# Foliage Residues of Insecticides on Cotton

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In a previous study (Ware et al. 1974) the rate of disappearance of the dislodgable residues of certain organophosphate insecticides from cotton leaf was determined. It is the purpose of this paper to confirm these results and in addition to compare the quantities of dislodgable methyl and ethyl parathion residues obtained by water vs. benzene extraction.

## MATERIALS AND METHODS

Test plots were located in a field on the Wilbur Wuertz farm, La Palma, Pinal County, Arizona. The plots were treated on August 6, 1973, when the cotton averaged 45" in height, varying from 28" to 56", and had abundant bolls but few squares or blooms. The plots consisted of 12 rows with 40" spacing, 1000 ft 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 #TX-6 nozzles per row, at 40 psi, from approximately 7:00 a.m. until approximately 12:00. The materials in application sequence, rate of active ingredient (AI) per acre, and formulation were:

Methyl parathion, 1.0 lb, 4 lb/gal EC
Azodrin, 1.0 lb, 5 lb/gal water miscible form
Toxaphene-methyl parathion, 4 + 1 lb, 8 + 2 lb/gal
Ethyl parathion, 1.0 lb, 4 lb/gal EC
Guthion, 0.5 lb, 2 lb/gal EC
Ethyl-methyl parathion, 1.0 + 0.5 lb, 6 + 3 lb/gal EC
Galecron, 1.0 lb, 4 lb/gal EC
Lannate, 0.5 lb, 1.8 lb/gal
Methyl parathion, (microencapsulated), 1.0 lb, 2 lb/gal

The temperatures throughout the study were as follows: Aug. 6, 104°-72°; Aug. 7, 104°-71°; Aug. 8, 107°-68°; Aug. 9, 108°-70°; and Aug. 10, 104°-74°. No precipitation was measured.

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Triplicate leaf samples were collected in each treated and one control plot from the middle six rows, leaving the three rows on each side as a buffer zone at 0, 24, 48, 72 and 96 hours after treatment. Each sample was composed of 75 leaves (no stems) picked singly and consecutively from the top, middle and bottom portions of the plant canopy, in two rows, not less than 50' from the plot end.

Cotton leaf surface areas were determined by obtaining imprints on blueprint paper of leaves collected in the same manner as those to be extracted.

The prints were fixed by first wetting the paper with tap water followed by immersion in a (1:7) 32% hydrogen peroxide: tap water solution until most of the reaction ceased. The paper was then rinsed with tap water and allowed to dry. The dried prints were cut out, weighed and compared to a known area of blueprint paper treated as above, to establish an area-to-weight relationship.

Each sample (75 leaves) was placed in a plastic bag during picking, brought to the field laboratory, weighed and transferred to a two-quart widemouth mason jar. The leaves were immediately extracted with 900 ml of appropriate solvent for 30 seconds using a gentle tilting action. The extracts were decanted through large $60^{o}$ funnels into one-quart bottles, and refrigerated until analyzed.

The plot treatment and corresponding solvent used in the extractions were: methyl and/or ethyl parathions, benzene; Guthion, benzene; Azodrin, distilled acetone; Galecron, distilled hexane; and Lannate, tap water. Additional samples from the ethyl-methyl parathion treated plot were extracted with water. The water extracts were partitioned into benzene at the laboratory.

The Lannate-containing extracts were analyzed by the duPont Experimental Station Laboratories, Wilmington, Delaware, and Galecron by Bio/dynamics, Inc., East Millstone, New Jersey.

All remaining extracts, whether in benzene or in acetone, were analyzed without cleanup using a Microtek 220 GC equipped with a flame photometric detector. The following GC parameters apply to all compounds analyzed by this method: the columns, 4 mm ID pyrex, were 16" long for Guthion and 5 ft for the other compounds. Column temperatures were $165^{o}C$ for Azodrin and $198^{o}C$ for the rest. Gas flow rates were: Nitrogen carrier 80 ml/min, hydrogen 200 ml/min, oxygen 20 ml/min and compressed air 50 ml/min. Quantitation was by peak height with the standard curve ranging from 1-10 ng. Volumes ranging from 1.5-7.5 $\mu$l were injected for both standards and samples.

The results are shown in Table 1, expressed as milligrams of insecticide per square meter of cotton leaf ($mg/m^2$). The rates of disappearance of the dislodgable residues correlate well with last year's results except for the three most persistent compounds. The rates of disappearance are: Lannate > Galecron > methyl parathion $\approx$ toxaphene/methyl parathion $\approx$ methyl parathion mixed with ethyl parathion > ethyl parathion $\approx$ ethyl parathion mixed with methyl parathion

>Azodrin≈Guthion>methyl parathion encapsulated. Last year's results for the last three compounds indicated Guthion>methyl parathion encapsulated>Azodrin.

These differences may be explained by several changes in extraction technique between the two studies. The leaf samples were extracted immediately after picking in this study while last year they were extracted after about a 3-4 hour delay. Although these samples were placed in coolers, they were often warm 3-4 hours later for extraction. Guthion was analyzed at a column temperature of 198° compared to 235° for last year, a temperature approximately 38° above its decomposition point.

As expected the benzene rinse of the parathions contained more residue than the water rinses. The benzene rinses contained from 2-3 times the parent compounds and 5-10 times the oxons as the water rinses.

The use of benzene, a solvent that does dissolve plant cuticle, has been questioned because it was felt that dislodgable residues are strictly superficial and should be removed by water extractions. however, our experience with workers and personnel involved in reentry studies indicates that dislodgable residue is probably greater than just surface residue. This is evidenced by the large amounts of chlorophyll on the clothing and hands of individuals after a day conducting inspection for cotton insects. This is particularly true for workers in tall or maturing cotton in which there are fallen stalks.

It is probable that the real dislodgable residue as it applies to field workers is intermediate between that which can be removed by benzene and water extractions.

## REFERENCES

Ware, G. W., Betty Estesen and W. P. Cahill. 1974. Dislodgable leaf residues of insecticides on cotton. Bull. Environ. Contam. Toxicol. 11(5): 434-437.

Table 1. Insecticide residues expressed as $mg/m^2$ of cotton leaf following application by ground equipment. La Palma, Arizona, August 6, 1973.

| 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 | 20.2 | 0.13 | 5.8 | 0.080 | 28.1 | 0.073 | 14.9 | 0.38 |
| 24 | 1.9 | 0.22 | 2.9 | 0.12 | 3.5 | 0.19 | 3.2 | 0.11 |
| 48 | 0.64 | 0.13 | 2.8 | 0.10 | -- | -- | 1.0 | 0.043 |
| 72 | 0.22 | 0.093 | 1.7 | 0.10 | 0.52 | 0.077 | 0.42 | 0.027 |
| 96 | 0.14 | 0.057 | 1.3 | 0.066 | 0.32 | 0.064 | 0.32 | 0.020 |

| | Ethyl-Methyl Parathion | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Ethyl Parathion (1#/A) | | Ethyl Paraoxon | | Methyl Parathion (0.5#/A) | | Methyl Paraoxon | |
| | Benzene Extract | $H_2O$ Extract | Benzene Extract | $H_2O$ Extract | Benzene Extract | $H_2O$ Extract | Benzene Extract | $H_2O$ Extract |
| 0 | 27.9 | 14.9 | -- | -- | 13.5 | 9.9 | 0.31 | 0.079 |
| 24 | 4.5 | 1.9 | 0.23 | 0.040 | 1.4 | 0.58 | 0.21 | 0.020 |
| 48 | 1.5 | 0.55 | 0.13 | 0.012 | 0.37 | 0.098 | 0.073 | 0.008 |
| 72 | 0.6 | 0.073 | 0.072 | 0.009 | 0.13 | 0.027 | 0.029 | 0.005 |
| 96 | 0.4 | 0.12 | 0.056 | 0.009 | 0.090 | 0.034 | 0.023 | 0.005 |

| | Azodrin 1#/A | Guthion 0.5#/A | Lannate 0.5#/A | Galecron 1#/A |
|---|---|---|---|---|
| 0 | 25.1 | 13.8 | 7.7 | 5.4 |
| 24 | 13.9 | 6.7 | 0.32 | 1.0 |
| 48 | 8.4 | 3.1 | 0.08 | 0.76 |
| 72 | 4.3 | 2.9 | 0.074 | -- |
| 96 | 3.0 | 2.3 | 0.023 | -- |