

## Composition of Commercial, Segment, and Peel Juices of Florida Oranges

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Comparative composition studies were conducted throughout a season. Peel juices were always highest in pH, Brix-acid ratio, soluble pectic substances, ascorbic acid, flavonoids, diacetyl, and color, and lowest in acidity and fluorescence. Peel juices were usually highest in soluble solids, specific gravity, and viscosity. During the early part of the season, sucrose was lowest and reducing sugars were highest in peel juices. Peel juices added at a level of 3% in reconstituted concentrate were detected with significance by a taste panel.

THE YIELD of single strength orange juice per 90-pound box of oranges for the season of 1940-41 was 27.46 No. 2 (18-ounce) cans. By the 1953-54 season, after a progressive increase, this yield had risen to 39.83 cans per box (4). This is an increase from 3.86 to 5.60 gallons per box or a gain of 45%. Without a doubt, much of this increase was due to more efficient extraction and finishing equipment and some might have been due to inherently juicier fruit resulting from improved grove and handling practices. Nevertheless, it seemed possible that this trend, if due to increasing extraction and finishing pressures, might eventually result in the incorporation of substantial amounts of peel and rag extractives in the segment juice. With the exceptions given below, little is known about the over-all effects of incorporated peel juice on the quality of commercially extracted segment juice. It was to supply some of these missing data that the present study was undertaken.

Olsen, Huggart, and Asbell (9) found that increased pressure during juice extraction and the use of pulpy cutback juice increased the tendency for frozen concentrated orange juice to gel in the can and clarify upon reconstitution.

Atkins and Rouse (2) extracted orange juice with four makes of juice extractors and observed small variations in Brix-acid ratios, total solids, and acid content. Wide variations were found in juice yield, pulp by volume, water-insoluble solids, pectinesterase, flavonoids, and pectin.

Pobjecky (10) has mentioned the

possibility of obtaining bitter flavors from the peel, seeds, or pulpy portion of the orange by overextraction.

Hall (5) found that the juice from the outermost parts of the edible portions of the fruit was higher in soluble solids and lower in acid than was that from the innermost portions.

The general plan of the work was to make certain physical and compositional comparisons of commercially extracted orange juice, peel juice, and hand-pressed segment juice. Only soluble constituents were considered and the juices were clarified by filtration before analysis. It was thought that if these three juices were collected simultaneously at a single plant at weekly intervals for an entire season, the analyses should furnish some basis for estimating the effect of current extraction and finishing procedures and the probable effects of increased extraction and finishing pressures, if they were contemplated in the future.

### Experimental Procedure

Samples were collected at weekly intervals throughout the 1954-55 operating season from a commercial concentrate plant in central Florida. This period extended from December 14 to June 16, with the exception of the month of March, when no oranges were handled at this plant because of insufficient quantity of fruit of suitable maturity. Samples of fresh commercially extracted juice, fruit for hand juice extraction, and peel juice from peel oil recovery operations were ob-

tained each time. The fresh commercial juice was taken directly from the finishers. The fresh fruit was taken to the laboratory, halved, and lightly extracted on a glass reamer, care being taken to include a minimum of rag and pulp. This was termed segment juice. The peel juice came from an auxiliary operation in which cold-pressed orange oil was recovered. For this purpose fresh peel from the extractors passed directly to fluted Pipkin rolls, which expressed an emulsion of peel oil in peel juice. The emulsion passed through finishers which removed pulp, seeds, and pieces of peel and then to high-speed centrifugals where cold-pressed peel oil was recovered and the juice discarded. There was no dilution with water. Samples of this juice were collected for study.

All juices were placed in 46-ounce cans and frozen as soon as possible after the samples were obtained. All samples were held at  $-18^{\circ}$  C. until the end of the season, when they were thawed, filtered clear with Hyflo Supercel, refrozen in 4-ounce cans, and held until analyzed.

Early and midseason varieties were being processed during December, January, and February. During the first part of this period the fruit was principally of early (usually Hamlin) varieties and during the latter part principally midseason (usually Pineapple) varieties. Generally both kinds were used and blended in making the final product. It was impossible to determine at a particular moment which variety was being processed and this should be borne in mind in evaluating the results.

During the April–June period (late season) nearly all the fruit entering processing plants was of the Valencia variety.

### Analytical Methods

All colorimetric measurements in this study were made with a Lumetron Model 401 colorimeter. Refractive index measurements were made with a Bausch & Lomb Precision refractometer; and per cent soluble solids by weight was estimated using sucrose refractive index tables.

Acidity was determined by titrating with standard alkali to the phenolphthalein end point and calculating as per cent citric acid by weight. Density measurements permitted expression of the acid content on a weight percentage basis. The pH was determined with a Model N Beckman battery-operated, glass electrode meter.

Reducing sugars were determined by the Lane–Eynon (7) procedure and expressed as invert sugar. Total sugars were determined by the same method after acidifying and allowing the solution to stand overnight to invert the non-reducing sugars. Nonreducing sugars were calculated by multiplying the differences by 0.95 and were reported as per cent sucrose.

Pectic substances were determined by the method of McCready and McComb (8), in which the precipitated and de-esterified pectin was treated with concentrated sulfuric acid and carbazole, the color development was measured using a 530-m $\mu$  filter, and comparisons were made with a standard curve.

Ascorbic acid was determined by titrating 5 ml. of juice with standardized dichlorophenolindophenol dye solution after the addition of 5 ml. of 0.5% oxalic acid solution.

Flavonoids were estimated by the Davis (3) procedure, using a reference curve obtained with solutions of known hesperidin content.

Diacetyl and acetoin were estimated together as diacetyl by a modification of the method used by Hill, Wenzel, and Barretto (6), in which 100 ml. of juice was distilled until 25 ml. of distillate was collected. After mixing, 10 ml. of distillate was treated with 5 ml. of 1-naphthol solution and 2 ml. of alkali-creatinine reagent in a colorimeter tube. The mixture was shaken for 15 seconds, allowed to stand for 10 minutes, and again shaken for 15 seconds. The tubes were read using a 550-m $\mu$  filter. No calibration curve was prepared and the results are expressed as absorbances, which are approximately proportional to the sum of the diacetyl and acetoin contents.

Ash was determined by evaporating 100 ml. of juice in a platinum crucible to a thick sirup, which was gradually

charred and finally ignited at 525° C. in a muffle furnace. After cooling and moistening, it was dried and again ignited at 525° C. overnight.

Specific gravity values were obtained by weighing portions in a calibrated Reischauer flask at 20° C. and comparing with the weight of water at the same temperature.

Relative viscosities were estimated on a Stormer viscometer at 20° C. by comparing the values thus obtained with those for distilled water at the same temperature.

Color was evaluated by diluting the samples with an equal volume of water and comparing with a water blank using a 420-m $\mu$  filter. The results are presented as absorbance.

Fluorescence was determined with a Coleman Model 12 electronic photofluorometer, using 2 ml. of juice mixed with 20 ml. 95% ethyl alcohol. The mixtures were allowed to stand for a short time and filtered. The filtrate was used in the cuvette of the instrument. The B-1 filter was used in the No. 1 position and the PC-1 filter in the No. 2 position. The instrument had been set to give a reference reading of 90 with a standard quinine sulfate solution (3 mg. per liter in 0.1*N* sulfuric acid). Meter readings are reported.

Taste tests were made to estimate the relative flavor potencies of the peel juice at different times during the season. The season was divided into four equal periods and the weekly samples from each period were pooled in equal amounts. Each of these composites was added in varying amounts to a reconstituted orange concentrate of good quality and presented to a taste panel in comparison with controls containing no peel juice. The triangular method of presentation was used and the order, codes, and selection of odd samples were randomized. All samples were presented at the same temperatures in ruby-colored glasses. A total of 20 judgments was obtained for each comparison. The table of Krum (7) was used in estimating the significance of the data.

### Results and Discussion

With the exception of ash and taste data, all results of analytical procedures are given in Figure 1. Overlapping of varieties during the early season no doubt accounts for some of the wide compositional variations found. Later season fruit is almost exclusively Valencia, and consequently, less variability would be expected. Usually, this is borne out in the data.

As would be expected, the soluble solids contents of both early and late season juices increased as the season progressed. During the early part of the season the solids content of the peel

juice was generally higher than that of the other types and always higher during the late season by 1 to 2%.

The acidities of segment and commercial juices varied between 0.80 and 1.20% anhydrous citric acid, and did not show the expected decrease. Peel juice acidities ranged from 0.44 to 0.70 and showed a slight increase as the season progressed, which is the reverse of the usual findings for segment juices. For peel juices, pH values remained very constant, but for the segment and commercial juices they rose somewhat during the latter part of the season. Brix-acid ratios for peel juices ranged from 22.1 to 29.4% and segment juices from 10.3 to 16.4%.

As might be expected, there was an upward trend in reducing sugars with time in both the early and late parts of the season. The reducing sugar content of early season peel juice at all times exceeded that of the other juices, but with late season juices this was not the case.

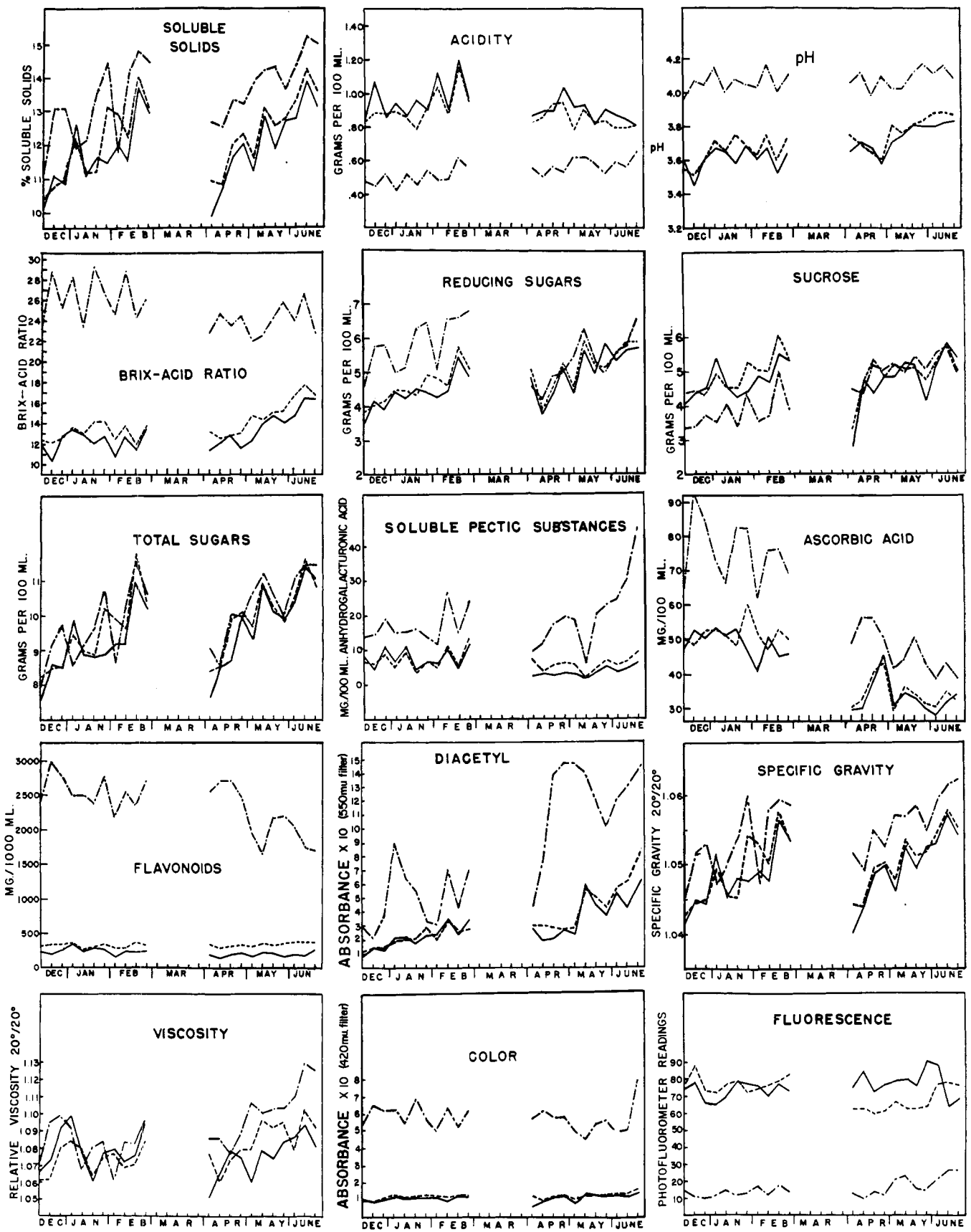
Sucrose contents increased with time, but the content of the early season peel juices was uniformly lower than that of the others.

Total sugars, consequently, all showed an upward trend with time. The total sugar contents of late season juices were very similar and offered no clue as to the identity of the substance or substances responsible for the high soluble solids findings in peel juices.

The hand-pressed segment and commercially extracted juices showed no particular change in soluble pectic substances throughout the season; but the peel juice, always higher in these constituents than the others, showed a marked upturn late in the spring. The commercial juices contained more pectic substances than did segment juices during the late season.

As expected, segment and commercial juices were much higher in ascorbic acid during the early part of the season than later. Peel juices at all times exceeded the corresponding segment and commercial juices in ascorbic acid but decreased in this constituent throughout the entire season. Thus the final sample of peel juice taken in June contained only about 5 mg. more of ascorbic acid per 100 ml. than did the other juices. The difference had been nearly 40 mg. per 100 ml. in December.

In flavonoid content, the segment and commercial juices were remarkably constant at 300 to 500 mg. per liter throughout the season; but peel juices ranged from 1630 to 2980 mg. per liter, with the lower limit coming at the end of the season. The test employed (3) indicates certain flavonoids, regardless of whether they influence flavor or not. Hesperidin, which gives the Davis reaction and is the principal flavonoid of orange juice, is not very bitter, probably be-



SEGMENT (HANDPRESSED) JUICE——, COMMERCIAL JUICE-----, PEEL JUICE— · — ·

Figure 1. Comparisons of composition of commercial, segment, and peel juices of Florida oranges

cause of its low solubility in water. On the other hand, the Davis test does not indicate flavonoids having no free phenolic groups.

The method for determining diacetyl plus acetoin has been employed to serve as an index of bacterial activity in fresh juice. These compounds, however, may be normal constituents of juices, as the test was given by all types of juices and all were handled rapidly without much opportunity for fermentation. It is difficult to explain the diacetyl-acetoin values given by the peel juices, unless these actually contained more of these substances as normal constituents, or, if the fermentation theory is to be credited, the cleanup operations on the oil line may have been less frequent and rigorous than on the juice line and greater build-up of fermented material occurred. The values show an upward trend in all juices during both early and late parts of the season, but the rise was particularly marked during the latter part, especially with peel juice, although it was at all times the highest with respect to diacetyl.

Although the total sugar contents of the three juices were about the same for the latter part of the season, the soluble solids of the peel juices were from 1 to 2% higher than those of the other juices. To determine if the nonsugar constituents responsible for this excess were mineral salts, ash determinations were made on both segment and peel juices collected on May 4 and May 11 (Table I). Although the data show somewhat greater mineral content for the peel juices, the excess was not enough to account for the high solids, unless the salts involved acids of very high equivalent weights.

Total nitrogen determinations on the same quarterly composites of peel juices used for tasting and also on corresponding composites of segment juices gave, respectively, the following results expressed as crude protein ( $N \times 6.25$ ): first quarter, 0.428 and 0.364%; second quarter, 0.564 and 0.442%; third quarter, 0.645 and 0.440%; fourth quarter, 0.733 and 0.554%. This comparison does not indicate that the unidentified nonsugar part of the peel juice solids is of a nitrogenous nature, as the small differences observed do not go very far toward accounting for the much greater differences in solids.

In general, specific gravity variations closely followed the data on soluble solids. Similar high values were obtained for the late season peel juice, showing conclusively that the high solids values were due not merely to some minor nonsugar constituent having an abnormal influence on refractive index, but to considerable amounts of some other substance or substances that influenced solution densities as well.

To characterize the juices further,

**Table I. Ash in Segment and Peel Juices**

Type of Juice	Date of Collection	Ash, %
Segment	3-4-55	0.435
Segment	5-11-55	0.464
Peel	5-4-55	0.502
Peel	5-11-55	0.508

relative viscosity was determined. While these values were nearly always greater for the peel juices than for the other types during the latter part of the season, the differences were small and were probably the result of higher concentrations of known constituents rather than of any appreciable amount of substances like dextrans. The viscosity curves show some resemblance to those for soluble pectic substances, but the correlation is not impressive.

The color curves resemble those of the flavonoids closely enough to suggest that the flavonoids contribute to the total color in filtered orange juices.

Peel juices were far below the segment and commercial juices in fluorescence. With the exception of a slight upturn for peel juice at the end of the season, there was little in the way of trends. The most striking feature lies in the comparison of the peel juice fluorescence curve with the color curve for these juices. These curves show strong negative correlation—when the color is high, the fluorescence is low. If the color is related to flavonoid content, perhaps the flavonoids present have a quenching effect on fluorescence. Whether this is due to their absorption of the incident ultraviolet light or to absorption of the emergent visible light is not known. If a connection between the flavonoids and the quenching effect could be established, it would suggest a possible basis for estimating flavonoids. Grapefruit segment, commercial, and peel juices all fluoresced so brilliantly that, even at one fourth the concentration of the orange juices, the meter needle was thrown off scale.

The results of the taste tests are given in Table II. In all cases, 20 judgments were made by the laboratory panel.

In all instances the taste panel was able to detect the presence of 3% of peel juice in reconstituted orange juice with

significance. The peel juice composites for the first two quarters of the season were identified with the least certainty, while the composite of the third quarter was identified with the greatest assurance. When tasted full strength, the clarified peel juices had a disagreeable flavor characterized as being bitter, astringent, or harsh.

Although flavonoids are probably involved in the bitter flavor of peel juice, it has not been possible to show to what extent this is true. Two other classes of compounds that might account for the bitter flavor are alkaloids and tannins. In previous work at this laboratory on peel juices not belonging to the present series, it was not possible to detect alkaloids. Tannins were found to be present in concentrations of less than 0.05%, which is lower than that of many nonbitter fruit juices.

#### Acknowledgment

The authors wish to acknowledge the generous counsel and assistance of W. C. Scott, W. H. Miller, and J. C. Lastinger of this laboratory.

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Received for review April 23, 1956. Accepted August 7, 1956. Division of Agricultural and Food Chemistry, 129th Meeting ACS, Dallas, Texas, April 1956. The use of trade names in this article is for identification only and implies no endorsement of manufacturer or product. U. S. Citrus Products Station is one of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.

**Table II. Taste Perception of Composite Peel Juices in Reconstituted Orange Juice Concentrate**

Composite Sample	% Composite Added to Juice	Judgments		Significance, % Level
		Correct	Incorrect	
Dec. 14-Jan. 11	3.0	12	8	5.0
Jan. 25-Feb. 23	3.0	12	8	5.0
April 6-May 4	3.0	14	6	0.1
May 18-June 15	3.0	13	7	1.0