## Organophosphate Residues on Cotton in

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Considerable interest has been shown recently in establishing safe reentry times for personnel working in fields treated with insecticides. One of several pieces of information needed for such decisions is the residue history of a given insecticide on a specific crop. This study was designed to give a general picture of the residual lives of four organophosphates on cotton in mid-summer.

The test plots were DPL-16 cotton on the Marana Experiment Farm, planted April 12. Treated on July 12, the cotton averaged 23" in height, varying from 12" to 30", and had abundant squares and blooms, with some bolls. Each spray plot was five rows wide and 150' long (0.057 acre).

Methyl parathion, ethyl parathion and Azodrin were applied at the rate of 1.0 lb/A, and Guthion at 0.5 lb/A. The sprays were applied at 9 gal/acre using a Hahn HiBoy (high-clearance ground rig) three nozzles/row, 40 psi, beginning at 7:00 a.m. on July 12.

Weather conditions during the study were warm and dry, with no rainfall on the plots until after the 96-hour sampling. High and low daily temperatures and relative humidities at the Marana Station for that period were: July 12,  $102^{\circ}$ - $71^{\circ}$ , and 17-90%; July 13,  $103^{\circ}$ - $74^{\circ}$ , and 18-70%; July 14,  $100^{\circ}$ - $71^{\circ}$ , and 33-80%; July 15,  $99^{\circ}$ - $71^{\circ}$ , and 40-98%; July 16,  $98^{\circ}$ - $70^{\circ}$ , and 32-95%.

Triplicate residue samples were collected from the inside three rows of each plot immediately after spraying and at 12, 24, 48, 72 and 96 hours. Each sample consisted of ten whole cotton plants removed two inches above the soil level. The samples were brought immediately to the laboratory and minced on a salad chopper.

The methyl and ethyl parathion and Guthion cotton samples were extracted using 50 grams of minced cotton, soxhletted with

Contribution to Regional Project W-45, "Residues of Selected Pesticides--Their Nature, Distribution, and Persistence in Plants, Animals and the Physical Environment." University of Arizona Agricultural Experiment Station journal series # 1963.

Table 1. Organophosphate residues expressed as ppm on whole cotton plants following application by ground equipment.

Hours	Methyl thion	Para- oxon	Ethyl thion	Para- oxon	Guthion	Azodrin
0	106	0.55	117	0.83	40.2	196
12	19.9	0.60	35.7	1.79	30.2	158
24	10	0.29	21.4	1.51	22.4	150
48	6.1	0.10	12.6	1.30	15.7	125
72	5.1	0.06	7.6	0.91	13.9	110
96	3.9	<0.02	5.5	0.58	9.0	71.7

225 ml of chloroform and 25 ml of methanol for 2 hours. The methanol was removed by washing the extracts with water. The extracts were then dried through sodium sulfate and diluted appropriately for gas chromatography using a flame photometric detector sensitive to phosphorus-containing insecticides.

The Azodrin treated cotton was extracted using 25 grams of minced material soxhletted with 250 ml of chloroform for 2 hours. This was followed by drying through sodium sulfate and diluting for GC analysis.

The chromatograph conditions for analysis involved a 6', 4mm I.D. Pyrex glass column, containing 100/120 mesh Chromosorb-W treated with 5% SE-30. Gas flow varied from 95 to 120 cc/min. depending on retention time of the material. Operating temperatures were  $225^{\circ}-250^{\circ}$ ,  $185^{\circ}-229^{\circ}$ , and  $205^{\circ}-210^{\circ}$ , for the injection port, column, and detector, respectively, depending on the compound.

Percent recoveries were Guthion, 96.6; methyl parathion, 88; ethyl parathion 88; and Azodrin, 120%. The results presented in Table 1 have been corrected to compensate for recoveries. Metabolites were measurable only in the case of methyl and ethyl paraoxons.

One analytical anomaly appears in Table 1, and that is the relatively high Azodrin residues. Every possible source of error was examined to explain these high residues, including reextraction and analysis. The characteristic systemic quality of Azodrin could explain the high terminal 96 hour residues, but not the original levels. Consequently the only conclusion which can be drawn is that the amount of Azodrin applied to the cotton was considerably higher than either methyl or ethyl parathion.

Since these residues were determined on a part-per-million basis, these data are only distantly related to the removable or hazardous residue, e.g., that portion which can be removed by hands or clothing of workers in the treated field.