

Persistence of Omite Residues on and in Navel Oranges and Lemons and in Laboratory-Processed Citrus Pulp Cattle Feed

William E. Westlake,* Francis A. Gunther, and Lee R. Jeppson

Residues of the acaricide Omite [sulfurous acid, 2-(*p*-*tert*-butylphenoxy)cyclohexyl 2-propynyl ester] were determined on and in mature Navel oranges, Eureka lemons, and in citrus pulp cattle feed prepared from field-treated oranges. The persisting residue half-life was about 80 days for oranges and 50 days for lemons. Washing the fruit reduced by 12 to 20% the residue on oranges in samples

picked 7 and 28 days after spraying but had no effect at the 75-day interval. No reduction of residues by washing lemons was observed at any sampling interval. There was no detectable (<0.05 ppm) penetration of Omite into the edible portions of the fruits. Approximately 50% of the residue on and in the orange rind was lost during processing of the 28-day sample into citrus pulp cattle feed.

Omite [sulfurous acid, 2-(*p*-*tert*-butylphenoxy)cyclohexyl 2-propynyl ester] is a promising acaricide for the control of mites on citrus. This paper reports the dissipation rates of the persisting residues on and in mature Navel oranges, Eureka lemons, and residues remaining in citrus pulp cattle feed prepared from the rind of the field-treated oranges at the 28-day interval. The data presented here are to establish the residue behavior of Omite, to assist in determining the tolerance required, and to define the minimum permissible intervals between spraying and harvest using a wettable powder formulation. An earlier full-coverage study (Gunther, 1969) was made with an emulsive concentrate formulation, but it was felt these data could not be used to predict the behavior of the wettable powder formulation. Gunther (1969) has shown that prediction of the behavior of any given formulation cannot safely be made on the basis of data derived from a different one.

PROCEDURE

Navel orange trees and Eureka lemon trees were sprayed on October 22, 1969, and September 16, 1969, respectively, with approximately 800 gal of spray mixture per acre for oranges and 450 gal per acre for lemons, containing 0.3 or 0.6 lb (1 or 2 lb of Omite 30W) of technical grade Omite per 100 gal applied to each variety. These were full-coverage sprays although the gallonage was low because the trees were small. Untreated plots were maintained as controls. For each variety the plot sprayed with the lesser dosage was of double size, and half of it was resprayed at the same rate 32 days after the first spray for oranges and 30 days for lemons.

The plot arrangement and size and detailed sampling procedure were as described by Gunther (1969). Samples of fully-sized fruits were collected before spraying and at 7-, 14-, 28-, 49-, 75-, 105-, and 131-day intervals after treatment for oranges and at 27-, 41-, 69-, 108-, and 132-day intervals for the lemons. Following the second application, oranges were sampled at 8-, 21-, and 42-day intervals and lemons were sampled at 11-, 27-, 39-, and 78-day intervals. At every other sampling interval, double samples were collected and one was analyzed with and the other without washing. Pulp (peeled fruits) samples were also analyzed at every other interval.

Cattle feed was prepared in the laboratory from 100 lb of oranges picked at the 28-day interval from the plot sprayed at the higher rate, using the procedure described by Gunther (1969). Analyses were made of the rind after grinding and of the finished feed dried to about 4% water content.

ANALYTICAL METHOD

The samples were extracted as described by Gunther (1969) using hexane as the solvent. The extractives were concentrated as necessary in a Kuderna-Danish evaporative concentrator and injected into a gas chromatograph without cleanup. Volumes of 2 to 4 μ l were injected, and standards containing amounts of Omite to give nearly equivalent peak heights were injected after each sample for quantitation.

Quantitative measurements were made with a gas chromatograph fitted with a Melpar flame photometric detector with a 394 m μ filter for sulfur determination. The operating conditions were as follows: Column: 2% SE-30 on Gas Chrom Q; 60/80 mesh; 5 ft \times 6 mm o.d. borosilicate glass. Temperatures were 223 $^{\circ}$ C, 186 $^{\circ}$ C, and 267 $^{\circ}$ C for the column, detector, and injection port, respectively. Gas flow rates were 100, 25, and 160 ml per min for nitrogen (carrier), oxygen, and hydrogen, respectively.

The retention time was approximately 1.5 min and was very satisfactory in the absence of interfering compounds. If the presence of other sulfur-containing pesticides is a possibility, a lower column temperature to give a longer retention time might be advisable. However, of the following pesticides, only ethion interfered with the Omite peak under the conditions employed: azinphos-ethyl, carbophenothion, diazinon, ethion, ovex, and tetradifon. No background was noted in any control sample, and the laboratory recoveries (Table I) were entirely acceptable.

The minimum amount of Omite that could be accurately measured was 10 ng (signal:noise level 5:1), although it was not necessary to go to this low level for any of the rind samples analyzed. The aliquots injected usually contained from 20 to 50 ng of Omite. For the pulp samples the equivalent of 200 mg of sample could be injected, giving 10 ng at the 0.05 ppm level. Actually, no response was encountered that approached this level.

RESULTS AND DISCUSSION

The residues found in the rind of field-treated Navel oranges are shown in Table II and those for lemon rind are found in Table III. Figures 1 and 2, respectively, show the persistence

* Department of Entomology, University of California, Citrus Research Center and Agricultural Experiment Station, Riverside, California 92502

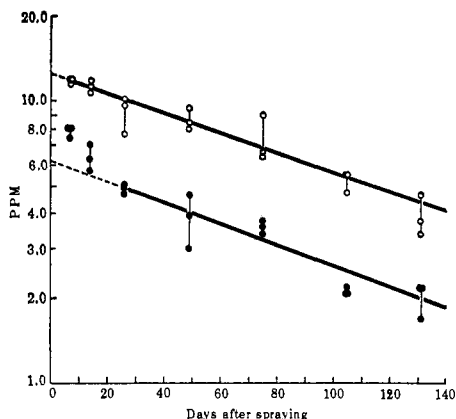


Figure 1. Persistence of Omite on and in Navel orange rind after two different treatments: ○—○ = 0.6 lb of technical grade Omite per 100 gal of spray; ●—● = 0.3 lb of technical grade Omite per 100 gal of spray, 800 gal/acre

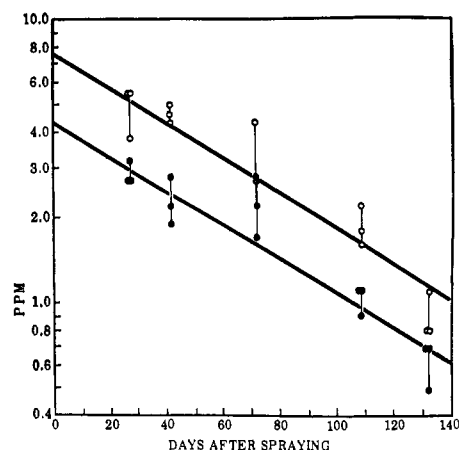


Figure 2. Persistence of Omite on and in lemon rind after two different treatments: ○—○ = 0.6 lb of technical grade Omite per 100 gal of spray; ●—● = 0.3 lb of technical grade Omite per 100 gal of spray, 450 gal/acre

Table I. Percent Recoveries of Omite Added to Extracts from Orange and Lemon Rind and Dried Citrus Pulp Cattle Feed

Level of fortification, ppm	Orange		Lemon		Cattle feed, orange
	Rind	Pulp	Rind	Pulp	
0.0	ND ^a	ND ^b	ND ^a	ND ^b	ND ^a
0.1				98 ^c	
0.2				119 ^d	
0.5	110 ± 7	96 ± 7			
0.6			104 ^c		
1.0			110 ± 13		
1.2			105 ± 15		
2.0	93 ± 11		106 ± 9		109 ± 6
3.0	96 ^e		107 ^c		
4.0	99 ^e		105 ± 9		
5.0	100 ± 15				
14.0					105 ± 2

^a Less than 0.1 ppm. ^b Less than 0.05 ppm. ^c Average of two samples. ^d Single sample. ^e Average of three samples.

Table II. Omite Residues (ppm) on and in the Rind of Navel Oranges, Triplicated Field Samples

Days after spraying	Plot 1 ^a		Plot 2 ^b		Plot 4 ^c
	Unwashed	Washed	Unwashed	Washed	Unwashed
7	7.5, 8.1, 8.1	6.5, 7.3, 5.3	13.1, 13.7, 13.5	11.3, 9.9, 8.8	
8					8.8, 8.5, 6.3
14	7.0, 5.7, 6.3		13.7, 11.5, 12.8		
21					6.9, 5.9, 6.3
28	5.0, 4.8, 5.1	4.2, 4.6, 4.3	9.9, 10.7, 7.6	8.3, 8.0, 8.3	
42					8.5, 5.0, 8.7
49	4.0, 3.0, 4.7		9.5, 8.5, 8.0		
75	3.9, 3.6, 3.4	5.1, 2.7, 4.3	6.5, 6.7, 9.0	7.2	
105	2.1, 2.2, 2.1		4.8, 5.6, 4.6		
131	1.7, 2.1, 2.1		3.8, 4.7, 3.4		

^a 1 lb of Omite 30-W (0.3 lb technical) per 100 gal. ^b 2 lb of Omite 30-W (0.6 lb technical) per 100 gal. ^c Plot sprayed at 1-lb rate, resprayed at same rate 32 days after first treatment.

Table III. Omite Residues (ppm) on and in the Rind of Eureka Lemons, Triplicated Field Samples

Days after application	Plot 7 ^a		Plot 8 ^b		Plot 9 ^c
	Unwashed	Washed	Unwashed	Washed	Unwashed
11					5.3, 5.4, 5.4
27	2.7, 3.2, 2.7	3.4, 4.1, 4.6	5.5, 5.5, 3.8	4.6, 4.9, 4.9	4.1, 3.7, 3.5
39					3.7, 4.9, 5.1
41	2.2, 1.9, 2.8		5.0, 4.6, 4.3		
69	1.7, 2.2, 2.8	1.6, 2.0, 1.4	4.3, 2.7, 2.2	3.4, 3.3, 2.8	
78					2.8, 2.8, 2.4
108	1.1, 1.1, 0.9		2.2, 1.6, 1.8		
132	0.7, 0.5, 0.7	0.6, 0.5, 0.5	1.1, 1.1, 1.1	0.8, 0.8, 1.1	

^a 1 lb Omite 30-W (0.3 lb technical) per 100 gal. ^b 2 lb Omite 30-W (0.6 lb technical) per 100 gal. ^c Plot sprayed at 1-lb rate, resprayed at same rate 30 days after first treatment.

Table IV. Residues (ppm) of Omite in Orange Rind and Dried Citrus Pulp Cattle Feed Prepared from It

Laboratory replicate	Ground rind	Finished feed ^a
1	8.3	13.8
2	8.0	16.2
3	8.3	16.4

^a Dried to about 4% water content. Normally the cattle feed is dried to 8-10% water content. This lot was inadvertently dried further, but this would not markedly affect the residue level in the dried product.

of Omite on oranges and lemons. Based upon extrapolation of these persistence curves to estimate the effective initial deposits, the residue half-life on oranges is 80-90 days and on lemons is 40-50 days. The initial deposits on lemons were about two-thirds those on oranges. There was no detectable (<0.05 ppm) Omite in the pulp (edible part) samples of either fruit at any sampling interval.

Washing the fruit to simulate commercial packinghouse procedure (Gunther, 1969) resulted in a reduction of 12 to 30% in the 7- and 28-day orange samples and no reduction at the 75-day interval. No appreciable reduction of residues on lemons was achieved by washing.

Residues of Omite in dried citrus pulp cattle feed and in the rind used to prepare it are given in Table IV. These data

show a loss of approximately 50% of the pesticide during processing, taking into consideration the loss in total weight through loss of water. A previous study reported in Gunther (1969) with a full-coverage emulsive concentrate formulation but on mature Valencia oranges showed a loss of approximately 75% of the Omite during processing into cattle feed.

In the previous study reported by Gunther (1969) and referred to earlier the residue half-life on Valencia oranges was *ca.* 75 days. Washing of these fruits 30 days after spraying gave *ca.* 40% reduction in the residue in and on the rind. Orange rind containing 2.5 ppm of Omite was processed into citrus pulp cattle feed, and the dried product contained 2.4 ppm of the acaricide. These data agree remarkably well with the data now reported; much greater differences are sometimes observed when different formulations are compared.

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LITERATURE CITED

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