

## THE EFFECT OF DIFFERENT METHODS OF JUICE EXTRACTION ON THE PECTIC CONTENT OF VALENCIA ORANGE JUICE<sup>1</sup>

C. D. ATKINS AND A. H. ROUSE

*Florida Citrus Experiment Station*

Lake Alfred

Variations in the composition of freshly extracted citrus juices are of importance to those interested in maintaining the quality of frozen concentrates. It was indicated by the authors (6) that the pectic content of Pineapple orange juice was more affected by processing of the juice and storage of the concentrate than by the degree of maturity of the fruit. With several different procedures of juice extraction being available to citrus processors, it seemed desirable to determine if variations in juice composition would result when different methods of extraction were used. Since the primary purpose was to determine variations in the pectic substances in juices, this investigation also made possible the use and discussion of a revised extraction procedure in the method previously reported by Dietz and Rouse (3) for estimating pectic substances in citrus juices. The purpose of this paper was not to determine which commercial juice extractor was the best for commercial use, since the scope of this preliminary investigation was too limited for such a purpose.

### EXPERIMENTAL PROCEDURE

**Juice Extraction.** Sufficient Valencia oranges were washed and sized to yield eight boxes of medium size fruit. These were randomly mixed into four groups of two boxes each. Two boxes of fruit were juiced on each of the four commercial types of extractors now used in Florida. The juice, pulp, and seeds were finished in a Food Machinery, Model 35, finisher with an 0.030 inch perforated screen. Samples of the screened juices were canned in 6-oz. cans and stored at  $-8^{\circ}$  F. until analyzed.

**Estimation of Pectic Substances.** A rapid method for estimating pectic substances in citrus juices by Dietz and Rouse (3) has been

improved by a revision of the extraction procedure.

Citrus juice or concentrate is comminuted for three minutes in an Osterizer or similar blender. Weigh 16 g. of juice or four g. of concentrate into a tared 50 ml., graduated, short conical bottom, centrifuge tube. To the four g. of concentrate are added 12 ml. of distilled water. Hot ( $75^{\circ}$  C.) 95% ethyl alcohol is added to a volume of 40 ml. and the mixture heated for 10 minutes in a water bath at  $85^{\circ}$  C. with occasional stirring using a glass rod. The stirring rod is then rinsed off with 95 per cent alcohol and the volume made up to 50 ml. Centrifuge the tube at 2100 r.p.m. for 15 minutes and after decanting discard the supernatant solution. Repeat the leaching with hot 60 per cent alcohol for 10 minutes in a water bath at  $85^{\circ}$  C., centrifuge, and again decant and discard the supernatant solution.

Add about five ml. distilled water to the tube and disperse the precipitate with a rubber policeman. Rinse policeman, make contents to a volume of 35 ml. with distilled water at room temperature, and stir vigorously and continuously for 10 minutes. This is accomplished either by a mechanical stirrer or by bubbling air through the mixture in the tube. The bubbling device is made by connecting to a source of air a capillary tube about six inches in length which is inserted into the centrifuge tube. The stirrer or capillary tube is rinsed with approximately five ml. distilled water increasing the volume to 40 ml., the tube centrifuged at 2100 r.p.m. for 15 minutes, and the liquid decanted into a 100 ml. volumetric flask. Repeat the water extraction and after centrifuging, decant into the same volumetric flask. Add five ml. 1 N. sodium hydroxide to the water extract and dilute to volume. Mix and let stand 15 minutes before beginning the colorimetric procedure.

To the residue add five ml. of 0.5 per cent ammonium oxalate solution and disperse the precipitate with a rubber policeman. Rinse policeman and make the tube to a volume of 35 ml. with the oxalate solution at room temperature and stir vigorously and continuously for 10 minutes as above. Make to volume of 40 ml. with oxalate solution. Centrifuge as

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before and decant into a 100 ml. volumetric flask. Repeat the oxalate extraction, centrifuge, and decant into the same 100 ml. flask. Add five ml. 1 N. sodium hydroxide to the oxalate extract and dilute to volume. Mix and let stand for 15 minutes before beginning the colorimetric procedure.

The residue remaining in the centrifuge tube is washed into a 100 ml. volumetric flask, five ml. 1 N. sodium hydroxide is added, and the contents made to volume with distilled water. Mix, let stand 15 minutes with occasional shaking, and filter.

Analyze one ml. aliquots from each of the above three extracts by the colorimetric procedure as described by Dietz and Rouse (3).

*Other Analytical Methods.* Pulp content was estimated by centrifuging a 50 ml. sample of juice at 1600 r.p.m. for 15 minutes in a Size 1, Type SB International Centrifuge.

Total solids and water-insoluble solids were determined by the A.O.A.C. methods (1) for fruits and fruit products.

Pectinesterase activity was assayed essentially by the procedure of MacDonnell, Jansen, and Lineweaver (5), using a Beckman Model K Automatic Titrator. Pectinesterase units (PE.u.) were expressed as the milliequivalents

of ester hydrolyzed per minute per gram of juice and multiplied by 10,000 for easy interpretation.

Light transmission of the centrifuged juice was determined using a 10 mm. rectangular cell and Filter No. 730 in a Lumetron colorimeter.

The glucoside content was determined as hesperidin by an adaptation of the Davis method (2) as described by Kesterson and Hendrickson (4).

#### EXPERIMENTAL RESULTS AND DISCUSSION

Analytical results (Table 1) from the examination of four Valencia orange juices obtained by different methods of extraction definitely showed that the composition of the juices varied. Significant differences were found in pulp content (8.0 to 12.0 per cent), water-insoluble solids (0.060 to 0.136 per cent), pectinesterase activity (17.6 to 34.5 units), hesperidin (0.035 to 0.065 per cent), and total pectin (0.044 to 0.080 per cent). Pectinesterase activity, pectin, and hesperidin increased as the pulp or the water-insoluble solids increased. The percentage light transmission, indicative of "cloud" in the juices, varied slightly from 58 to 66 per cent. The

Table 1  
Chemical analyses of Valencia orange juices extracted by  
different methods

Analyses	Finished juices from four extractors			
	A	B	C	D
Soluble solids, °Brix by refractometer at 28°C.	12.2	12.2	12.2	12.2
Acid as citric, %	0.64	0.68	0.67	0.64
Brix/acid ratio	19	18	18	19
Gal. of juice per box, approx.	6.0	5.0	5.0	5.5
pH	4.0	4.0	4.0	4.0
Pulp, % by volume	12.0	8.0	10.0	11.0
Total solids, %	12.19	12.09	12.29	12.33
Water-insoluble solids, % (PE.u.)g. of juice x 10 <sup>4</sup>	0.136	0.060	0.096	0.124
Light transmission of centrifuged juice, %	60	61	66	58
Glucosides as hesperidin, % by volume	0.065	0.035	0.052	0.053
Total pectin, %	0.080	0.044	0.056	0.068

yield of finished juice obtained varied from 5 to 6 gal./box.

Data reported in Table 2 show the pectic substances found in four Valencia orange juices when the following extractants were used, (a) water, 0.4 per cent sodium metaphosphate, and 0.05 N. sodium hydroxide and (b) water, 0.5 per cent ammonium oxalate, and 0.05 N. sodium hydroxide. The sodium metaphosphate-soluble pectins in the four orange juices were 0.023, 0.013, 0.016, and 0.023 per cent, whereas the ammonium oxalate-soluble pectins were 0.021, 0.014, 0.016, and 0.023 per cent, respectively. Total pectin varied in the juices from 0.044 to 0.080 per cent when sodium metaphosphate was used as the extracting agent and varied from 0.047 to 0.083 per cent when extracted with ammonium oxalate.

Attention should be called to the following techniques that result in more consistent data when used in the analytical procedure for determining pectic substances in citrus products.

Centrifugation of the water and oxalate extractions of pectin from the alcohol precipitate of citrus concentrate facilitates complete sedimentation, whereas when the water and oxalate extraction of pectin are separated by centrifugation from the alcohol precipitate of fresh citrus juice, inevitably some insoluble solids remain floating. On decantation, it is impossible to prevent this floating material from passing into the volumetric flask. To prevent the occurrence of floating material about  $\frac{1}{2}$  teaspoonful of disintegrated, wetted paper pulp (Whatman Ashless Tablets) is added to the

50 ml. centrifuge tube during the first water extraction. The paper pulp aids the sedimentation during centrifugation by carrying the insoluble solids to the bottom of the centrifuge tube.

A technique that should be carefully followed in the colorimetric procedure is regulation of the time required to add the six ml. of concentrated sulfuric acid. The analyst should have a dispenser set up to deliver six ml. sulfuric acid in seven seconds to obtain a temperature of 185° F. (heat of solution) and immediately place the large test tube into a water bath heated to 185° F. Otherwise, varying intensities of red color may be produced in duplicate samples. The tube should remain in the bath for ten minutes and during this waiting period the temperature need not be maintained.

#### SUMMARY

Examination of four Valencia orange juices, extracted by different methods, showed significant variations in juice composition. Pectinesterase activity, pectin, and glucoside content, factors of importance to juice quality, were greater in those juices containing the larger amounts of pulp or water-insoluble solids.

A revised procedure and techniques for the extraction of pectic substances in citrus juices, prior to their determination by a colorimetric method, are presented and discussed. No significant differences were found in the total pectin content of orange juices when ammonium oxalate was used in place of sodium metaphosphate in the extraction procedure.

Table 2  
Comparison of pectin yields in Valencia orange juices using sodium metaphosphate and ammonium oxalate as extracting agents

Finished juices	Procedure A				Procedure B			
	Pectin as anhydrogalacturonic acid				Pectin as anhydrogalacturonic acid			
	H <sub>2</sub> O-sol. %	(NaPO <sub>3</sub> ) <sub>n</sub> -sol. %	NaOH-sol. %	Total %	H <sub>2</sub> O-sol. %	(NH <sub>4</sub> ) <sub>2</sub> C <sub>2</sub> O <sub>4</sub> -sol. %	NaOH-sol. %	Total %
A	0.023	0.023	0.034	0.080	0.026	0.021	0.036	0.083
B	0.010	0.013	0.021	0.044	0.010	0.014	0.023	0.047
C	0.015	0.016	0.025	0.056	0.015	0.016	0.026	0.057
D	0.015	0.023	0.030	0.068	0.013	0.023	0.033	0.069

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## EXPLORATORY STUDIES OF PRESCHOOL CHILDREN'S TASTE DISCRIMINATION AND PREFERENCE FOR SELECTED CITRUS JUICES

RICHARD L. D. MORSE

*Professor of Family Economics  
Florida State University*

Tallahassee

These studies constitute the first published attempt to estimate preschool children's discriminability and preferences for citrus juices. An extensive search of the literature for previous studies in the area yielded reports on children's likes and dislikes for specific foods, the effect of size of serving on food consumption, the social dynamics of food preferences, e.g., the effect of the group leader's preference on the preferences of the followers in the group, and studies involving children's taste reactions to salt, bitter, sweet, and sour. Since none pertained directly to this problem, these investigations constitute an effort to discover methods of taste-tests appropriate to this age level of children.

### DISCRIMINATION AND PREFERENCE

*Discriminability* is measured in terms of degree to which the individual or group can distinguish between two stimuli and can communicate this distinction to the investigator. Factors which affect discriminability are: individual's taste acuity at the time of the test; the consistency or stability of this ability over time, if the results of tests conducted at several different times are to be compared; the "distance" or difference between the stimuli, measured in terms of their physical properties; the design of the test, especially its complexity and premium it places on memory; and, the

method of communicating the results from the subject to the investigator. Furthermore, any declaration as to the discriminability depends on the arbitrary standard the investigator sets as the number of correct versus incorrect judgments required. For example, this investigator required that the subject make at least 10 correct judgments out of 12 trials to be declared discriminating, reasoning that such ratio of judgments between equal stimuli could have occurred by chance guess in slightly less than 5% of similar repeat trials. If this arbitrary level were reduced, more subjects could qualify as discriminating. The lowest limit would be to include all those subjects who made more correct than incorrect judgments. Such discussion soon leads to the probability theory. The point of emphasis here is the arbitrariness of the level set.

*Preference* assumes discriminability; that is, it is logically inconsistent that one can prefer one stimulus over another if the two stimuli are not distinguishable (discriminable) by the subject. This point is well developed by Clements (1951). (If, on the other hand, the arbitrary level for discriminability were set too high, it is possible that stimuli which are declared non-discriminable may be declared unequally preferred.) Frequently overlooked is the point that preferences are relative; that is, one cannot declare a preference for a stimulus without reference to some standard for the product or its close substitute. In the simplest case, two stimuli are provided and one serves as a standard for the other; one is ranked relative to the other in the paired comparison test. Interpretation of such paired tests is obvious, although here again, as in the discriminability