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Residues of Dimethoate and Its Oxygen Analog on and in Citrus Leaves following a Helicopter Treatment of the Trees with Dimethoate Ultra-Low Volume Concentrate and High Volume Spray

Residues of dimethoate and its oxygen analog (a metabolite of dimethoate) on and in citrus leaves were investigated by a gas chromatographic–flame photometric detector (gc–FPD) procedure following treatment of the trees with (1) a dimethoate ultra-low volume (ULV) concentrate and (2) a high volume (HV) spray, both applied by helicopter at a rate of 1.0 lb/acre. Residues detected 1 day following treatment indicated uniform deposition of the insecticide with both types of application. This was true for all segments of the trees, including the bottom and center sections. The ULV treatment produced higher initial residues than the HV spray, probably due to excessive runoff of the aqueous HV spray solution from the waxy leaf surface. After 14 days weathering, residues on and in leaves from the ULV treated trees were slightly higher than those on leaves from HV sprayed trees.

The insecticide dimethoate, *O,O*-dimethyl *S*-(*N*-methylcarbamoylmethyl) phosphorodithioate [registered as Cygon (American Cyanamid Co.); also known as Rogor (Soc. Montecatini)], is a compound exhibiting both systemic and contact action against certain insect pests attacking plants and animals. Studies by de Pietri-Tonelli and Barontini (1963), Gunther *et al.* (1965), and Woodham *et al.* (1974) and others have shown the importance of this insecticide for the control of citrus pests. However, these tests were performed utilizing conventional ground treatment equipment with an average cost of approximately \$25/acre. Aerial ultra-low volume (ULV) treatment of cotton with dimethoate has been studied by Petty and Bigger (1966). Information is not available concerning helicopter treatment of citrus trees with dimethoate.

This report concerns deposition and disappearance of residues of dimethoate and its oxygen analog on and in citrus leaves following a helicopter treatment of the trees with (1) a Cygon 267 ULV concentrate (35% dimethoate) and (2) a Cygon 267 high-volume (HV) spray. These tests were conducted due to the numerous advantages utilizing this means of application: lower cost (approximately \$8/acre), speed of application, and more uniform coverage of the leaves due to their constant swirling movement as a result of the helicopter's rotor movement.

EXPERIMENTAL SECTION

Type of Citrus, Application Rates, and Procedure. Mature navel orange trees were selected for this study. A 20-acre block was treated with the ULV concentrate at a rate of 1.0 lb of dimethoate per acre. A 156-acre block was treated with the HV spray (3 pints of Cygon 267 diluted to 5 gal with water) also applied at a rate of 1.0 lb of dimethoate per acre. The insecticide was applied with a Bell 47-G helicopter fitted with a 55-gal capacity spray tank, pump, and boom with 48 standard spray nozzles. Helicopter speed was adjusted to 40 mph for the HV treatment with all 48 spray nozzles operating. Speed was increased to 60 mph for the ULV treatment with only 8 spray nozzles operating. Normal operating pressure of the pump was 40 psi for both treatments.

Random trees were selected from each treated block for leaf sample collections. Five trees were sampled in the HV treatment, four in the ULV treated block (Figure 1). Due to heavy rainfall during the 2-day sample collections, alternate trees were sampled for four of the trees in the HV treatment. This was necessary because of the flooding

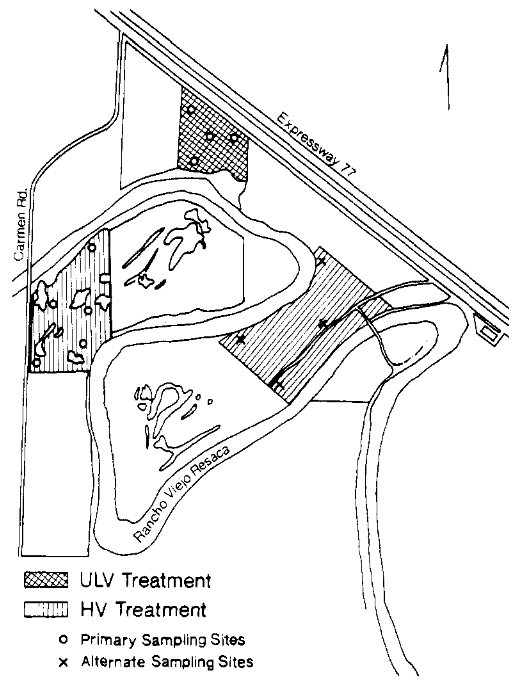

Figure 1. Map indicating location of ULV and HV treatment areas and sampling sites. Samples were collected from the alternate HV sites due to heavy rain preventing collection from regular sites.

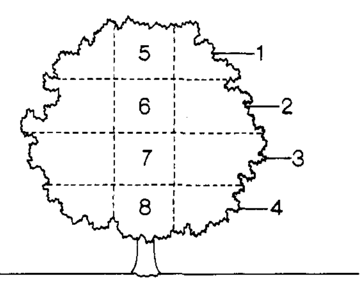

Figure 2. Drawing indicating sampling locations for collection of leaf samples from treated citrus trees. Complete sampling procedure is described in text.

conditions prohibiting sampling of the four routine test trees. Conditions were improved enough for sampling of the original five test trees after 7-days exposure. Each test tree was roughly divided into four sampling areas (Figure 2). Random leaf samples of 100 leaves were collected from selected areas of each tree. Samples 1–4 were collected from the outside perimeter of each tree in each sampling area, beginning with the top of the tree as sample no. 1 and working downward to the bottom area as sample no. 4. Samples 5–8 were collected inside the canopy of the tree, as near the trunk as possible. These samples were collected in the same manner, starting at the top as sample no. 5 and proceeding downward to the bottom area as sample no. 8.

Samples were extracted and analyzed as described in a previous paper by Woodham *et al.* (1974). Briefly this procedure involved extraction of residues by thoroughly macerating the leaves in a Waring Blendor (Waring Products, Inc.) with a solution of 1% (v/v) glacial acetic acid in distilled water, filtering of the extract through glass wool, and then extracting the dimethoate and its oxygen analog from the aqueous solution with dichloromethane. The dichloromethane was evaporated and residues were redissolved in benzene, and then diluted or concentrated

Table I. Dimethoate and Dimethoxon Residues on and in Citrus Leaves following Treatment of the Trees with a Cygon 267 ULV Concentrate[a]

Sampling area	Sampling interval, days	Residue, ppm[b,c]		
		Dimethoate[d]	Dimethoxon[e]	Total
1	1	88.11	0.97	89.08
2	1	58.11	0.47	58.58
3	1	70.29	0.73	71.02
4	1	56.18	0.71	56.89
5	1	44.42	0.78	45.20
6	1	85.90	0.57	86.47
7	1	96.60	0.57	97.17
8	1	59.51	0.43	59.94
1	2	34.25	1.25	35.50
2	2	85.33	1.89	87.15
3	2	44.97	1.73	46.71
4	2	38.62	1.39	40.01
5	2	45.07	1.59	46.66
6	2	33.95	1.53	35.49
7	2	38.90	0.72	39.61
8	2	49.49	1.34	50.83
1	7	3.42	0.92	4.34
2	7	2.30	0.30	2.60
3	7	1.05	0.28	1.33
4	7	1.06	0.41	1.50
5	7	2.72	0.46	3.18
6	7	0.84	0.34	1.18
7	7	0.31	0.15	0.46
8	7	0.63	0.23	0.86
1	14	0.10	<0.05	0.10
2	14	0.11	<0.05	0.11
3	14	0.08	<0.05	0.08
4	14	0.03	<0.05	0.03
5	14	0.65	<0.05	0.65
6	14	0.04	<0.05	0.04
7	14	0.01	<0.05	0.01
8	14	0.04	<0.05	0.04

[a] Treated by helicopter with 1 lb of Cygon 267 ULV concentrate/acre. [b] Corrected for dimethoate and dimethoxon recovery from fortified samples. [c] Average of values obtained on leaf samples from five trees. [d] Lower limits of sensitivity, 0.01 ppm. [e] Lower limits of sensitivity, 0.05 ppm.

to the appropriate volume for injection into the gas chromatograph. No prior cleanup of extracts was required. A series of control samples consisting of a solvent check, untreated sample, and a sample fortified with dimethoate and the oxygen analog was extracted and analyzed utilizing the same procedures used for the test samples. Average recoveries of 95.8 and 60.2% were obtained for the dimethoate and oxygen analog, respectively. All values have been corrected for these recoveries.

The gas chromatograph was a Model MT-220 (Tracor, Inc.) equipped with a dual flame photometric detector (FPD) operated in the sulfur (394 mμ) and the phosphorus (526 mμ) modes simultaneously. Isothermal temperatures were 200, 200 and 250° for the column, detector, and injector, respectively. Flow rates were 50, 15, 40, and 110 cm^3/min for hydrogen, oxygen, air, and nitrogen carrier gas, respectively. The column used was a 6 ft × 0.25 in. o.d. glass tube containing 10% DC-200 on 100–120 mesh Gas Chrom-Q (Applied Science Laboratories, State College, Pa.). Sensitivity was adjusted to obtain half-full scale deflection with a 5-ng injection of dimethoate. The column was preconditioned by making consecutive 10-μl injections of an extract of citrus leaves fortified with dimethoate and its oxygen analog until optimum sensitivity and reproducibility were attained between injections.

RESULTS AND DISCUSSION

Residues of dimethoate and its oxygen analog on and in leaves from trees treated with the ULV concentrate were

Table II. Dimethoate and Dimethoxon Residues on and in Citrus Leaves following a Cygon 267 High Volume Treatment of the Trees[a]

Sampling area	Sampling interval, days	Residue, ppm[b,c] Dimethoate[d]	Dimethoxon[e]	Total
1	1	31.19	0.72	31.91
2	1	24.87	0.51	25.38
3	1	13.77	0.61	14.38
4	1	18.09	0.62	18.71
5	1	17.82	0.44	18.26
6	1	7.26	0.13	7.39
7	1	5.98	0.17	6.06
8	1	6.67	0.34	7.01
1	2	16.42	0.69	17.11
2	2	11.68	0.51	12.19
3	2	9.20	0.36	9.56
4	2	8.37	0.30	8.67
5	2	10.50	0.34	10.82
6	2	7.81	0.24	8.05
7	2	3.80	0.20	4.00
8	2	4.49	0.25	4.74
1	7	1.05	0.17	1.22
2	7	0.42	0.07	0.49
3	7	0.19	0.11	0.29
4	7	0.54	0.13	0.67
5	7	0.74	0.14	0.88
6	7	0.05	<0.05	0.05
7	7	0.06	<0.05	0.06
8	7	0.09	<0.05	0.09
1	14	0.02	<0.05	0.02
2	14	0.07	<0.05	0.02
3	14	0.01	<0.05	0.01
4	14	0.07	<0.05	0.07
5	14	0.02	<0.05	1.02
6	14	0.01	<0.05	0.01
7	14	0.01	<0.05	0.01
8	14	0.01	<0.05	0.01

[a] Treated by helicopter with 1 lb of Cygon 267 high volume spray/acre. [b] Average value of five treated trees. [c] Corrected for dimethoate and dimethoxon recovery from fortified samples. [d] Lower limits of sensitivity, 0.01 ppm. [e] Lower limits of sensitivity, 0.05 ppm.

significantly higher than those on and in leaves from the HV treated trees. This was true for all segments of the tree for the entire 14-day test with the exception of sampling area no. 4, which showed higher residues for the HV (0.07 ppm *vs.* 0.03 ppm) than for the ULV residues and sampling area no. 7, which showed equal residues, both after 14-days exposure (Tables I and II). The large differences in residues between the ULV and HV treatments were probably due to excessive runoff of the aqueous HV spray solution from the waxy leaf surface.

A more uniform deposition of dimethoate and its oxygen analog was noted for the ULV treatment than for the HV spray. Average ULV residues ranged from 45.20 ppm (sampling area no. 5) to 97.17 ppm (sampling area no. 7), while HV residues produced a range of 6.06 ppm (sampling area no. 7) to 31.91 ppm (sampling area no. 1). This was true for each sampling interval, except for the 14-day samples where residues were almost equal for both the ULV and HV samples.

Residues had almost disappeared after 14-days weathering. The highest average residue was 0.68 ppm on and in leaves from ULV sampling area no. 5; the HV leaves showed a high level of 0.07 ppm on and in samples from sampling area no. 2 and 4.

Results demonstrate the potential for utilization of helicopters in the aerial treatment of citrus and other fruit trees for the control of insect pests. This is indicated by the uniform deposition of dimethoate obtained, speed of treatment, and low cost of treatments.

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