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RESIDUES OF 2,4-DICHLOROPHENOXYACETIC ACID HERBICIDE (2,4-D) IN VALENCIA ORANGES FOLLOWING POST-HARVEST DIP[1]

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Abstract. **2,4-Dichlorophenoxyacetic acid (Dow Chemical Company Formula 40) as a postharvest dip has been shown to be effective in reducing stylar end breakdown and** Alternaria **rot in mature Valencia oranges. Residues were determined in the pulp and peel of whole fruit after storage at 4.4°C for 0, 8 and 10 weeks; residues ranged from 1.0 to 6.8 ppm for peel and 0.01 to 0.17 ppm for pulp.**

2,4-Dichlorophenoxyacetic acid (2,4-D) has long been used as a postharvest application to extend storage life of lemons (3). The U.S. Environmental Protection Agency (U.S.E.P.A.) has now approved a tolerance of 5.0 ppm of 2,4-D applied as a water solution of the alkanolamine salts (Dow Formula 40) for all types of citrus, in order to prevent peel necrosis due to senescence and desiccation, and to aid in control of postharvest decay (3). Reduced decay is due to improved disease resistance by the fruit. This is in contrast to action on pathogens by fungicides, such as benomyl (Benlate®), which have been shown to be effective in controlling varied types of decay causing organisms in citrus (2).

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This report describes the residues which were measured and reported to the U.S.E.P.A. in order to obtain the above described tolerance.

Materials and Methods

Approximately 3 cartons (4/5 bushel) were treated at 0, 500 and 1000 ppm as a one-half minute dip in water solutions of 2,4-D alkanolamine salt (Dow Formula 40). The fruit were not dried but sent immediately to cold storage (4.4°C) for 0, 8 and 10 weeks after which they were held at −8°C until residue analyses were performed.

Pulp and peel were analyzed separately by hydrolyzing 25 g of each in 35 ml of pH 12 NaOH solution at 100°C for 15 minutes. This releases all conjugates which may be present giving 2,4-D sodium salt. The solns were cooled and adjusted to pH 1 with 5N H_2SO_4, after which they were extracted with 3 20 ml portions of ethyl ether; the extracts were combined. After the ether fraction was reduced to 20 ml (ambient temp.) it was extracted 4 times with 5 ml portions of 0.2 M K_2HPO_4; these portions were combined and adjusted to pH 1 with 5N H_2SO_4 after which the 2,4-D was extracted into 3 5 ml portions of benzene, the portions again combined. After drying with Na_2SO_4 the benzene was gently evaporated to dryness at room temp.

The butoxyethyl ester was made of the 2,4-D free acid by adding 0.1 ml 5% acetyl chloride in butoxyethanol and heating at 100°C for 15 minutes. The esterification solution was diluted with 2 ml of benzene, the mixture washed twice with 2 ml portions of 0.2 M NaOH (discarded) then twice with 2 ml portions of water (discarded). Anhydrous Na_2SO_4 was added and the solution analyzed within 24 hours by electron capture gas chromatography.

Gas chromatography was performed on a Varian model 1200 equipped with a tritium electron capture detector, a 1.8 meter x 2 mm I.D. glass column packed with 10% DC-200 operated at 210°C with a carrier flow of 60 ml/min.

Results and Discussion

Table 1 illustrates that high recoveries were obtained throughout a wide range of spiked sample concentrations.

Table 1. Recoveries of fortified control Valencia orange peel and pulp.[z]

Sample	Fortification	% Recovery
Peel	0.00 ppm	—
"	0.05	90
"	0.10	71
"	0.25	76
"	0.50	80
"	1.0	82
"	2.0	85
"	4.0	84
"	8.0	82
Pulp	0.00	—
"	0.05	87
"	0.10	80
"	0.25	86
"	0.50	94

[z]Average of 3 separate determinations.

Table 2 presents the residues observed in the control and treated 'Valencia' oranges. The edible portion exhibited a maximum of 0.17 ppm (0 week, 1000 ppm), while the peel of this sample was also the highest (6.8 ppm). Storage time at 4.4°C had a pronounced effect on the 2,4-D levels observed, a steady decline being evident. Also, as expected, the 1000 ppm treatment produced consistently higher residues than did the 500 ppm. At no time did the recommended concn of 500 ppm give residues in either part of the orange above the listed 5 ppm tolerance.

Table 2. 2,4-D residues in Valencia oranges following a half-minute dip.[z,y]

Storage	Pulp		Peel	
	500 ppm	1000 ppm	500 ppm	1000 ppm
0 week	0.042 ppm	0.170 ppm	1.9 ppm	6.8 ppm
8	0.015	0.041	1.0	4.4
10	0.010	0.044	1.1	4.1

[z]Average of two separate determinations.
[y]Average RSD of duplicates 7.7%.

Note that the label for postharvest use on citrus fruits generally, i.e. over and beyond the traditional use solely on lemons, was on a "24(c) state label," thus restricting its use to the state of Florida.

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WEED CONTROL PRACTICES IN THE INDIAN RIVER CITRUS AREA[1]

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Abstract. **Forty-one growers representing 32,070 ha (79,249 acres) of citrus in Indian River, St. Lucie, Martin and Palm Beach counties were interviewed and 13,456 ha (33,250 acres) of groves were inventoried in 1976 to determine current weed control practices and their effects on weed populations. All growers used some type of rotary mower as the primary means of weed control in middles between tree rows. A sickle bar mower was the most common type of mechanical equipment for under-tree weed control. Three growers had never used herbicides, 16 were on a regular program, 18 were using chemicals to a limited extent, 4 had stopped for various reasons and only 10 applied them to their entire acreage. A combination of bromacil and diuron with additives applied once or twice a year was the most common herbicide treatment. The weed control program generally received a low priority in the production schedule, and many aspects were therefore erratic. There was no standard under-tree weed control program and the timing of mechanical cultivation was not consistent. Herbicide applications were not always adequate and many groves required supplemental mowing. Hand labor was usually necessary for vine control regardless of the type of weed control program. Improper timing of herbicide applications is the major problem in most weed control programs.**

Weed control is an important component of the production program for citrus grown on poorly drained soils in the Indian River (I.R.) area (3). Tree rooting depth is limited, necessitating planting on shaped, raised beds and increasing the probability of moisture stress (4, 5). Much of the land was previously planted in torpedograss, bermudagrass, bahiagrass or pangolagrass (Table 1) for pasture, and weed control programs have been hampered by the presence of these and other problem weeds and vines (1, 4). Suitable mechanical equipment is limited due to the shallow rooting depth and bed slope (2). The prevalence of certain perennial, herbicide-tolerant weed species, variable soil types and the need to maintain a sod to minimize water and wind erosion are constraints upon the use of chemical weed control (6, 7, 9). Reduced availability of hand labor and increasing costs of mechanical and chemical methods have

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