

Dislodgeable Insecticide Residues on Cotton Foliage: Permethrin, Curacron, Fenvalarate, Sulprofos, Decis, and Endosulfan

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Our most recent study (WARE et al. 1978) compared the dislodgeable residues of chlordimeform (Fundal), EPN + methyl parathion, toxaphene + methyl parathion, chlorpyrifos (Lorsban), methyl parathion, diazinon, microencapsulated methyl parathion (Pencap M) and microencapsulated diazinon (Knox Out) on cotton over 96-h post-application.

This report continues to explore the disappearance rates from cotton of several insecticides: permethrin (Ambush), Curacron, fenvalarate (Pydrin), Sulprofos (Bolstar), Decis (FMC-45498), and endosulfan (Thiodan).

METHODS AND MATERIALS

The insecticide test plots were located in a 16-acre block of Stoneville 265 short staple cotton on the Agricultural Experiment Station, Marana, Arizona. The average height of cotton plants was 107 cm when plots were treated on August 8, 1977. Each plot was 4 rows, with 102 cm spacing, 30.5 m long. The sprays were applied at 122 L/ha, at 4 km/h and 276 kPa pressure. The spray rig was manually drawn, and treated two rows, using 3 DC 2-13 Spraying System nozzles per row. Pressure was maintained from a 1.37 kg CO₂ tank having a single stage regulator.

The insecticides in order of application, formulation, and rate of active ingredient (AI) per hectare were: Ambush (2EC) @ 0.11 kg/ha, Curacron (6EC) @ 1.1 kg/ha, Pydrin (2.4EC) @ 0.11 kg/ha, Bolstar 6 (6EC) @ 1.1 kg/ha, Decis (0.21 lb/gal) @ 0.023 kg/ha, and Thiodan (2EC) @ 1.1 kg/ha.

Temperature maximums and minimums during the test were Aug. 8, 38.5° - 23.9°; Aug. 9, 37.2° - 22.8°; Aug. 10, 36.7° - 21.1°; Aug. 11, 39° - 22.2°; Aug. 12, 36.7° - 22.2° C. Two significant rains challenged the residues of this study: 0.79 cm and 0.44 cm fell on the evenings of Aug. 9 and 11, respectively.

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Triplicate samples were collected in each treated plot at 0, 24, 48, 72 and 96 h after treatment, with controls collected on 0, 48, and 96 h. Each sample consisted of 100 leaves punched singly and consecutively from the top, middle and bottom portions of plants in all 4 rows, using 2.54-cm diameter leaf punches.

In the field each sample was extracted with 100 mL of the appropriate solvent for one min. The extract was then transferred to an 8-oz labeled sample bottle, the solvent levels marked, and the jars placed on ice until transferred to the laboratory refrigerator. The extracting solvents were benzene for Curacron, Sulprofos and endosulfan, and hexane for permethrin, fenvalarate and Decis. Controls were extracted with either hexane or benzene.

Curacron and Sulprofos were analyzed directly using a MicroTek MT-220 GC equipped with a flame photometric detector. A 120 cm x 4 mm I.D. Pyrex column containing 1.5% OV-17 and 2% QF-1 on 100/120 mesh Chromosorb W (H.P.) was used. Quantitation was by peak height. Carrier gas flow was 60 mL/min and temperatures were: column, 210°C; detector, 213°C; and inlet, 230°C. No cleanup was required.

Sulprofos sulfone and sulfoxide were analyzed on a 90 cm Pyrex column containing 2% OV-101 on 100/120 mesh Chromosorb W (H.P.). Carrier gas flow was 120 mL/min and temperatures were: column, 205°C; detector, 210°C; and inlet 225°C.

Fenvalarate hexane extracts were cleaned up on a 2.5 cm column of activated Florisil (120°C for 24 h), covered with a 1.25 cm layer of Na₂SO₄, after pretreating with 25 mL of hexane. The column was rinsed with hexane, and then eluted with 50 mL of hexane followed by 50 mL of 5% ethyl acetate in hexane. The total eluate was concentrated and transferred to a 12-mL centrifuge tube for analysis. Extracts were analyzed on the MicroTek MT-220 equipped with a ⁶³Ni electron capture detector. The 30 cm x 4 mm I.D. Pyrex column was packed with 5% SE-30 on 100/120 mesh Chromosorb W (H.P.). Carrier gas flow was 95 mL/min and temperatures were: column, 200°C; detector, 270°C; and inlet, 218°C.

Permethrin extracts were analyzed directly by ECGC with no cleanup. GC and detector were the same as for fenvalarate. The Pyrex column, however, was 71 cm long and 4 mm I.D., packed with 3% SP-2330 on a mixture of 27% Chromosorb W (A.W.) and 70% Chromosorb W (H.P.). Carrier gas flow was 62 mL/min and temperatures were: column, 195°C; detector, 278°C; and inlet, 218°C. Standard curves were established using an analytical standard of 40:60 mixture of the cis and trans isomers.

Decis extract aliquots were concentrated in a centrifuge tube to 40% of their original volume. Transesterification was according to the method of its basic manufacturer, the FMC Corp. (FULLMER 1978), in which 1 mL of benzene and 0.1 mL of 0.02 N KOH in methanol

TABLE 1. Dislodgeable Residues Expressed as ($\mu\text{g}/\text{cm}^2$) of Cotton Leaf Following Application by Ground Rig.
Marana, AZ. August 8, 1977.

Hours	Thiodan		Curacron		Bolstar	
	1.1 kg/ha		1.1 kg/ha		1.1 kg/ha	
	Low Melting Isomer I	High Melting Isomer II	Thiodan Sulfate	Bolstar Sulfone	Bolstar Sulfone	Bolstar Sulfoxide
0	1.1	0.65	<0.004	2.6	0.014	0.14
24	0.18	0.28	0.036	1.0	0.084	0.44
48	0.095	0.12	0.083	0.49	0.049	0.17
72	0.061	0.10	0.11	0.35	0.13	0.19
96	0.040	0.073	0.14	0.12	0.024	0.070

Hours	Pydrin		Ambush		FMC 45498 (Decis)	
	0.11 kg/ha		0.11 kg/ha		0.023 kg/ha	
	Cis	Trans	Total			
0	0.20	0.099	0.19			0.043
24	0.17	0.10	0.20			0.041
48	0.18	0.061	0.12			0.026
72	0.20	0.042	0.083			0.016
96	0.13	0.047	0.093			0.014

was added to 4 mL of Decis extract. The solution was heated at 50°C for 15 min, cooled to ambient, diluted to 10 mL with hexane and analyzed by ECGC. GC and detector were the same as for fenvalarate and permethrin. The Pyrex column was 45 cm long and 4 mm I.D., packed with 5% SE-30 on 100/120 mesh Chromosorb W (H.P.). Carrier gas flow was 31 mL/min, and temperatures were: column, 120°C; detector, 262°C; and inlet, 195°C. The standard curve was made from transesterified analytical grade Decis.

Endosulfan extracts were cleaned up on 10 cm columns of activated Florisil topped with 1.25 cm of Na₂SO₄. Following column rinsing with 60 mL of hexane, a 10-mL aliquot of sample was added and allowed to flow through. After rinsing with hexane, the column was eluted with 200 mL of 40% ethyl ether containing 2% ethanol in hexane. The total eluate was collected, evaporated to 5 mL, and transferred to a 12-mL centrifuge tube for analysis by ECGC. GC and detector were the same as for fenvalarate and permethrin. The column was the same as that used for Decis. Carrier gas flow was 83 mL/min, and temperatures were: column, 170°C; detector, 277°C; and inlet, 217°C.

RESULTS AND DISCUSSION

The results are shown in Table 1, expressed as micrograms of toxicant or metabolite per square centimeter of cotton leaf ($\mu\text{g}/\text{cm}^2$), one surface only. The two rains, between the 24-48, and 72-96 h samplings seemed to affect only Sulprofos sulfoxide. Endosulfan, Curacron and Sulprofos residues diminished most during the first 24 h to levels that were probably less than efficacious, while the three pyrethroids appeared to sustain little loss from the combination of heat, sunlight and rain. Fenvalarate was 65%, permethrin 47%, and Decis 32% of their original residues at the end of 96 h of intense weathering.

Insecticides that can withstand the harsh ultraviolet, high temperatures and heavy thundershowers of Arizona can undoubtedly survive with improved efficacy in other cotton areas having less stringent climatological conditions. This study indicates that a part of the reported efficacy of the synthetic pyrethroids can be attributed to their resistance to photolysis and removal by rain.

REFERENCES

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