Dislodgable Insecticide Residues on Cotton[1]

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Our last study (Cahill et al., 1975) compared the dislodgable residues of methyl and ethyl parathion obtained by water vs. benzene extraction, and also included disappearance data on Azodrin, Guthion, Lannate and Galecron over 96 hours post-application. The purpose of the present study was to explore the disappearance rates from cotton foliage of methyl parathion alone and with toxaphene, two formulations of Sevin, Fundal, Supracide, and Carzol.

MATERIALS AND METHODS

The test plots were located in a 25-acre cotton field at La Palma, Arizona. The plots were treated on August 12, 1974, when the cotton averaged 43.8" in height. Each plot consisted of 12 rows with 40" spacing, 1000 ft long, approximating 1.0 acre. The sprays were applied in 9.6 gallons per acre with a 12-row International high-clearance (Hi-Boy), self-propelled, ground sprayer using 3 No. 6 cone nozzles per row, operated at 40 psi and 4 mph, from 7:30 a.m. until 9:45. The materials in the sequence of application, rate of active ingredient (AI) per acre, and formulations were:

Supracide (2 EC) @ 0.5 lb. AI/acre
Fundal (4 EC) @ 1.0 lb. AI/acre
Sevimol (4 EC) @ 1.5 lb. AI/acre
Methyl Parathion (4 EC) @ 1.0 lb. AI/acre
Toxaphene-MePara (6 + 3 EC) @ 2.0 + 1.0 lb AI/acre
Carzol (92% SP) @ 0.5 lb. AI/acre
Sevin (80% WP) @ 1.5 lb. AI/acre
Control

The daily maximum-minimum temperatures during the study were as follows: Aug. 12, 102°-63°; Aug. 13, 102°-69°; Aug. 14, 101°-72°; Aug. 15, 104°-74°; and Aug. 16, 105°-71°. No precipitation occurred during the 5-day study.

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Foliage sampling techniques consisted of collecting three replicates of 100 leaf discs, 1.0" in diameter, in each treated and one control plot at 0, 24, 48, 72, and 96 hours after treatment from the middle six rows, leaving the three rows on each side as buffer zones. The discs were punched singly and consecutively from the top, middle, and bottom portions of the plant canopy, not less than 50 ft from the row ends.

Each sample was returned to the field laboratory adjacent to the treated plots and extracted immediately by vigorous shaking for 30 seconds in 100 ml of the appropriate solvent:

Compound:	Extracting Solvent:
Supracide	Methylene chloride
Fundal	Hexane
Sevimol, Sevin	Methylene chloride
Methyl parathion, MP + Tox	Benzene
Carzol	H_2O pH 3.0

All extracts were then chilled in ice chests and returned to the laboratory where the Carzol extracts were frozen.

The Carzol and Fundal extracts were air-freighted under dry ice to the Nor-AM Agricultural Products, Inc., laboratories in Woodstock, Illinois, where they were analyzed.

The extracts of the two methyl parathion and Supracide treatments were analyzed without cleanup using a Microtek 220 GC equipped with a flame photometric detector and phosphorus filter.

Dilutions of the Supracide extracts were made with benzene when required. The following GC parameters apply to the analysis of Supracide, methyl parathion and methyl paraoxon: column, 4 mm ID pyrex and 5 ft in length, packed with 5% SE-30 on 100/120 mesh Chromosorb W, HP; nitrogen carrier 70 ml/min, hydrogen 200 ml/min, oxygen 20 ml/min and compressed air 50 ml/min. Quantitation was by peak height using a standard curve ranging from 1-10 ng. Volumes of 1.5 to 7.5 μl were injected for both standards and samples.

Sevin residues were measured first in this laboratory by the Union Carbide colorimetric method (1974), which is an adaptation of the official AOAC method, and later by gas chromatography of the bromine derivative described by Gutenmann and Lisk (1965). The latter method proved to be faster, with less deviation of replicates, and sensitivity equal to the colorimetric method. It has the added advantage in that the derivative is stable for days, while the colorimetric product is extremely unstable and must be analyzed immediately upon development.

The results are shown in Table 1, expressed as milligrams of insecticide per square meter of cotton leaf (mg/m^2). Several observations were made. First, the addition of toxaphene appar-

Table 1. Insecticide residues expressed as mg/m^2 of cotton leaf following application by ground equipment. La Palma, Arizona, August 12, 1974.

Hours	Methyl Parathion 1#/A	Methyl Paraoxon	Toxaphene Methyl Parathion 2# + 1#/A	Methyl Paraoxon	
0	44.6	0.51	46.2	0.28	
24	2.9	0.38	21.1	0.53	
48	0.51	0.20	10.3	0.44	
72	0.28	0.16	4.7	0.28	
96	0.20	0.06	1.9	0.20	
	Sevin 1.5#/A	Sevimol 1.5#/A	Fundal 1#/A	Carzol 0.5#/A	Supracide 0.5#/A
0	36.0	46.7	5.7	9.7	14.6
24	47.3	63.2	0.78	1.3	3.5
48	58.3	30.5	0.42	0.83	0.93
72	27.3	29.4	0.53	0.38	0.48
96	36.4	21.2	0.48	0.10	0.25

ently protects methyl parathion from dissipation rather significantly. Whether this preservation results in a residue available and toxic to insects is speculative. Even to the casual observer the residue (obviously toxaphene, containing the dissolved parathion), is readily visible on the leaf surfaces two days after application.

Second, the Fundal residue performed precisely as it has in the past. It appears impossible to collect samples quickly enough to measure residues in the magnitude of their application, e.g. 5.7 mg at 0 hours versus the 44.6 mg of methyl parathion, both applied at the 1.0 lb. rate. In an earlier study (Ware et al. 1974) no residue of Fundal was recovered at time of application when the leaves were returned to the laboratory and extracted, a delay of three hours. This loss would seem to be a physical phenomenon in which the applied insecticide is very rapidly sorbed by the leaf or volatilized. In either case, there does not appear to be enough time for the material to exert its ovicidal and larvicidal effects before dissipation, an insecticidal anomaly, particularly in view of its use as a bollworm (*Heliothis* spp.) ovicide at rates as low as 1/8 lb/acre.

And finally, the peculiar inconsistency in the Sevin and Sevimol data point to the original leaf extraction as the source of error and not the analyses. Each of the two analytical methods gave within 20% of the other results, indicating that both were reliable. The data suggest that extraction is incomplete, and that methylene chloride should not be the solvent of choice. We would suggest instead the use of acetone. Despite the erratic nature of the residue data Sevin and Sevimol are unquestionably the most persistent of the materials tested, followed by the toxaphene-methyl parathion combination. The other materials have no significant dislodgable residue qualities beyond 24 hours.

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