

consistent loss of cloud when pH was adjusted from the normal value (3.9) to 3.2. Configuration and peak location of this curve are approximated closely by the cloud removal curve for 200 ppm of the PE-derived sample with 5.72% MeO content. This suggests the juice contained upon extraction a certain amount of native LM pectin with MeO content near 5.7%. Comparison of the peak heights of the two indicates that the juice contained roughly 50 ppm of this LM pectin.

In summary, there is no single critical MeO content at which pectins become effective juice clarifiers. The critical MeO content depends both upon method of demethylation and pH of the juice. Critical MeO content may range from 0.3% MeO for chemically derived pectins in juices with pH near 4, to 4.5% MeO for enzymically derived pectins in juices with pH near 3. In very acid juices (pH 2.6-3.2) both enzymically and chemically derived LM pectins become less effective clarifiers when MeO contents drop below certain optima. These optima are 2.4% MeO for chemically derived pectins and 4.5% for enzymically derived pectins. Above pH 3.4, in both series clarification was optimum with the pectins of very low MeO. These results provide helpful guidelines for preparation of clarified, natural juice products.

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## PARTICLE SIZE DISTRIBUTION OF COMMERCIAL TOMATO JUICES<sup>1</sup>

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**Abstract.** The particle size distribution of four brands (19 samples) of commercial canned tomato juice was measured using a Coulter Counter Model T Particle Size Analyzer. The analysis revealed that the particles with a diameter range of 8 $\mu$ m to 25.4 $\mu$ m comprised 85% of the total particles but comprised only 9.6% of the particle volume. The volume mean diameter for the four different brands was 63.2  $\mu$ m, 56.6  $\mu$ m, 49.9  $\mu$ m and 48.9  $\mu$ m. The relationship between particle size distribution and gross viscosity of the juice was evaluated.

Particle size distribution is a major contributor to the viscosity of tomato juice. When tomato juice is passed through a fine screen during the finishing process, small particles are incorporated in the juice. The diameter of the holes in the finisher screen affects the particle size and the juice viscosity (4). The speed (rpm) of operation of the paddle finisher also has an effect on juice viscosity. At higher operating speeds there is an increase in the number of particles produced and the particles tend to be more elongated (3). The shape of the suspended particles also influences the viscosity of the product. Tomato juice having

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elongated shape particles has a greater viscosity than juice with spherical shaped particles. The juice serum contributes to the viscosity both by its inherent viscosity and by contributing electrolytes which can alter the colloidal activity of the suspended particles (8).

At the present time, the only particle measurements which have been published on tomato juice, used either wet sieving techniques or microscopic techniques (3, 4, 5, 7, 8). This research was undertaken to determine the particle size distribution of four commercial brands (19 samples) of tomato juice utilizing a Coulter Counter Model T Particle Size Analyzer. In addition, the relationship of the distribution of particles to viscosity was determined.

## Materials and Methods

**Samples.** Four or five lots of four different brands of commercial tomato juice were purchased in supermarkets in Florida, during a 6 month time period. Juices were stored at 2°C prior to analysis.

**Particle Size Analysis.** Particle size analysis of the juices was determined with a Coulter Counter Model T Particle Size Analyzer, a 16 channel analyzer, equipped with a 400  $\mu$ m aperture which allowed measurements of particles with an equivalent spherical diameter of 8.0  $\mu$ m to 161  $\mu$ m. This instrument was calibrated with 41.5  $\mu$ m polystyrene beads (Coulter Electronics, Hialeah, Florida). Samples to be analyzed were diluted so that the particle coincidence was less than 5% in a counting solution consisting of 1.46M sucrose, 20.1 mM NaCl, 1.30 mM EDTA, 5.37 mM KCl, 1.19 mM NaF and 13.30 mM citric acid at pH 4.4. The counting solution was filtered through a 0.45  $\mu$ m and a 0.22  $\mu$ m Millipore filter prior to use. The volume mean diameter (VMD) was calculated by a method of Haile *et al.* (2).

**Viscosity.** The gross viscosity was determined using the Calab capillary viscometer and the Brookfield viscometer Model

Table 1. Gross viscosity of four different brands of tomato juice measured by the Brookfield viscometer and the Calab capillary viscometer.

Brand	Brookfield Method (CPS)		Calab Method (SEC)	
	Mean SE	Range	Mean SE	Range
A	846 ± 82	728-927	97.5 ± 20.5	81.1-128.9
B	588 ± 57	504-633	52.3 ± 9.2	43.5- 65.5
C	698 ± 89	641-852	62.6 ± 5.8	55.0- 69.5
D	495 ± 118	288-587	35.8 ± 7.2	25.1- 42.7

LVF. Juice samples were mixed well in their original containers prior to evaluation. When using the Calab capillary viscometer both the capillary and the body of the viscometer were completely filled with the liquid being tested. The viscometer reading of the juice is the time in seconds required for the upper edge of the meniscus which forms as the juice runs out of the viscometer to reach the calibration line. The product temperature was 30°C. The determinations with the Brookfield viscometer were done using 500 ml of tomato juice at 30°C. The conditions used were spindle No. 2, 12 rpm for 60 seconds (6).

*Statistical Analysis.* All experiments used to determine the

Table 2. Population particle size analysis of four different brands of tomato juice.

Brand	Total No. of particles X 10 <sup>-5</sup> /ml ± SE	Particles X 10 <sup>-5</sup> /ml with a dia. of 8-25.4 μm ± SE	Particles X 10 <sup>-5</sup> /ml with a dia. of 25.4-161 μm ± SE	Percentage of particles with a dia. of 8-25.4 μm ± SE	Percentage of particles with a dia. of 25.4-161 μm ± SE
A	7.82 ± 2.11	6.94 ± 1.87	0.874 ± 0.246	88.6 ± 2.7	11.4 ± 2.7
B	8.41 ± 2.91	7.29 ± 2.21	1.11 ± 0.492	86.5 ± 2.8	13.5 ± 2.8
C	15.7 ± 1.72	14.6 ± 1.56	1.02 ± 0.157	93.5 ± 0.4	6.5 ± 0.4
D	14.0 ± 4.76	13.1 ± 4.58	0.8 ± 0.183	93.2 ± 1.4	6.8 ± 1.4

Table 3. Volume particle size analysis of four different brands of tomato juice.

Brand	Total volume of particles X 10 <sup>-9</sup> (μm <sup>3</sup> /ml)	Volume of particles X 10 <sup>-9</sup> with a dia. 8-25.4 μm (μm <sup>3</sup> /ml)	Volume of particles X 10 <sup>-9</sup> with a dia. of 25.4 μm-161 μm (μm <sup>3</sup> /ml)	VMD (μm)	Percent of volume of particles with a dia. of 8-25.4 μm	Percent of volume of particles with a dia. of 25.4-161 μm
A	7.81 ± 2.11	0.686 ± 0.170	7.12 ± 1.93	63.2 ± 2.0	9.0 ± 2.8	91.0 ± 2.8
B	7.83 ± 4.12	0.711 ± 0.244	7.12 ± 0.388	56.6 ± 4.7	9.6 ± 3.3	90.4 ± 3.9
C	7.06 ± 1.32	1.57 ± 0.223	5.49 ± 1.08	49.9 ± 1.2	22.8 ± 3.4	77.2 ± 3.4
D	6.39 ± 1.36	1.14 ± 0.372	5.24 ± 0.993	48.9 ± 7.4	17.5 ± 4.2	82.5 ± 4.2

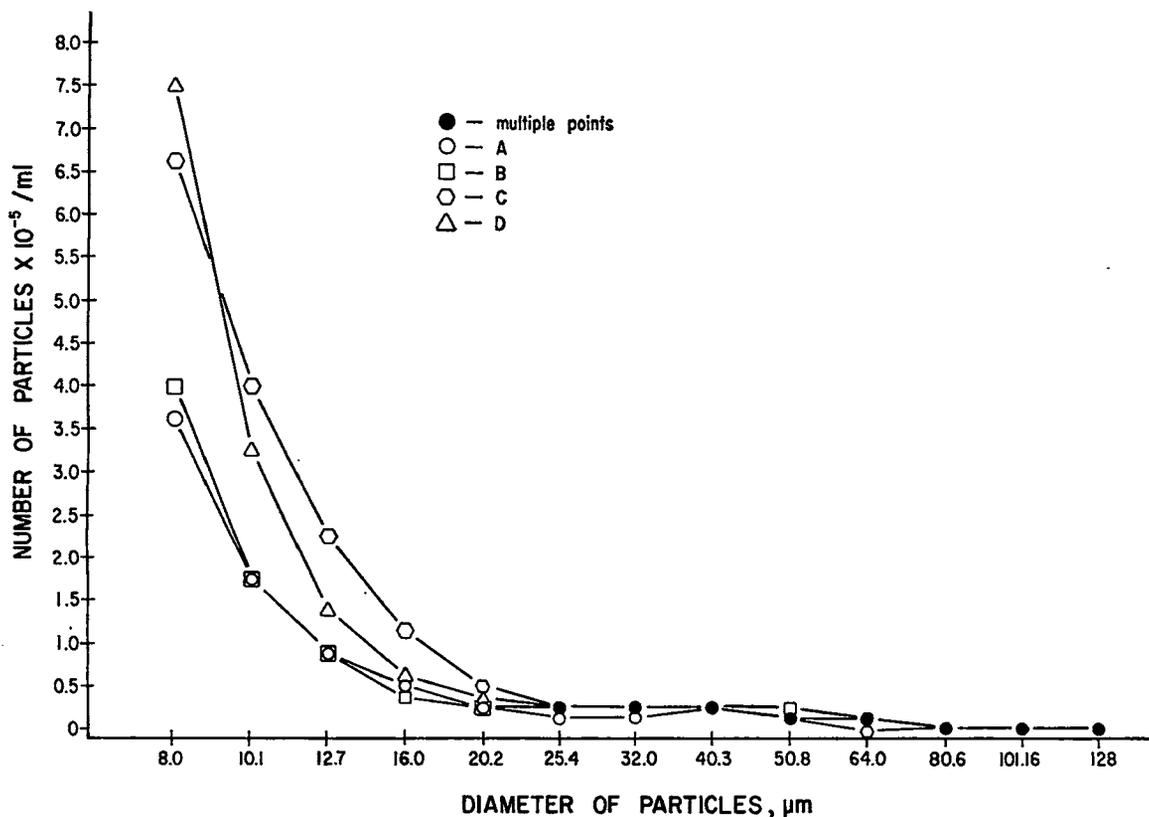


Fig. 1. Particle size distribution as number of particles per ml of four brands of tomato juice.

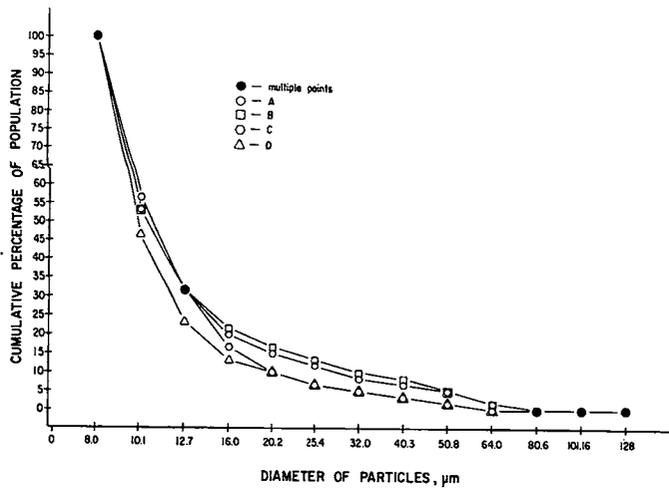


Fig. 2. Particle size distribution as cumulative percentage of population of four brands of tomato juice.

particle size distribution and viscosity were performed two to five times and the data presented are means  $\pm$ SE. Correlations between particle size analysis and viscosity were performed by regression analysis (1).

### Results and Discussion

The viscosities of the tomato juice ranged from 288 to 927 cps. when measured by the Brookfield viscometer and 25.1 to 128.9 sec. when measured by the Calab Capillary viscometer (Table 1). Within a given brand, there was a substantial variability in sample viscosity between lots. Three of the brands had standard errors of approximately 10% of the mean, however, Brand D had a mean standard error of 24% (Table 1).

A significant positive linear correlation ( $r = 0.88$ ) occurred between the two methods used to measure viscosity, indicating the possibility of using either method in determining the viscosity of tomato juice. The juice industry prefers the capillary viscometer method.

Particle size distribution was performed on the tomato juices (Tables 2 and 3, Figures 1-4). Eighty-five percent of the particles in the juices had an equivalent spherical diameter of 8 to 25.4  $\mu$ m (Table 2). In addition there was a wide variation in the number of particles per ml among different lots of the same brand. In contrast, the percentage of particles in this size range varied only 2-3% (Table 2). This suggests that even though there may be a wide variation in the quantity of particles in a given brand, the relative distribution of particles tends to be constant.

Ninety percent of the total volume of particles was in the size range of 25 to 161  $\mu$ m (Fig. 3 and 4, Table 5). This indicated that a small number of particles contributed to a large percentage of the volume. In addition a wide variation in the volume of particles among a given brand was observed but the relative distribution of particles was constant (Table 2). The volume mean diameter (VMD) of the equivalent spherical diameter where 50% of the total volume was greater than the given diameter, ranged from 48.9  $\mu$ m to 63.2  $\mu$ m (Table 3).

Regression analysis was performed between the particle size distribution of each individual lot to the respective viscosity. A significant positive linear correlation was observed between the VMD and viscosity when the viscosity was measured using either the Brookfield viscometer or the Calab Capillary viscometer (Fig. 5 and 6). In addition there was a positive linear correlation between the total volume and viscosity when the viscosity was measured by the Calab capillary viscometer (Fig. 7). This data is in agreement with previous data where the viscosity of tomato

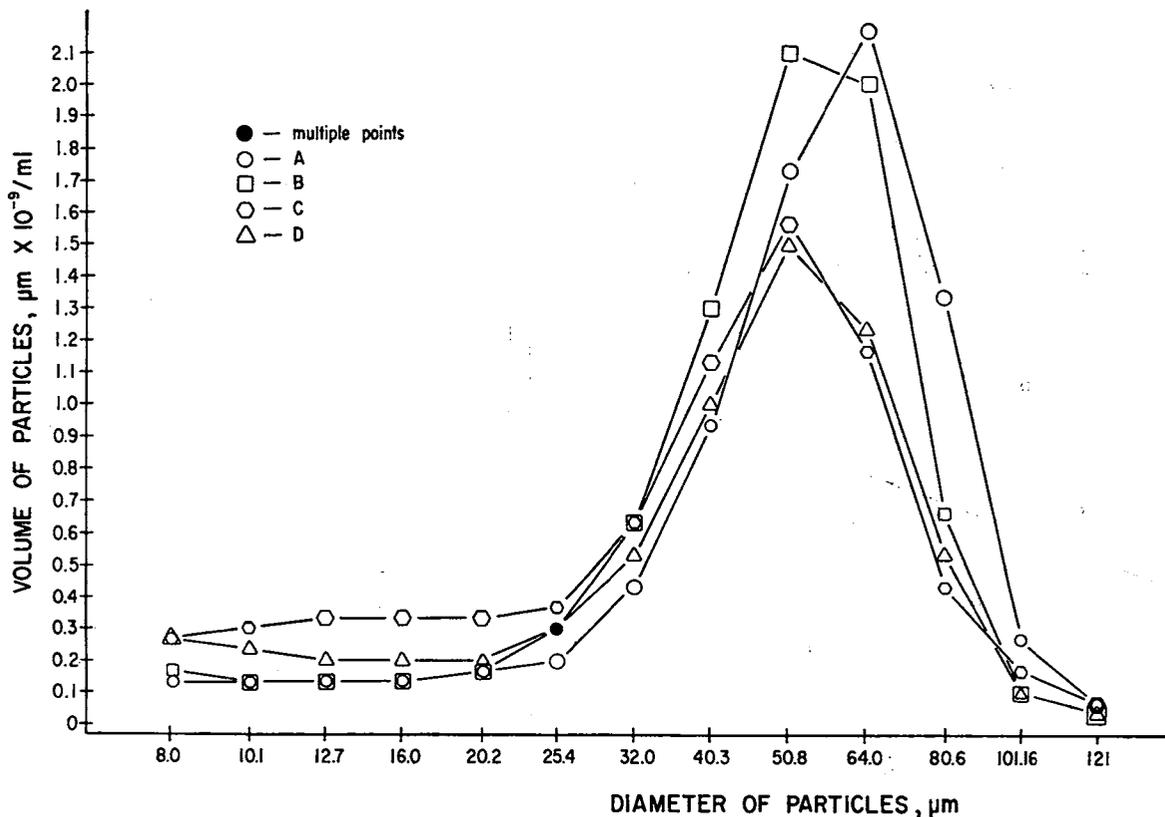


Fig. 3. Particle size distribution as volume of particles per ml of four brands of tomato juice.

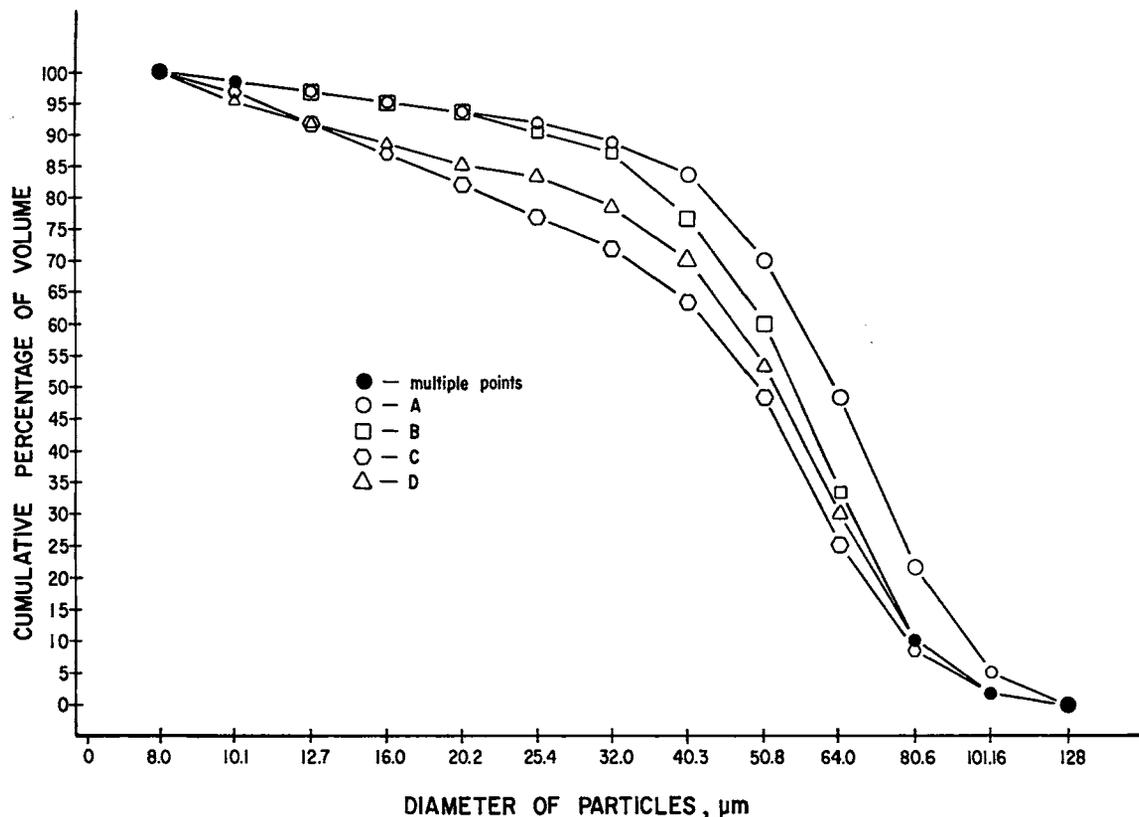


Fig. 4. Particle size distribution as cumulative percentage of volume of four brands of tomato juice.

juice was shown to be a partial function of the quantity of suspended particles and the shape of the suspended particles (3). There was an increase in the viscosity of the tomato juice with an increase in the number of particles. With an increase in the size of particles, the shape of particles tended to be elongated (3).

### Conclusions

The particle size distribution of tomato juices was accurately measured using electronic means. The VMD and the total volume of particles per ml can be correlated with the viscosity of the juices. Twenty-eight to forty-two percent of the viscosity was explained by these two factors. Work is

continuing to determine the relationship of particle size distribution to other physical parameters of tomato juice.

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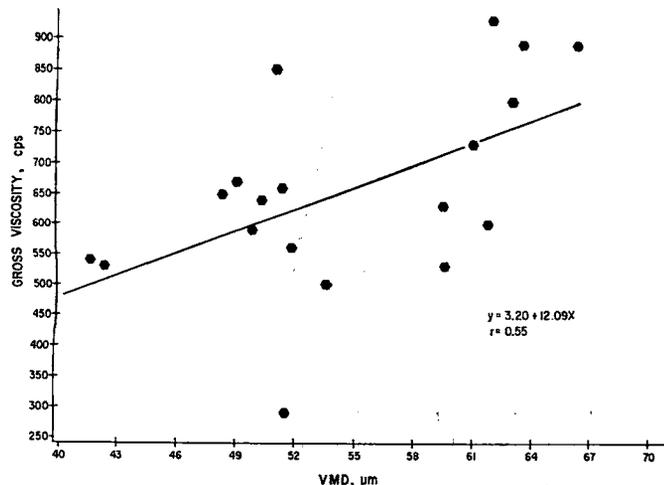


Fig. 5. Regression analysis between VMD and gross viscosity as measured by the Brookfield viscometer.

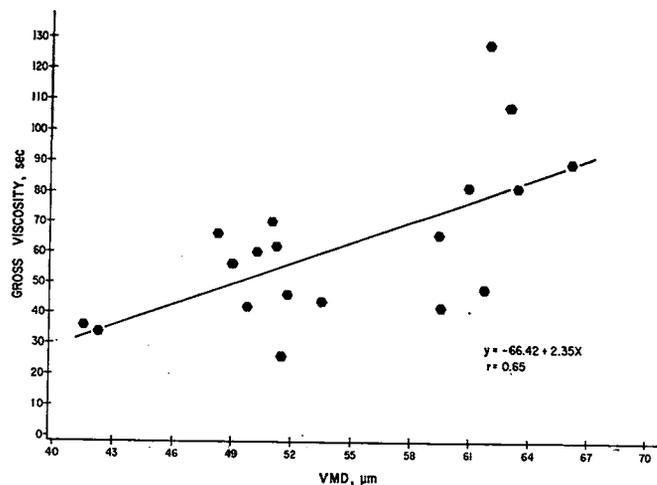


Fig. 6. Regression analysis between VMD and gross viscosity as measured by the Calab capillary viscometer.

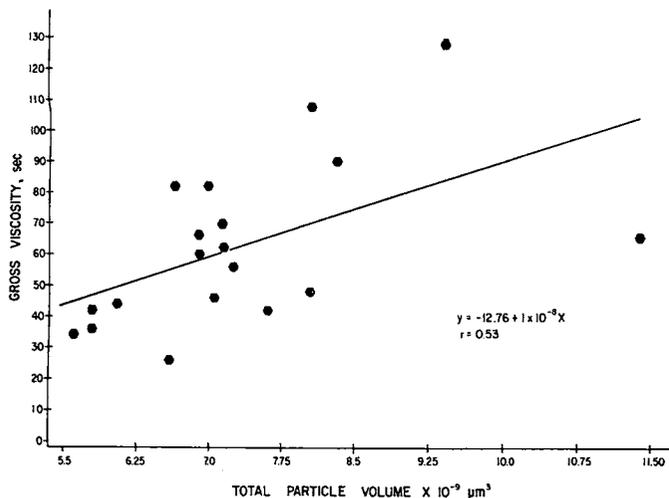


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## RESIDUES OF ETHYLENE DIBROMIDE PRESENT IN FUMIGATED FIBERBOARD CARTON MATERIAL<sup>1</sup>

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**Abstract.** Samples of fiberboard were fumigated with ethylene dibromide (EDB) at dosages approved for quarantine commodity treatments of grapefruit infested by fruit fly larvae. Residual EDB present in the samples was determined periodically from 2 to 576 hr after fumigation. Data were subjected to regression analysis. In general EDB residues were similar in covers and bodies of fiberboard cartons from the same samples. Fiberboard samples that had been treated with a wax copolymer retained EDB longer than untreated samples.

Ethylene dibromide (EDB) has been used for several years to meet quarantine requirements for export shipments of grapefruit destined for Japan (3). Treatments are applied to prepackaged fruit in semi-trailer vans at fumigation facilities located in Fort Pierce, Wahneta, and Gainesville, Florida. From these facilities the fruit is taken by truck to various shipping facilities, such as Port Everglades or Tampa, where the fruit is held in warehouses until loaded on ships destined for Japan.

Recently investigations have been undertaken to improve techniques for handling fumigated fruit in order to reduce

any possible environmental pollution that might result from such treatments. We have found significant concns of EDB gas in semi-trailer vans that had remained overnight in a closed chamber after fumigation and aeration for over 4 hr (1). Apparently the fiberboard cartons used in our research to simulate commercial fumigation of grapefruit exhibited a high rate of sorption for EDB during fumigation, and the EDB was subsequently desorbed after the treatment was completed. These findings indicated that similar results could be expected after commercial fumigations plus the usual fumigation chamber aeration, transit in van containers enroute to the dock, storage at the dock warehouse, and shipboard transit to Japan. Research was undertaken to determine variability in absorption, retention, and desorption of EDB from fiberboard used in cartons.

### Methods

Grapefruit usually are shipped in full telescope fiberboard cartons. The standard carton cover has a paperboard weight of 42 - 33 - 42 (42.00/47.52/42.00 lb/1000 ft<sup>2</sup>), and the body has a paperboard weight of 90 - 33 - 90 (90.00/47.52/90.00 lb/1000 ft<sup>2</sup>) (2). Such cartons have a bursting strength of 200 and 300 lb respectively. Two samples of fiberboard cartons, one used for domestic shipments and designated "A" and the second used for export of grapefruit to Japan, designated "B", were used in these tests. Samples "C" and "D" were corrugated board that had been treated on either one or both surfaces, respectively, with a certain coat application of a wax copolymer blend. Sample "E" was fiberboard that had been treated with Resisto Coat I.S.

The samples of fiberboard were obtained from fabricators and tested in an attempt to determine whether there were significant differences in absorption, retention, or loss of EDB during the fumigation and shipping of grapefruit in fiberboard cartons. The fiberboard was cut into 4 x 4-inch squares (10.2 x 10.2 cm) and weighed, and the edges were sealed with Teflon tape. The weights of the squares,

<sup>1</sup>The use of trade names in this publication is for description and clarification and does not constitute a guarantee, warranty or endorsements of the products by the United States Department of Agriculture.

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