Dislodgable Leaf Residues of Insecticides on Cotton

by

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In a previous study (Ware et al. 1972) we attempted to show the relative degradation rates of selected organophosphate insecticides on cotton. However, the dislodgable nature of these residues was not taken into consideration, since they were expressed on a ppm basis after being extracted from macerated plant material. It was the purpose of this study to determine the dislodgable or easily removed surface residues of seven commonly used insecticides on cotton over a 4-day period following application. Such information is essential in establishing safe reentry times for personnel working in treated fields.

MATERIALS AND METHODS

Test plots were in a 90-acre field of DPL-16 cotton on the Wilbur Wuertz farm adjacent to the Picacho Reservoir, La Palma, Arizona. Planted in mid-March, the plots were treated on July 10, when the cotton averaged 27" in height, varying from 14" to 42", and had abundant squares and blooms, with few bolls. The plots consisted of 12 40" rows 1000' long, making each approximately 1.0 acre.

The sprays were applied at 9 gallons per acre with a 12-row International high clearance (Hi-Boy) self-propelled, ground sprayer, using 3 #6 cone nozzles per row, and 40 psi, beginning at 7:20 a.m. and finishing at 11:15 a.m. The materials in application sequence, rate of active ingredient (AI) per acre, and formulation were:

Azodrin, 1.0 lb, 5 lb/gal EC
Methyl parathion, 1.0 lb, 4 lb/gal EC
Toxaphene-Methyl parathion, 2 + 1 lb, 6 + 3 lb/gal EC
Ethyl-Methyl parathion, 1 + 0.5 lb, 6 + 3 lb/gal EC
Ethyl parathion, 1.0 lb, 4 lb/gal EC
Guthion, 0.5 lb, 2 lb/gal EC
Galecron, 1.0 lb, 4 lb/gal EC
Lannate, 0.75 lb, 90% water-sol. powder
DDT, 1.0 lb, 2 lb/gal EC
Methyl parathion (encapsulated), 1.0 lb, 2 lb/gal

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The temperature ranged from 89°F at the beginning to 100°F at the end of applications, reaching 107° later that day. The max-min temperatures for the remainder of the study were: July 11, 108°-78°; July 12, 103°-80°; July 13, 101°-79°; and July 14, 100°-79°. Weather conditions for the 4-day residue study were warm, dry and windy with no rainfall.

Triplicate leaf residue samples were collected in each treated and one control plot from the middle six rows, leaving the outside six as guard rows, at 0, 24, 48, 72 and 96 hours after treatment. Each sample was composed of 100 leaves picked singly and consecutively from the top, middle and bottom portions of the plant, in two rows, not less than 50' from the plot end. Each sample was placed in a plastic bag, identified, and held in an ice chest for return to the laboratory.

Because the study involved only the dislodgable residues, the leaves were center punched with a 36-mm, sharpened aluminum tube, the discs were rinsed twice with a total of 450 ml of the appropriate solvent, and allowed to stand 2 minutes per rinse. The solvents were distilled water for Azodrin and Lannate, hexane for Galecron, and benzene for the remainder. The rinses were combined and refrigerated or held in the freezer.

The Lannate extracts were analyzed by the DuPont Experimental Station laboratories, Wilmington, Delaware, and Galecron by the CIBA-Geigy Agricultural Div. laboratories, Ardsley, N.Y.

The aqueous Azodrin leaf washes were partitioned into chloroform by adding 14 g NaCl to 50 ml and extracting three times with 50 ml of chloroform. The combined chloroform extracts, in order to be gas chromatographed, were concentrated with an air stream to about 0.1 ml and returned to volume with 10% acetone in hexane.

The remaining materials in benzene were adjusted in volume and gas chromatographed using a Microtek 162 with a ^{63}Ni electron capture detector for DDT, and a Microtek 220 for the organophosphates.

All determinations of Azodrin, methyl- and ethyl parathion and their oxons, and Guthion, were made by gas chromatography, using a flame-photometric detector sensitive to phosphorus-containing compounds. For the parathions, their oxons, and Guthion, the chromatograph was equipped with a 183 cm X 4 mm ID Pyrex glass column packed with 100/120 mesh Chromosorb W HP containing 5% by weight of SE-30. For methyl parathion the operating parameters were: 73 ml/min, 225°, 197° and 235°; methyl paraoxon 73 ml/min, 225°, 177° and 235°; ethyl parathion, 73 ml/min, 225°, 197° and 235°; ethyl paraoxon, 73 ml/min, 225°, 177° and 235°; and Guthion, 73 ml/min, 245°, 235° and 260°C, respectively for nitrogen gas flow, inlet, column and detector temperatures.

For Azodrin the chromatograph was equipped with an 81 cm X 4 mm ID Pyrex glass column packed with a 1:1 mixture of 80/100 mesh Gas Chrom Q containing 2% Reoplex 400, and 60/80 mesh Chromosorb W (Fe free and acid washed) containing 5% QF1. Operating parameters were 68 ml/min, 230°, 196° and 239°C, respectively for nitrogen flow and inlet, column, and detector temperatures.

Quantitation was by peak height, compared to standard curves made from 2-6 μl of standard solutions containing 0.2 to 1.0 ng/μl for the organophosphates and 0.01 ng/μl for the DDT-related materials. Because of the experimental design, recovery standards were run only for the DDT-related materials which were cleaned up on a florisil column. They were: p,p'-DDT, 93%; o,p'-DDT, 93%, and p,p'-DDE, 92%.

RESULTS AND DISCUSSION

The results are shown in Table 1, expressed as milligrams per square meter (mg/M^2) of leaf. The rate of disappearance from initial deposit was Lannate > methyl parathion > ethyl parathion > Toxaphene-methyl parathion > Guthion > encapsulated methyl parathion > DDT > Azodrin. The analytical results for Lannate and Galecron, which were conducted by laboratories of the basic manufacturers, are not consistent with the others. The Galecron-treated plot showed no detectable residue at any time, including application, though the tank sample collected immediately after spraying contained the appropriate concentration. Lannate residues, on the other hand, were present but at much lower levels than the other compounds. This is to be expected based on a report (Pease 1971) that surface residues decrease 71-93% during the first 12 hrs on corn, bean and cabbage. During this time interval the concentration of Lannate in the plant remains essentially constant, indicating rapid surface loss with some absorption.

It is conceivable, but unlikely, that the application of Galecron was incomplete due to nozzle clogging or other mechanical failure. The pH of spray dilution water and temperature at time of application may account for part of the Galecron discrepancies. These will be explored in the 1973 studies.

REFERENCES

PEASE, H. L. Unpublished data. (1971)

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Table 1

Insecticide residues expressed as mg/M^2 of cotton leaf following application by ground equipment. LaPalma, Arizona, July 10, 1972.

Hours	Methyl Parathion 1#/A	Methyl Paraoxon	Methyl Parathion (Encapsulated) 1#/A	Methyl Paraoxon	Toxaphene Methyl Parathion 2# + 1#/A	Methyl Paraoxon	Ethyl Parathion 1#/A	Ethyl Paraoxon
0	9.53	.19	8.87	0.015	12.04	.16	9.5	0.08
24	1.38	.21	4.33	0.016	2.09	.12	1.24	0.15
48	.28	.11	3.36	0.013	0.64	.07	0.51	0.10
72	.18	.07	3.90	0.013	0.23	.04	0.28	0.05
96	.11	.04	2.87	0.012	0.14	.03	0.17	0.04

Hours	Ethyl-Methyl Parathion				DDT			
	Ethyl Parathion 1#/A	Ethyl Paraoxon	Methyl Parathion 0.5#/A	Methyl Paraoxon	p,p' 1#/A	o,p' 1#/A	DDE 1#/A	Total
0	14.5	Trace	5.94	.13	17.0	2.96	0.44	20.4
24	2.09	0.19	0.61	.15	12.0	1.26	0.21	13.5
48	0.71	0.12	0.16	.06	8.42	0.63	0.13	9.2
72	0.37	0.09	0.008	.04	4.62	0.33	0.06	5.0
96	0.20	0.05	0.004	.03	3.72	0.27	0.03	4.0

Hours	Azodrin 1#/A	Guthion 0.5#/A	Lannate 0.75#/A	Galecron 1#/A
0	14.2	8.43	1.68*	0.0
24	14.8	5.68	1.19	0.0
48	11.0	2.89	.65	0.0
72	7.7	2.29	.41	0.0
96	7.0	1.49	.37	0.0

*Based on 1 Sample.