Aldicarb Residues in Oranges, Citrus By-Products, Orange Leaves, and Soil after an Aldicarb Soil-Application in an Orange Grove

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A 15% granular formulation of aldicarb (Temik 15G) was soil applied to field plots of Valencia and navel oranges at 2.5, 5.0, 10, and 20 lb of active ingredient per acre. Total carbamate residues (aldicarb, its sulfoxide and sulfone) remaining in the soil and translocated to the leaves and fruit are reported. Residues were found in both rind and pulp. Total carbamate residues in molasses and dried citrus pulp, both used to supplement animal feed, orange juice, and orange oil resulting from processing residue-bearing fruit for by-products are also reported.

Aldicarb [2-methyl-2-(methylthio)propionaldehyde *O*-(methylcarbamoyl)oxime, Temik, Union Carbide UC-21149] is a broad-spectrum soil-applied systemic pesticide for the control of insects, mites, and nematodes. In moist soil, aldicarb is absorbed by plant roots and translocated upward to other parts of the plant. The first commercial sales of aldicarb in the United States were in 1970 following the federal registration of a label for use on cotton. In the U.S., aldicarb is currently labeled for use on cotton, peanuts, potatoes, sugar beets, sugarcane, sweet potatoes, and several ornamental and nursery plants.

Test data indicate that aldicarb may be effective against a number of pests (mites, scale, thrips) attacking young and mature citrus trees (Tashiro and Beaver, 1967; Boling and Dean, 1968; Tashiro et al., 1969; Beaver et al., 1970; Shaw et al., 1970). Field studies are currently underway to evaluate the effectiveness of aldicarb against citrus nematodes and phytophagous citrus pests. Registration of aldicarb for use on citrus requires residue data to show that high levels of toxic residues do not persist in the orchard environment, especially in fruit, at recommended dosages used. Reported here are results of analyses of soil, leaves, fruit, and citrus by-products for total carbamate residues of aldicarb (aldicarb, aldicarb sulfoxide, aldicarb sulfone) resulting from a soil application.

EXPERIMENTAL SECTION

Treatment. Temik 15% granular formulation (15G) was applied to experimental plots of 11-year-old Olinda Valencia orange on Troyer citrange root stock on the Irvine Ranch, Tustin, Calif. It was applied to the soil on April 8, 1974 in perpendicular 4-ft bands around the periphery of each tree using a spreader and the area was rototilled to a 2- to 4-in. depth to mix the formulation with the soil. Two days later, portable low-head sprinklers applied over 2 in. of water over a 24-h period to leach the pesticide into the root area. The plots were subsequently furrow irrigated on the regular ranch schedule. Nine-tree (3 × 3) plots, replicated three times, were used for each of the four dosages. Plots I, II, III, and IV were treated at 2.5, 5.0, 10.0, and 20.0 lb of active ingredient (AI) per acre, respectively; plot II received a second 5.0 lb of AI per acre treatment 36 days after the initial application. The per acre amounts were applied in application bands around the trees which represented approximately 50% of the orchard floor area.

On March 19, 1975, 345 days after initial application, Temik 15G was applied to plot I at 20 lb of AI per acre only on the two furrow irrigated sides of the trees and to plots III and IV at 10 and 20 lb of AI per acre, respectively, in the same manner as the first application. Plot II was discontinued. The rototilled plots were sprinkler irrigated for 24-h on March 20 and again on March 22. The orchard was then returned to furrow irrigation.

Field Sampling. Each soil sample consisted of eight cores, 12 in. in depth, taken with a 1-in. diameter soil sampling tube. Four trees from each replicate, forming a square, were used for sampling; two cores were taken from the inner side of each tree near the center of the treated band and near the periphery of the tree. Fruit samples consisted of 32 fruit each, four from each of eight trees, with one from each quadrant of each tree at about shoulder height. Leaf samples consisted of five 1-in. diameter leaf disks taken from each of eight trees using the system of Westlake et al. (1973); newly mature leaves were selected for sampling. Duplicate leaf samples were taken from each replicate plot, but only one sample per replicate plot was taken of fruit and soil. Duplicate laboratory subsamples were used for fruit and soil. Fruit for making by-products were picked from the 20 lb band-treated plots and were processed by the Sunkist Growers, Inc. plant in Corona, Calif.; 1180 lb of mature treated fruit were processed on June 10, 1975.

Laboratory Processing. Soil samples were air dried, pulverized in a mortar if necessary, and mixed in a Twin-Shell blender. The fruits were peeled (Gunther, 1969); the rind was chopped in a Hobart food cutter and pulp (oranges after rind removal) was sampled by cutting out cylinders (Gunther, 1969). All samples were stored at about −4 °C until analyzed.

Analysis. The toxic residues of aldicarb in biological substrates are composed of aldicarb and its sulfoxide and sulfone. All three of these compounds were determined as a total residue by first oxidizing aldicarb and its sulfoxide to the sulfone with peracetic acid and then determining total aldicarb sulfone by gas chromatography.

All samples, except orange oil, were analyzed by the method furnished by Union Carbide Corporation, South Charleston, W. Va., entitled "A Method for the Determination of Total Toxic Aldicarb Residues in Citrus Fruit, July 1975". This method was satisfactory for soil samples as well as the plant parts. Analysis of citrus oil utilized the Union Carbide method "A Method for the Determination of Total Toxic Aldicarb Residues in Peanut Oil by Gas Chromatography, February, 1973". The final determinations were made with a Tracor MicroTek MT-220 gas chromatograph as described in the methods except that a 1.8 m × 4 mm i.d. glass column, packed with 5% Reoplex 400 on 80/100 mesh Gas-Chrom Q and operated at 190 °C with a carrier gas (nitrogen) flow rate of 80 mL/min, was used. The detector was a Melpar flame photometric detector (FPD) incorporating a 394-nm filter specific for

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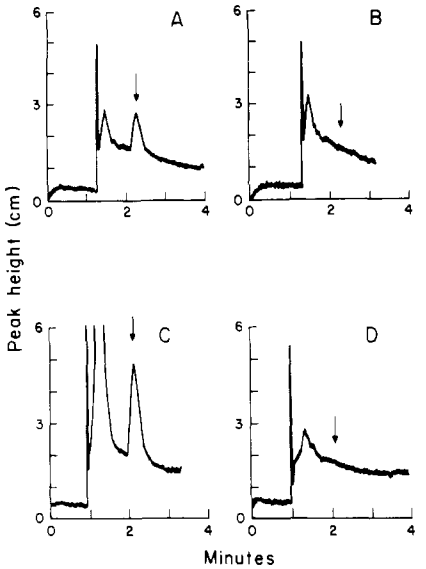

Figure 1. Gas chromatographic responses (FPD) for: (A) orange rind fortified at 0.05 ppm with aldicarb, (B) control rind, (C) orange pulp fortified at 0.06 ppm with aldicarb, and (D) control pulp. Extract injected was equivalent to 75 mg of rind and 100 mg of pulp.

sulfur-containing compounds. In our laboratory the Reoplex column gave better separation of the "aldicarb sulfone" peak [the response results from the thermal decomposition product, 2-methyl-2-(methylsulfonyl)-propionitrile (Knaak et al., 1966)] from early-eluting interference than the Carbowax column specified in the method. The retention time for the Reoplex column was 2.3 min. Chromatograms for rind and pulp fortified at 0.05 ppm with aldicarb prior to extraction are shown in Figure 1.

The minimum detectable levels for the analytical procedure used varied from 0.01 to 0.04 ppm and were dependent on the substrate analyzed and the daily variations in the gas chromatographic response. All values reported have been corrected for recovery as determined from fortification of control samples with aldicarb prior to analysis. Recovery values for rind, pulp, leaves, and soil were 97 ± 16, 97 ± 13, 96 ± 6, and 87 ± 5%, respectively.

RESULTS AND DISCUSSION

Figure 2 shows the residue levels found in the orange rind. In plot IV (20 lb/acre) detectable residues were found in the 14-day samples and a maximum residue level of about 0.25 ppm was reached at about 45 days after soil treatment. Following this there was a gradual reduction to about 0.1 ppm after 154 days. In plot III (10 lb/acre) detectable residues appeared in the 28-day samples and reached a peak of about 0.1 ppm at about 45 days after treatment. Residues were generally less than half of that for plot IV which received twice the treatment rate. Plot II which received the same amount as plot III, but in two treatments, peaked at the same level as plot III after the second treatment (71-day sampling), then dropped off. Rind residues for plot I treated at 2.5 lb of AI per acre were always below the detectable limit except for two of the three 35-day samples that contained 0.04 and 0.05 ppm.

Residues in the pulp (edible portion including juice and tissue) did not reach detectable levels in plots I, II, and III for any sample during the entire 154-day experimental period. In plot IV detectable residues appeared in the pulp of the 28-day samples and a residue of 0.03 to 0.05 ppm persisted from the 52nd day to the end of the experimental period (Table I).

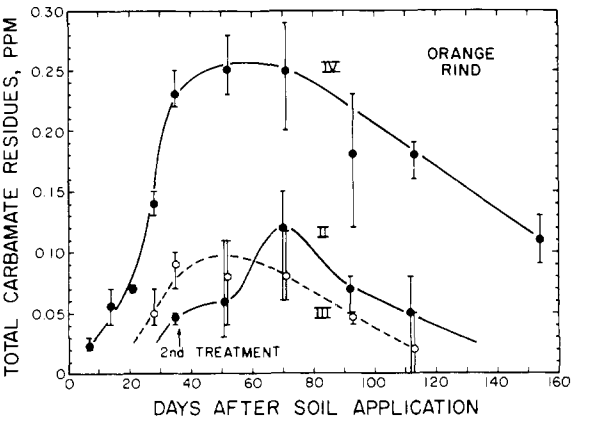

Figure 2. Total carbamate residues found in orange rind after soil treatment with aldicarb (April 1974). Plots II, III, and IV were treated at 5, 10, and 20 lb of AI per acre, respectively; plot II was retreated on day 36 at 5 lb of AI per acre. The circles give the mean value obtained from three replicate field plots while the vertical bars give the ranges of values found.

Table I. Total Carbamate Residues (ppm) of Aldicarb in Valencia Orange Pulp Obtained from Plot IV Treated (April 1974) at 20 lb of AI per Acre with Aldicarb

Days after treatment	Replicate A	Replicate B	Replicate C	Mean
7	<0.02	<0.02	<0.02	<0.02
14	<0.02	<0.02	<0.02	<0.02
21	<0.02	<0.02	<0.02	<0.02
28	0.02		0.02	0.02
35	0.02	0.02	0.03	0.02
52	0.04	0.04	<0.02	0.04
71	0.02	0.02	0.05	0.03
93	0.05	0.04	0.03	0.04
113	0.05	0.05	0.05	0.05
154	0.05	0.04	0.03	0.04

Samples of fruit were taken 245 days after application from plot IV which represented fruit which had been "green" when the aldicarb had been applied. For the three plot replicates, rind residues were 0.13, 0.14, and 0.13 ppm and pulp residues were 0.06, 0.05, and 0.04 ppm. Thus, the rind residue was approximately one-half and the pulp residue about equal to the maximum value found in the fruit which had been mature when the aldicarb application was made.

As Valencia oranges are 18.7 ± 6.3% rind by weight (Gunther, 1969), whole-fruit residues can be approximated by adding 20% of the rind residue value to 80% of the pulp residue value. Thus, a fruit with a rind and pulp residue of 0.29 and 0.02 ppm, respectively, contains approximately 0.07 ppm. On March 1, 1976, the U.S. Environmental Protection Agency (EPA) granted to Union Carbide Corporation a temporary tolerance of 0.3 ppm total toxic aldicarb residues in oranges resulting from application of Temik Aldicarb Pesticide in conjunction with an experimental use permit which expires Feb 24, 1977.

Figure 3 shows the residue levels found in the newly mature orange leaves. Residues on a ppm basis were much higher in the leaves than in the fruit. Residues were found in the 7-day leaf samples for all application rates and reached a maximum value in about 50 days after application. The foliar residue pattern shown in Figure 3 is qualitatively similar to that found for the rind shown in Figure 2. As observed with the rind, the residue levels in the leaves from the 10-lb treatment are less than one-half those from the 20-lb treatment. Foliar residues declined

Table II. Total Carbamate Residues (ppm) in Soil, Leaves, and Fruit after Soil Treatment with Aldicarb (March 1975)[a]

		10 lb of AI per acre (four-side)			20 lb of AI per acre (four-side)			20 lb of AI per acre (two-side)	
Substrate	Sample	Range	Mean	1974[b] mean	Range	Mean	1974[b] mean	Range	Mean
Soil	Pretreat	<0.01-0.01	<0.01		<0.01-0.14	0.04		<0.01	<0.01
	35-day	0.26-0.69	0.52	1.02	0.94-1.45	1.20	2.45	0.27-1.16	0.69
	118-day	0.12-0.36	0.20	0.14	0.19-0.52	0.42	0.21	0.10-0.37	0.23
Leaves	Pretreat	<0.01	<0.01		0.7-1.2	0.9		<0.01-0.4	0.2
	35-day	2.4-5.0	3.7	2.7	9.6-12.1	10	11	2.4-4.2	3.4
	118-day	4.1-7.0	5.4	2.7	11-22	15	12	5.7-7.6	6.7
Rind	Pretreat	<0.01-0.01	<0.01		0.01-0.05	0.04		<0.01	<0.01
	35-day	0.05-0.11	0.09	0.09	0.14-0.38	0.28	0.23	0.07-0.12	0.10
	118-day	0.06-0.16	0.10	0.02	0.18-0.36	0.25	0.18	0.08-0.11	0.10
Pulp	Pretreat	<0.01-0.02	<0.01		<0.01-0.02	<0.01		<0.01	<0.01
	35-day	<0.02-0.03	0.01	<0.02	0.02-0.04	0.03	0.02	0.01-0.03	0.02
	118-day	<0.02-0.04	0.03	<0.02	0.03-0.09	0.05	0.05	0.02-0.05	0.04

[a] Applied March 19, 1975, to soil in a Valencia orange grove located on the Irvine Ranch, Tustin, Calif; sprinkler irrigation for 24 h on March 20 and again on March 22; four replicate plots. [b] Applied April 8, 1974. These values are the data used to construct Figures 1, 2, and 3 and Table I.

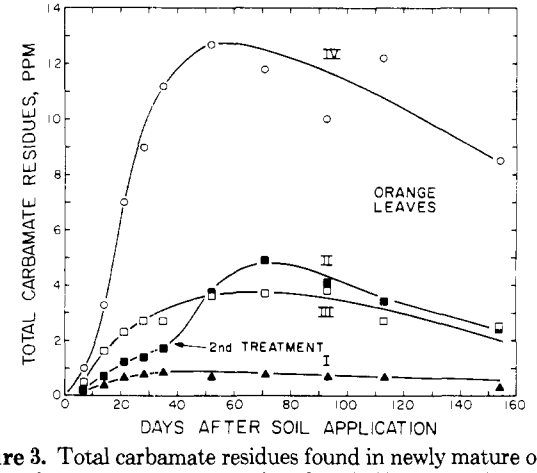

Figure 3. Total carbamate residues found in newly mature orange leaves after soil treatment with aldicarb (April 1974). Plots I, II, III, and IV were treated at 2.5, 5, 10, and 20 lb of AI per acre, respectively; plot II was retreated on day 36 at 5 lb of AI per acre. Symbols represent the mean values obtained from three replicate field plots.

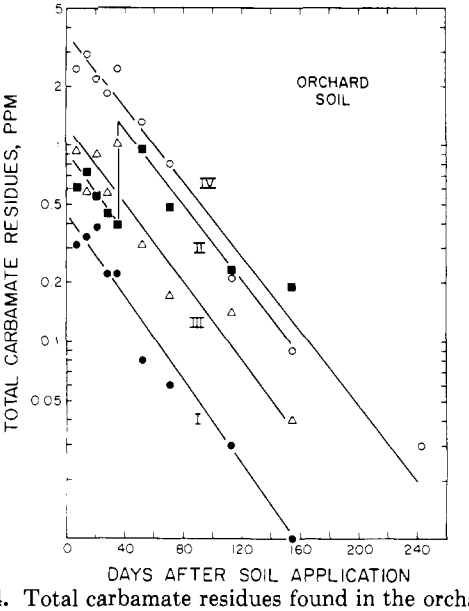

Figure 4. Total carbamate residues found in the orchard soil after soil treatment with aldicarb (April 1974). Plots I, II, III, and IV were treated at 2.5, 5, 10, and 20 lb of AI per acre, respectively; plot II was retreated on day 36 at 5 lb of AI per acre. Symbols represent the mean values obtained from three replicate field plots.

more slowly relative to the rind residues. The amounts present at 154 days were 30 to 70% of the maximum levels attained. Leaves representing old growth and newly mature growth were sampled from plot IV 243 days after application. The old growth leaves contained 1.95 ppm which represented only 15% of the maximum level attained. The newly mature growth contained 2.52 ppm which was only 30% higher than that found in the old growth leaves.

Figure 4 shows the residue level in the soil over the 154-day study period. The data show that the total carbamate residues of aldicarb declined by what appears to be first-order kinetics in all plots with a half-life of approximately 32 days. Andrawes et al. (1971) found that in cultivated soil aldicarb is a compound of short persistence; it decreased from 10.8 to 0.16 ppm in 14 days. They found the major product at 7 days was aldicarb sulfoxide; its soil concentration remained at 1.5 to 1.7 ppm for 30 days and then began to decline. The aldicarb sulfone concentration increased to a maximum value of only 0.7 ppm at 30 days and also began to decline. After 90 days the total carbamate residue in soil was 0.04 ppm. The report that aldicarb is readily converted to the sulfoxide and sulfone in moist soil suggests that the data points in Figure 4 most likely represent the soil residues of aldicarb sulfoxide and sulfone and their dissipation.

The soil residues in plot IV are much more than twice that for plot III and explains the generally higher-than-expected residues found in fruit and foliage obtained from plot IV.

Table III. Total Carbamate Residues (ppm) of Aldicarb in Oranges and Orange By-Products[a]

Substrate	85-day	169-day
Whole fruit	0.18[b]	0.10
Rind	0.42	
Pulp	0.12	
Wet citrus pulp (includes peel frits and rag)		0.09
Dried citrus pulp	0.43[c]	0.07
Dilute juice (1×)	0.18	0.09
Concentrate juice (3×)		0.26
Molasses	0.04	<0.02
Oil	<0.02	<0.02

[a] Oranges were grown on the Irvine Ranch, Tustin, Calif., and were sampled 85 and 169 days after soil treatment with Temik 15G at 20 lb of AI per acre on March 19, 1975. Fruits were processed by the Sunkist Growers, Inc. plant, Corona, Calif. [b] Estimated by taking 80% of pulp value and 20% of rind value. [c] Moisture determination gave 9% water.

Table IV. Total Carbamate Residues (ppm) of Aldicarb in Navel Orange Rind and Pulp 178 Days after Soil Application

Treatment, lb of AI per acre	Rind				Pulp			
	A	B	C	Mean	A	B	C	Mean
2.5	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
5	<0.02	0.03	0.04	0.03	<0.02	<0.02	<0.02	<0.02
5[a]	0.06	0.06	0.07	0.06	0.03	0.04	0.05	0.04
10	0.05	0.06	0.05	0.05	0.03	0.08	0.04	0.05
20	0.10	0.12	0.12	0.11	0.06	0.06	0.03	0.05

[a] Plot was retreated after 24 days at 5 lb of AI per acre.

The 1974 Irvine Ranch Valencia orange plots were retreated with Temik 15G at the 10 and 20 lb of AI per acre rate on March 19, 1975, 345 days after the first treatment. An additional plot was treated at the 20-lb rate only on the two furrow irrigated sides of the tree. Residue data are in Table II. Pretreatment values show that residues from the first application had dissipated for the 10-lb plot and a small amount of carry-over occurred in the 20-lb plot. The residue data for the retreated 10- and 20-lb plots show good agreement with the previous year's data which are also given in Table II. The 20-lb two-side treatment plot gave residue levels similar to the 10-lb four-side plot rather than to the 20-lb four-side plot. The two-side treatment would be a more desirable method of application for the grower as less time and labor would be required. Beaver et al. (1970) reported aldicarb to be ineffective in controlling citrus red mite on mature navel orange trees when it was applied as sidedressing in furrows adjacent to the irrigation furrows.

Fruit from the 20 lb/acre plot were sampled 85 and 169 days after treatment and processed into by-products by the Sunkist Growers, Inc. plant in Corona, Calif. The by-products were dried citrus pulp used to supplement cattle feed, orange juice, molasses used to supplement cattle and chicken feed, and orange oil. Data are given in Table III. It was hoped that the study would enable the prediction of residue levels in by-products from whole fruit residue data. Dried citrus pulp residues do not correlate with whole fruit data. Single strength orange juice residues were coincidentally identical with whole fruit residues. Flash evaporation in vacuo of juice at 150–170 °C to prepare concentrate juice simply concentrated the residues to the same degree. Minimal residues appeared in molasses and detectable levels did not appear in oil, which is consistent with the water-soluble nature of aldicarb. Analysis of the same samples listed in Table III by the Union Carbide Corporation laboratory in South Charleston, W. Va., gave essentially identical values. A temporary food additive tolerance (expires Feb 24, 1977) of 0.6 ppm of total toxic aldicarb residues in dried citrus pulp was established March 1, 1976.

Temik 15G was soil-applied to experimental navel orange plots on the Penrod Ranch, Delano, Calif., on July 22, 1974, as a circular 2-ft band at the peripheral edge of each tree. This treatment procedure resulted in the application of the per-acre dosage amounts to only about 20% of the actual orchard area. The plots were rototilled to a 4-in. depth and normal sprinkler irrigation followed treatment. Harvest fruit samples were taken on Jan 16, 1975, 178 days after soil treatment. Residue data for the rind and pulp are given in Table IV. The values for the Delano navels after 178 days did not differ significantly from those earlier obtained for the Tustin Valencias. For the 2.5, 5 + 5, 10, and 20 lb treatments, the rind residues were <0.02, 0.06, 0.05, and 0.11 ppm, respectively, for the navels (Table IV) and <0.03, <0.04, <0.04, and 0.11 ppm, respectively, for the Valencias (Figure 2). The corresponding pulp values were <0.02, 0.04, 0.05, and 0.05 ppm, respectively, for the navels (Table IV) and <0.02, <0.02, <0.02, and 0.04 ppm, respectively, for the Valencias (Table I).

Temik 15G was also applied at the rates shown in Table IV to experimental navel orange plots on the Doty Ranch, Arlington, Calif. It was applied to the soil using a Scott spreader on May 31, 1974, as a circular 18-in. band at the peripheral edge of each tree; normal sprinkler irrigation followed the treatment. By this procedure the applications at the per-acre rates indicated were actually made to only about 20% of the orchard surface; the orchard soil area treated is dependent on the number of trees/acre and on the size of the tree which governs the radius of the treated band. The 5-lb retreatment plot was retreated on June 28, 1974. Harvest fruit samples were taken on Dec 10, 1974, 193 days after initial application. Rind residues were all below the detectable limit of 0.04 ppm and pulp residues were all below the detectable limit of 0.02 ppm except for the 20-lb treatment plots whose pulp sample replicates contained 0.03, 0.03, and 0.02 ppm. Unlike the other plots the aldicarb was not rototilled into the soil after application. The sprinkler irrigation possibly was ineffective in leaching the pesticide down into the soil where it could be taken up by the roots.

Extensive field tests are required to determine what rates of aldicarb are necessary to achieve pest control and what method of application is best in terms of grower acceptability and efficacy. The systemic action of aldicarb circumvents the worker reentry problem associated with the organophosphorus pesticides and may be largely compatible with biological control programs as parasites and predators should not be directly affected. Residue levels in fruit will depend upon the rate of application ultimately selected. The results of bioassay trials completed or in progress suggest that regulations governing the use of this material and considerations of acceptance for use by citrus groups should be based on the dosage rate of 10 lb of AI per acre and possibly the 20 lb of AI per acre rate. Currently, registration for use in citrus groves is being sought by Union Carbide Corporation only for the 5 and 10 lb of AI per acre rates.

ACKNOWLEDGMENT

The technical assistance of D. C. G. Aitken, R. C. Baines, T. M. Dinoff, M. E. Düsch, C. Gericke, J. R. O'Neal, J. L. Pappas, and J. Virzi are gratefully acknowledged. The cooperation and assistance of P. H. Darst, A. A. Puech, and R. R. Romine of Union Carbide Corporation are also gratefully acknowledged.

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Received for review December 17, 1976. Accepted March 11, 1977. A grant-in-aid from Union Carbide Corporation and support by the Citrus Advisory Board is gratefully acknowledged.

Residues of Dimethoate and Dimethoxon on Sweet Cherries Following Air Carrier Application

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Dimethoate was applied to sweet cherries at two Oregon locations using both the emulsifiable concentrate and the wettable powder formulations. The applications were made with an air carrier sprayer at rates ranging from 1.23 to 2.50 lb of active ingredient/acre. Only traces of dimethoate and its principal metabolite, dimethoxon, were present at harvest, 28 to 35 days after treatment. Total residues were reduced to levels below 2 ppm in 0 to 8 days after application. No differences in the initial residues or the rate of disappearance were observed between the emulsifiable concentrate and wettable powder formulations. This study indicates that dimethoate can be used for western cherry fruit fly control without excessive harvest residues.

Dimethoate [*O,O*-dimethyl *S*-(*N*-methylcarbamoylmethyl) phosphorodithoate] is registered on pome fruits for the control of a variety of insect pests in the United States and Canada. Following investigations of Banham (1974) in Canada, we evaluated efficacy of dimethoate for western cherry fruit fly (*Rhagoletis indifferens* Curran) control on sweet cherries in Oregon (Zwick et al., 1975) and found that one application of dimethoate provided excellent protection over an entire season. Presently, dimethoate is registered for control of cherry fruit fly in Canada but not in the United States. We have been attempting to collect sufficient efficacy and residue data to support registration of this compound on cherries in the United States. Since previous residue studies of MacNeil et al. (1975) and Zwick et al. (1975) were based on hydraulic handgun applications which are not in general commerical use, we are presenting here the results of dimethoate residue analyses resulting from air carrier applications as would be applicable to commercial cherry orchards. In addition, residues resulting from the use of a wettable powder formulation are compared with those resulting from an emulsifiable concentrate application.

MATERIALS AND METHODS

Treatment and Sampling of Crop. In 1975 studies were conducted in two eastern Oregon areas, Hood River and The Dalles, both of which have commercial cherry acreages. Two rates of two dimethoate formulations [Cygon brand, 2.67 lb of active ingredient (AI)/gal of emulsifiable concentrate (EC) and 25% AI wettable powder (WP)] were applied once in each area to several varieties (Bing, Royal Anne, Van) of sweet cherries. Applications were made with a Berthoud VT 1500 Model air carrier sprayer calibrated to deliver 370 gal/acre (gpa) and 500 gpa in The Dalles and Hood River orchards, respectively. The differences in total gallonage applied per acre were due to the 26 ft rows and 1.40 mph tractor speed in The Dalles as compared with 20 ft rows and 1.36 mph speed in Hood River. Fan rpm, hydraulic pump pressure, and discharge nozzling remained constant for all applications. Although the dilution rates (lb of AI dimethoate/100 gal) for comparable plots were identical in each location, the lb of AI per acre applied were different in each location due to the different total gallonages applied (Table I). Individual plots were 3–12 trees in The Dalles and 4–10 trees in Hood River.

Applications in each area were made within a week after western cherry fruit fly had emerged. This was June 5 in The Dalles and June 9 in Hood River, at which time fruit was about 0.5 in. in diameter. About 1 lb random samples of fruit, three per plot at each sampling date, were individually bagged in plastic containers and stored at −10 °C until analysis. Samples were taken 0, 3, 7, 14, and 28 days after treatment and also at harvest. Control samples were from unsprayed trees at each location.

Residue Analysis. The analytical method used was based on that developed by Stellar and Pasarela (1972). The fruit was subsampled in the laboratory as received, and randomly selected 100-g aliquots, were taken for extraction. In most cases the three replicates taken were combined, mixed well, and then one subsample taken. One subsample per replicate was taken from one plot at each location. Whole fruits were macerated with 400 mL of acetone in the presence of 50 g of anhydrous sodium sulfate for 5 min using an Omnimixer. The extract was recovered by filtering with suction, and the extraction jar, pulp, and filter paper were washed with additional 50 mL of acetone. The acetone extract was concentrated to 100 mL on a steam bath, 100 mL of water added, and the aqueous solution extracted with three 100-mL portions of dichloromethane. The dichloromethane extract was dried with 50 g of anhydrous sodium sulfate and purified with 3 g of activated charcoal (Nuchar C-190-N). The absorbent

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