	2.2	1.5
24	2.2	1.3
30	2.0	1.0
36	2.0	0.9

WAT, weeks after treatment

<sup>a</sup>Analysis by glc-ecd; injector and detector temperature, 290°C; column temperature, 290°C; column, 3% OV-101, Varaport 30 100/120 mesh; nitrogen flow-rate, 18 divisions on flowmeter.

<sup>b</sup>Analysis by glc-afid; injector and detector temperature, 245°C; column temperature, 205°C; column, 1.5% OV-17 + 1.95% OV-210, 80/100 Chromosorb B WHP; hydrogen flow rate, 35 mL/min; air, 250 mL/min; nitrogen, 18 divisions on flowmeter.

**Table 2.** Deposits and residues of grain protectants on maize after treatment of deltamethrin (1.0 mg/kg) + piperonyl butoxide (8.0 mg/kg).

WAT	Deposit/residue (mg/kg)
	Deltamethrin <sup>a</sup>
0	0.4
6	0.2
12	0.2
18	0.1
24	0.1
30	0.1
36	0.1

WAT, weeks after treatment

<sup>a</sup>Analysis by glc-ecd; injector and detector temperature, 290°C; column temperature, 260°C; column, 3% OV-101, Varaport 30 100/120 mesh; nitrogen flow rate, 18 divisions on flowmeter.

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**Table 3.** Deposits and residues of grain protectants in maize after treatment of pirimiphos-methyl (6.0 mg/kg).

WAT	Deposit/residue (mg/kg)	
	Pirimiphos <sup>a</sup>	Permethrin <sup>b</sup>
0	4.2	1.1
6	2.8	0.9
12	2.0	0.7
18	1.8	0.6
24	1.1	0.7
30	1.1	0.6

WAT, weeks after treatment

<sup>a</sup>analysis by glc-afid; injector and detector temperature, 220°C; column temperature, 180°C; column, 3% OV-101 Varaport 30 100/120 mesh; hydrogen flow rate, 35 mL/min; air, 250 mL/min; nitrogen, 18 divisions on flowmeter.

<sup>b</sup>Analysis by glc-ecd; injector and detector temperature, 290°C; column temperature, 270°C; column, 3% OV-101, Varaport 30 100/120 mesh; nitrogen flow rate, 18 divisions on flowmeter.

**Table 4.** Deposits and residues of grain protectants in maize after treatment of chloropyriphos-methyl 10.0 mg/kg + bioresmethrin (1.0 mg/kg) + piperonyl butoxide (8.0 mg/kg).

WAT	Deposit/residue (mg/kg)	
	Chloropyriphos <sup>a</sup>	Bioresmethrin <sup>b</sup>
0	3.5	nd
6	2.2	nd
12	1.2	nd
18	1.1	nd
24	0.1	nd
30	0.6	nd
36	0.4	nd

WAT, weeks after treatment

<sup>a</sup>Analysis by glc-afid; using the same conditions as in Table 3.

<sup>b</sup>Analysis by hplc-UV; mobile-phase, 85:15 (methanol: water); nd, not detectable.

**Table 5.** Maximum residue limits (MRLs) of grain protectants for cereal grain (FAO/WHO).

Chemical	MRLs (mg/kg)
Fenitrothion	10.0
Fenvalerate	5.0
Deltamethrin	1.0
Pirimiphos-methyl	10.0
Permethrin	2.0
Chlorpyriphos-methyl	10.0
Bioresmethrin	5.0

organophosphates were more persistent than synthetic pyrethroids under storage conditions. Residues of bioresmethrin were not detected (Table 4). In all the trials, deposits and residues of organophosphates and synthetic pyrethroids did not exceed the Maximum Residue Limits (MRLs) set by the Codex Alimentarius (Table 5). The persistence data indicated that grain can be stored for longer periods to reduce residues if required by more stringent MRLs.

## References

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