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SUGAR LEVELS IN CANNED SINGLE STRENGTH GRAPEFRUIT JUICE FROM FLORIDA

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Abstract. **One hundred forty-nine samples of canned, single strength grapefruit juice from the 1979-80 season were analyzed for glucose, fructose and sucrose using high performance liquid chromatography (HPLC). One sample had additional sugar added and was properly labeled. Its sugar values differed from 4 to 7 standard deviations from respective mean values and was easily differentitated from the other 148 samples. Juices were supplied throughout the season on a regular basis from the 13 major processors of grapefruit juice in Florida. Average composition of the 148 samples in terms of fructose, glucose and sucrose was 2.39, 2.21 and 2.73 % (w/w) respectively. The average glucose/fructose ratio was 0.921 with a standard deviation of 0.0549. Sucrose concentrations were highly variable, ranging from 0.2 to 5.3% (w/w). The average percentage of sucrose compared to total sugars was 36.6%. This information can be used to help judge the authenticity of Florida grapefruit juice.**

The recent rise in the price of citrus juices has encouraged some out of state processors of juice to substitute cheaper materials in their product labeled 100% juice. In order to protect the consumer from this economic fraud an exact knowledge of the chemical composition of citrus juices is required. The natural range of each chemical component must be established for different cultivars, horticultural practices, climates and processing practices. The Europeans, particularly the French, Germans, and Dutch, have been very active in defining juices in terms of their detailed chemical composition. The juice definition program (1) initiated by the Florida Department of Citrus defined the chemical composition of Florida orange juice. However no similiar program has been initiated for Florida grapefruit juice. The Germans, French, and Dutch have recently established detailed chemical composition standards for grapefruit juice, GFJ. Unfortunately most, if not all, of this information is based on non-Florida juice.

Sugars are the major chemical component in both orange and grapefruit juices. Both the German "RSK Values" (2) and the Dutch "Authenticity Standards" (3) include detailed acceptable sugar values for grapefruit juice. Therefore it is the purpose of this paper to establish the normal distribution of individual sugars, total sugars and sugar ratios for Florida grapefruit juice over an entire season and to determine if they would meet European standards.

Materials and Methods

Reagents and standards. Baker Analyzed high performance liquid chromatography (HPLC) grade acetonitrile (J. T. Baker Chemical Co., Phillipsburg, PA.) was used to prepare the chromatographic mobile phase. Laboratory deionized water was further purified using a Milli-Q (Millipore, Milford, MA) water purification system. High grade sucrose, glucose and fructose were obtained as crystalline standards from the Sigma Chemical Co. (St. Louis, MO). Each sugar was dried for 2 hr at 60°C under vacuum (about 50-mm Hg or less) and cooled in a desiccator before weighing. Five grams of a standard consisting of 2% glucose, 2% fructose and 4% sucrose (w/w) was prepared fresh each week.

Equipment. The HPLC system consisted of a Waters (Milford, MA) model M-6000A pump, a model 710B WISP auto sampler and a R-401 differential refractive index detector. Chromatographic solvent was kept in the reference side of the detector. Chromatographic peaks were integrated using a Spectra—Physics (San Jose, CA) model 4000 recording integrator.

Chromatography. A DuPont (Wilmington, DE) Zorbax NH_2 column 25 cm. x 4.6 mm i.d. was used to separate the sugars. A 5-cm. NH_2 Brownlee (Santa Clara, CA) guard column was used at the head of the analytical column. The chromatographic solvent consisted of 75% CH_3CN and 25% H_2O (v/v). Flow rate was 1.0 ml/min. Solvents were degassed prior to use with vacuum in an ultrasonic bath. The column was thermally stabilized with 1/2-inch preformed foam rubber insulation to stabilize the baseline.

Sample preparation. Single strength grapefruit juice was centrifuged in a bench top centrifuge (International Clinical) for 5 minutes at the highest setting. Approximately 5-6 ml of the centrifuged juice was passed through a Waters C_{18} Sep Pak or Baker 10 SPE C_{18} (6 ml) cartridge that had been conditioned by rinsing with 5 ml of MeOH and then 10 ml of deionized water. The first 2 ml of juice through the cartridge were discarded. The final 3 ml were collected and filtered (Millipore 3μ filter with micron pre-

Table 1. 1979-80 grapefruit juice sugar summary.[z]

	Mean	SD	Maximum	Minimum
Fructose	2.38[y]	0.320	3.50	1.70
Glucose	2.18[y]	0.329	3.40	1.60
Sucrose	2.74[y]	0.554	5.30	0.20
Total sugars	7.47[y]	0.782	9.50	4.90
Glucose/fructose	0.919[y]	0.058	1.05	0.724
% sucrose	36.6	5.85	56.4	2.74

[z]for 148 samples
[y]concentration in weight % (w/w)

filter) into a 4-ml septum sealed vial. Samples were refrigerated prior to analysis.

Calculations. All calculations were based on the external calibration method. Chromatographic response factors were established daily using average values obtained from injections of the standard. The standard was reinjected and the response factor updated after each 5 samples. Since the injection volume of the standard was the same as the samples, no volume correction was necessary. Sugar concentrations were reported as weight per cent.

Results and Discussion

Fructose and glucose concentrations. As shown in Table 1 the average fructose and glucose values for the 148 GFJ samples were 2.38% and 2.18% (w/w), respectively. The corresponding standard deviations were 0.320 and 0.329. Fructose concentration ranged from 1.7 to 3.5%; however, 93% of the samples were found from 1.8 to 2.9. A frequency histogram for fructose is shown in Fig. 1. Only 7 samples were greater than 2.9% fructose. Statistically, one would expect to find 95% of the samples within ± 2 standard deviations (σ) and 99.7% within ± 3 standard deviations. Only 2 samples fell outside the 3σ limit; both samples contained 3.5% fructose.

The distribution of glucose was not as symmetrical as that of fructose. Glucose concentrations ranged from 1.6 to 3.4% (w/w) with 93% falling between 1.6 and 2.7%. Again there were 2 samples (both 3.4%) that had glucose values greater than 3.18% or 3 standard diviations from the mean. Since these same 2 samples were also outside the 3σ limit for fructose, they are probably outliers.

The German RSK values (2) set an allowable range of 1.8 to 5.0% glucose and 1.9 to 5.0% fructose. None of the Florida GFJ's exceeded the maximum values but 3 samples were just below the 1.9% minimum fructose value and eleven samples were just below the 1.8% glucose minimum value.

Glucose/fructose ratio. The glucose/fructose ratio is a key indicator for determining the authenticity of citrus juices. Both the German RSK system (2) and the Dutch "Authenticity Criteria" (3) specify similar maximum glucose/fructose ratios. As shown in Table 1 the average ratio for 148 samples was 0.919. There were only 3 samples which exceeded the Dutch maximum glucose/fructose ratio of 1.03. The Germans have set a very tight glucose/fructose range of 0.9 to 1.02. As seen in Fig. 2 only 3 samples exceeded the maximum ratio whereas 26% of the 158 samples in this study fell below the 0.9 minimum value. This suggests that the minimum ratio should be lowered to at least 0.85 for Florida GFJ.

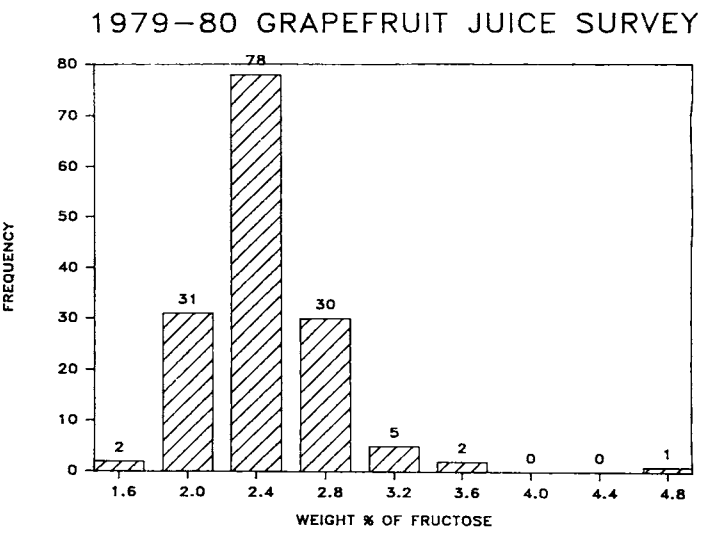

Fig. 1. Distribution of HPLC fructose concentrations in Florida grapefruit juice. Note the sample at 4.8% fructose. This was the single sample of "sugar add" grapefruit juice.

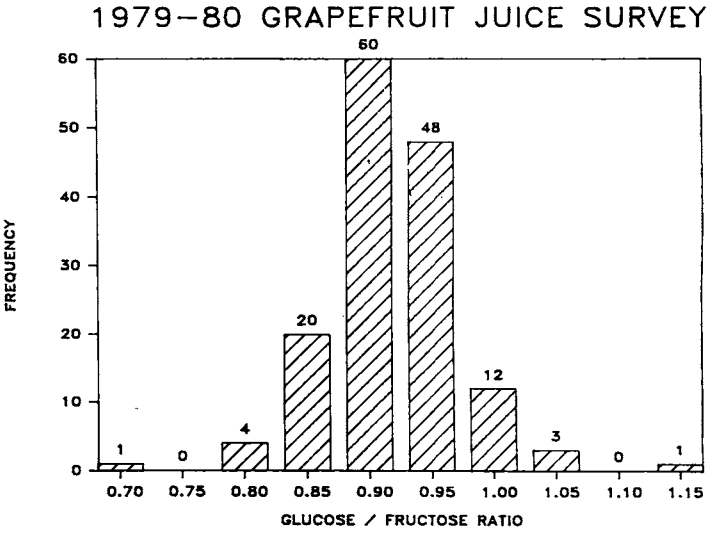

Fig. 2. Distribution of the glucose/fructose ratio in Florida grapefruit juice. The sample with a ratio of 1.15 was the "sugar add" sample.

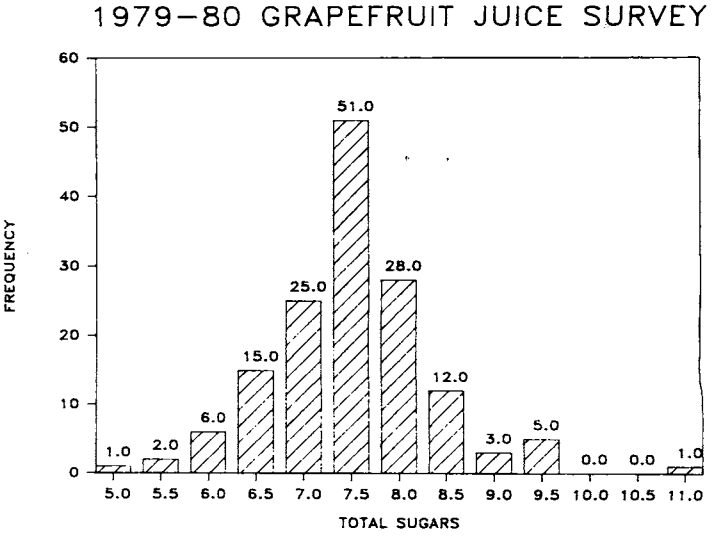

Fig. 3. Histogram of total HPLC sugars (glucose + fructose + sucrose) in Florida grapefruit juice. The sample on the far right (10.9%) was the "sugar add" sample.

The distribution of the glucose/fructose ratio is shown in Figure 2. The range for ± 3 standard deviations is 0.764 to 1.07. Therefore only the single sample at 0.72 falls outside of this range. This sample contained 2.9% fructose, 2.1% glucose, and only 1.2% sucrose (which is very low for sucrose). This sample is probably an outlier. The sample with a ratio of 1.15 will be explained next.

Sugar added grapefruit juice. In order to improve consumer acceptance, sugar is sometimes added to GFJ which is high in acid, low in sugars and high in bitter compounds. This process is perfectly legal providing the words "sugar added" appears on the label. One such sample was inadvertently included in this study. It was easily distinguished from the other 148 samples. In Figure 1 this sample is the extreme outlier (>6σ) shown at 4.8% fructose. The added sugar also disturbed the natural glucose/fructose ratio. This sample had a ratio of 1.15 which was greater than 4 standard deviations from the mean. As shown in Fig. 3 the sample with added sugars produced a very high total sugar value. Total sugars were 10.9% which was greater than four standard deviations from the mean of the other 148 grapefruit juices.

Sucrose concentrations and percent sucrose. Sucrose concentrations were highly variable ranging from a low of 0.2% to a high of 5.3%. As shown in Table 1 the average sucrose concentration was 2.7% (w/w). The standard deviation was 0.554, which is fairly high. Even though there was a relatively tight distribution around the mean (138 out of 148 samples were within ±2σ) there were a few high and several very low values. Since GFJ is reasonably acidic, pH approximately 3.5, low sucrose values could be explained if one considers the possibility of acid hydrolysis of the sucrose into glucose and fructose. High sucrose values are harder to explain. Two samples containing 4.5 and 5.3% sucrose, had concentrations greater than 3 standard deviations from the mean (4.40%).

Sucrose comprises a considerably smaller portion of the total sugars in grapefruit juice as compared to the normal 50% in orange juice. Since sucrose concentrations were highly variable, the percentage of sucrose was also highly variable with values ranging from 2.7 to 56.4%. Low percentages of sucrose may be explained in the same manner as low sucrose concentrations. Of the 2 previous samples which contained abnormally high sucrose concentrations only one also had an abnormally high (>3σ) percent sucrose and must therefore be considered an outlier.

Total sugars. As shown in Fig. 3, total sugars were distributed over a wide range of values. Total sugars (glucose + fructose + sucrose) ranged from 4.9 to 9.5%. The value at 10.9% was due to the "sugar add" sample and was therefore expected to be high. Average total sugars was 7.47% with a standard deviation of 0.782. Of the 148 GFJ's only one was outside the limit of ± 3σ (5.12-9.82%). The glucose/fructose ratio and percent sucrose for this sample was very normal (0.941 and 32.6%); however, as might be expected the concentrations of both fructose and glucose were low (1.7 and 1.6%, respectively). Therefore this sample is probably an outlier.

Conclusion

Glucose, fructose, and sucrose concentration profiles have now been established for Florida canned grapefruit juice. Fortunately the vast majority of Florida product meets European sugar standards for grapefruit juice. It was also interesting to note that "sugar added" grapefruit juice is readily distinguished from the normal grapefruit juice sugar profile.

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THE COSTS AND BENEFITS OF TRANSPORTING 72°BRIX ORANGE CONCENTRATE

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Abstract. **Bulk citrus concentrate is customarily shipped in 62 to 65°Brix concentrations. It now appears technically feasible to increase the concentration level to 72°Brix. In this paper, the economic impacts on transportation costs of going to 72°Brix are examined. The impacts on the costs of transporting concentrate to various destinations are calculated as well as the expected change in Florida's total transport bill for bulk concentrate. Changes in the relative costs of transporting concentrate to a major U.S. market from Florida and Brazil are estimated. These estimates are made for 4 scenarios which differ regarding adoption of 72°Brix. The effects of changing fuel costs on the relative costs of transporting concentrate from Florida and Brazil are examined. Florida has an advantage over Brazil in the amount of fuel required to deliver bulk concentrate. This is about a $417 storage/delivery cost advantage per truckload. The new 72°Brix technology**

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