Dislodgable Insecticide Residues on Cotton Foliage: Carbaryl, Cypermethrin, and Methamidophos¹

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The need for establishing safe reentry intervals for persons working in insecticide-treated cotton fields continues. The compilation and comparison of dislodgable residues of various insecticides may aid in establishing these intervals. WARE et al. (1980) compared the dislodgable foliar residues of acephate (Orthene), methomyl (Lannate or Nudrin), permethrin (Pounce), and AC 222,705 on cotton through 96-h post application. They also compared the 72-hr post application disappearance rates from cotton of EPN, methyl parathion, methyl parathion plus Coax (an insect feeding stimulant wettable powder), methyl parathion plus EPN (Trion 6), and thiodicarb (Larvin 500). This paper extends that series by comparing the 192-h post application disappearance rates from cotton of methamidophos (Monitor), methamidophos plus Pro-Tec (carboxylated hydrophilic polymer for extending the residual life of pesticides distributed by Agro-K Corporation, Minneapolis, MN), carbaryl (Sevin XLR), and cypermethrin (Cymbush).

METHODS AND MATERIALS

Test plots were located in a block of 'Deltapine 41' short staple cotton at the Agricultural Experiment Station, Marana, Arizona. Plots consisted of 4 treated rows, with 102 cm spacing, 30.5 m long. Cotton plant heights averaged 80 cm on the day of insecticide applications, August 25, 1981. Sprays were applied at 122 L/ha, 4.0 km/h (2.5 mph), and 276 kPa (40 psi) pressure. The manually drawn sprayer treated two rows, using 3 DC 2-13 Spraying Systems nozzles per row. The pressure was maintained from a 1.1 kg CO_2 tank with a two-stage regulator. The formulation and rate of active ingredient per hawere: Monitor 4 (4 #/gal liquid concentrate) @ 0.55 kg/ha, Monitor 4 @ 0.55 kg/ha plus Pro-Tec (Concentrate 25%) @ 40:1 dilution, Sevin XLR (4 #/gal flowable) @ 2.2 kg/ha, and Cymbush (3 E.C.) @ 0.066 kg/ha.

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Maximum and minimum air temperatures during the test were Aug. 25, $38^{\circ}-19^{\circ}$; Aug. 26, $39^{\circ}-21^{\circ}$; Aug. 27, $40^{\circ}-23^{\circ}$; Aug. 28, $39^{\circ}-21^{\circ}$; Aug. 29, $40^{\circ}-24^{\circ}$; Aug. 30, $38^{\circ}-21^{\circ}$; Aug. 31, $31^{\circ}-22^{\circ}$; Sept. 1, $32^{\circ}-17^{\circ}$; and Sept. 2, $32^{\circ}-18^{\circ}$ C. There was 1.22 cm rainfall the evening of Aug. 30 and another 1.22 cm rainfall the following evening.

Triplicate samples were collected in each treated plot at 0, 72, 144, and 192 h after treatment. Controls (one sample for each solvent used) were collected at 0 and 144 h. Each sample consisted of 100 leaf disks, 2.54 cm diameter, taken singly and consecutively from the top, middle, and bottom portions of plants in all 4 rows. Each sample replicate was extracted in the field with 100 mL of the appropriate solvent. The sample was shaken for 1 min (2 min for carbaryl samples), the extract transferred to a labeled storage bottle, the solvent level marked, and the bottles placed in an ice chest until transported to the laboratory refrigerator. The extracts of methamidophos were dried through anhydrous ${\rm Na_2SO_4}$ and then stored over anhydrous ${\rm Na_2SO_4}$ in storage bottles. The extracting solvents were redistilled ethyl acetate for methamidophos, redistilled dichloromethane for carbaryl, and redistilled hexane for cypermethrin. Controls were extracted with each of the solvents as described above.

All methamidophos-containing samples were analyzed directly by GLC with no cleanup. The Micro Tek gc was equipped with a flame photometric detector in the phosphorus mode. The 102 cm x 4 mm ID Pyrex column contained 2% Reoplex 400 on 80/100 mesh Gas Chrom Q. Nitrogen carrier flow was 60 mL/min and temperatures for inlet, column, and detector were 230° , 185° , and 215° C respectively.

Cypermethrin extracts were cleaned using the procedure described by WARE et al. (1980) for permethrin cleanup. Cypermethrin was analyzed by GLC using the Micro Tek MT-220 equipped with a Ni electron capture detector. The 61 cm x 4 mm ID Pyrex column contained 5% Dexsil 300 on 100/120 mesh Chromosorb W (H.P.). Nitrogen carrier gas flow was 60 mL/min and temperatures were 230° , 220° , and 280° C for inlet, column, and detector respectively.

A 10-mL aliquot of the dichloromethane extract of carbaryl was evaporated to dryness and the residue taken up in <u>ca</u>. 1 mL redistilled ethanol. The ethanol was diluted with 10 mL hexane. A 5.1-cm height of activated Florisil (120°C for 24 h) in a 22 mm ID column topped with 1 cm of Na₂SO₄ was prewashed with 50 mL hexane. The hexane-ethanol solution containing the carbaryl extract was transferred to the column with hexane and ethyl acetate rinses. The ethyl acetate rinses were <15% of the total volume. The carbaryl was eluted with 200 mL of 15% ethyl acetate in hexane at 1 drop per second. The analysis was by HPLC using a TRACOR 990 isochromatographic pump and TRACOR 970 variable wavelength detector at 280 nm. The 25 cm x 4.6

TABLE 1

Dislodgable residue* expressed as $\mu g/cm^2$ of cotton leaf (one surface only) following application by man-pulled ground rig. Marana, AZ. Aug. 25, 1981.

Hours After Appli- cation	Monitor © 4 Methamidophos @ 0.55 kg/ha	Monitor © 4 + Pro-Tec Methamidophos @ 0.55 kg/ha	Cymbush © Cypermethrin © 0.066 kg/ha	Sevin © XLR Carbary1 © 2.2 kg/ha
0 72 144 192	0.57 0.11 0.035 0.025	0.83 0.15 0.043 0.019	0.19 0.16 0.095 0.077	7.2 5.9 0.58 0.26
Controls	<0.003	<0.003	**600.0	<0.26

* No corrections made based on control values.

** Caused by Pydrin impurity in sample extracts.

mm stainless steel column contained Partisil 10 and was fitted with an HC Pellosil precolumn. The carbaryl was eluted with 10% absolute ethanol in hexane at 1 mL per min.

RESULTS AND DISCUSSION

The results are presented in Table 1 expressed as micrograms of toxicant per square centimeter of cotton leaf, one surface only. The 192 h residues show 40% cypermethrin remaining and <5% of the others remaining. The 72 h residues of both cypermethrin and carbaryl are >80%, while the methamidophos residues are ca. 20%. Rainfalls between the 72-144 h and the 144-192 h samplings measurably reduced Sevin XLR formulation of carbaryl bringing the residue level down to 8%. Under the experimental conditions, Pro-Tec did not extend the residual life of methamidophos on Arizona cotton. The methamidophos residue levels with and without Pro-Tec are basically the same, varying by only 1% at the 72- and 144-h samplings (19 vs 18% and 6 vs 5%) and 2% at 192 h. This study again supports the superior persistence of synthetic pyrethroids in Arizona cotton fields.

REFERENCES

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