#### LITERATURE CITED

 Beisel, C. Gordon, R. W. Dean, R. L. Kitchel, K. M. well, C. W. Nagel, and R. H. Vaughn. 1954. Sources d detection of Voges-Proskauer reactants in California nowell, C. W. Nagel, and R. H. Vaughn. 1954. Sources and detection of Voges-Proskauer reactants in California Valencia orange juice. Food Research. 19: 633-643.

2. Byer, E. M. 1954. Visual detection of either diacetyl or acetylmethylcarbinol in frozen concenterated orange juice. Food Technol. 8: 173-174.

3. Hays, G. L., and D. W. Riester. 1952. The control of "off-odor" spoilage in frozen concentrated orange inice.

3. Hays, G. L., and D. W. Riester. 1952. The control of "off-odor" spoilage in frozen concentrated orange juice. Food Technol. 6: 386-389.

4. Hill, E. C., F. W. Wenzel, and A. Barreto. 1954. Colorimetric method for detection of microbiological spoilage in citrus juices. Food Technol. 8: 168-171.

5. Hill, E. C., and F. W. Wenzel. 1957. The diacetyl test as an aid for quality control of citrus products. I. Detection of bacterial growth in orange juice during concentration. Food Technol. 11: 240-243. centration. Food Technol. 11: 240-243.

6. Minute Maid Company. Laboratory data (Unpublish-

6. Minute Maid Company. Dabotatory and J. F. Folinazzo. 7. Murdock, D. I., V. S. Troy, and J. F. Folinazzo. 1952. Development of off-flavor in 20° Brix orange concentrate inoculated with certain strains of lactobacilli and Leuconostoc. Food Technol. 6: 127-129.

8. Murdock, D. I., and R. E. Dennis. 1964. Detection of diacetyl and acetylmethylcarbinol in processing frozen concentrated orange inice—a preliminary report. Proc. Fla.

concentrated orange juice—a preliminary report. Proc. Fla.

State Hort. Soc. 77: 297-305. 9. Murdock, D. I. 1965. State Hort. Soc. 77: 297-305.

9. Murdock, D. I. 1965. Detection of diacetyl and acetylmethylcarbinol in processing frozen concentrated orange juice—an investigation of methods employed. Proc. Fla. State Hort. Sco. 78: 274-283.

10. Murdock, D. I., and C. H. Brokaw. 1958. Sanitary control in processing citrus concentrates. I. Some specific sources of microbial contamination from fruit bins to extractors. Food Technol. 12: 573-576.

11. Murdock, D. I. Personal obeservations of author.

# COMPARISON OF SUBJECTIVE AND OBJECTIVE METHODS FOR DETERMINING THE COLOR OF RECONSTITUTED FROZEN CONCENTRATED ORANGE JUICE

GEORGE J. EDWARDS<sup>2</sup>, F. W. WENZEL<sup>2</sup>, R. L. HUGGART<sup>3</sup> AND R. W. BARRON<sup>3</sup>

## ABSTRACT

One subjective and two objective methods were used to determine the color of reconstituted frozen concentrated orange juice. The USDA color scores for 21 samples ranged from 32 to 37 points when this subjective method was used. Two objective methods were used: (a) the Hunter Color and Color Difference Meter to obtain tristimulus color values and (b) the Bausch and Lomb Spectronic 505 recording spectrophotometer to obtain spectral curves. Hunter Rd values ranged from 20.3 to 27.1; the  $\alpha$  values values from -7.5 to -2.8; and the b values from 28.1 to 29.8. Dominant wavelengths computed from the spectral curves for the reconstituted juices, ranged from 576 to 581 m<sub>\mu</sub>; purity from 59 to 90%; and brightness from 20.3 to 33.8%. As the visual color score increased, the Hunter a value increased and the Rd value decreased; also, brightness decreased but no trend was evident between either the dominant wavelength or purity and the color score.

#### INTRODUCTION

Color to different disciplines of science means various things. To the chemist it is dye and pigments. To the physicist color is a phenomena in the field of optics and electromagnetc radiation. To the physiologist and psychologist color denotes a sensation to the human observer.

There are many procedures in use today that compare the color of one object with that of another or give a value for the color difference between them. Such methods are either subjective or objective, the former depending upon a visual evaluation while the latter uses different instruments.

Subjective methods.—The Maerz and Paul Dictionary of Color (5) contains examples of many colors. Plate 10 on page 43 of this book shows various colors that could apply to orange

The Macbeth-Munsell Disk Colorimeter (3, 4) can be used so that overlapping color wedges are spun and the resulting color compared to that in samples of orange juice. By varying the amount of white, gray, yellow, and orange, the color of orange juice may be matched.

Another example of a subjective method is the use of USDA color comparator tubes to obtain a color score for orange juice. A trial set at such tubes consisted of colored viscous plastic in capped glass tubes. These were avail-

Florida Agricultural Experiment Stations Journal Ser-

ies No. 2529.

1Cooperative research by the Florida Citrus Experiment Station and Florida Citrus Commission.

2Florida Citrus Experiment Station, Lake Alfred.

3Florida Citrus Commission, Lake Alfred.

able in Florida for a number of years and were numbered 1, 2, 3, and 4 corresponding to color score points 32, 34, 36, and 38. This trial set of color comparator tubes wes replaced in 1963 with a set of tubes made entirely of colored plastic (6). These tubes were designated as 0J1, 0J2, 0J3, 0J4, 0J5, and 0J6 and were referred to as USDA Orange Juice Color Standards. The procedure for evaluating the color of reconstituted frozen concentrated orange juice by using the 0J2, 0J3, 0J4, and 0J5 tubes is given in the U.S. Standards for Grades of Frozen Concentrated Orange Juice (7).

Objective methods.—Kramer and Twigg (3) and MacKinney and Little (4) described many instruments for measuring objectively the color or color difference of substances. Instruments described include spectrophotometers, Gardner Color and Color Difference Meter, Hunterlab Color and Color Difference Meter, Colormaster Differential Colorimeter, Color Eye, Photovolt Reflection Meter, and Agtron.

Color terminology.—Kramer and Twigg (3) list some of the physical and sensory terms used to denote different color attributes in the following manner.

Physical Measurement
Radiant energy
Reflectance
Dominate wavelength
Purity
Sensory Term Equivalent
Light
Lightness, value
Hue, color
Chrome, intensity, strength

The purpose of this paper is to present to the citrus processing and other related industries information pertaining to the use of subjective and objective methods for determining the color of reconstituted frozen concentrated orange juice. Data are needed so that undesirable variations, occurring when color scores of juices are subjectively determined by visual comparison, may be eliminated by the use of objective instrumentation.

## EXPERIMENTAL PROCEDURES

Sample preparation.—A set of reconstituted frozen concentrated orange juices were prepared by thawing and mixing together different samples of commercial frozen Florida orange concentrate so that the reconstituted juices would

have a wide range of USDA color scores and Hunter Color Difference Meter values.

Use of USDA orange juice color standards.—The set of USDA plastic color standards (0J2, 0J3, 0J4, 0J5, and 0J6) were used visually to obtain the color scores for these reconstituted orange juices. The juices were placed in 1-inch-OD screw-cap culture tubes. The juices and the standard tubes were viewed together in a Macbeth Examolite daylight model EBA-220 with a rated color temperature of 7400° Kelvin. The averages of the total score points given by five judges to each juice were used.

Use of Hunter Color and Color-Difference Meter.—The Hunter Color and Color Difference Meter (3, 4) was used in the objective evaluation of the color of the 21 reconstituted orange juices. This instrument is a photoelectric tristimulus colorimeter. It can measure small differences in color with its three filters, which approximate the standard observer of the International Commission of Illumination. Most of the information (1, 2, 8) on the color of citrus concentrates and juices has been obtained by the authors using this instrument. Three readings are obtained: the Rr, a, and b values. The Rdvalue indicates the lightness (whiteness) of a juice sample. Readings of either 0 or 100 mean that the sample is black or white, respectively. Values for Rd between 0 and 100 indicate different shades of grayness. The a values are measures of redness when positive or greeness when negative. Yellowness or blueness are indicated by positive or negative b values, respectively.

Use of Bausch and Lomb Spectronic 505 recording spectrophotometer.—A Bausch and Lomb Spectronic 505 (B & L g05) recording spectrophotometer was loaned to the Citrus Experiment Station for a short time by W. H. Curtin and Company, Jacksonville, Florida. This instrument made it possible to obtain color data by another objective method. Equipped with a reflectance accessory attachment, the B & L 505 recorded the reflectance from a sample of juice as compared to the reflection from magnesium oxide. Thus, a spectral reflectance curve was obtained over the wavelengths from 440 to 700 mu. This instrument recorded directly on a trichromatic coefficient computing chart. These charts made it simple for conversion to C.I.E. trichromatic coefficients (3, 4) which are needed to determine the dominant wavelength (DWL). purity, and brightness of the light reflected from the juice.

#### RESULTS AND DISCUSSION

Spectral curves for USDA plastic-in-glass color comparator tubes.—Spectral curves from the B & L 505 of the USDA plastic-in-glass color comparator tubes are shown in Figure 1. Characteristics of these special curves were calculated and are presented in Table 1. The dominant wavelength increased with the color scores (Table 1) but there was only 1 m $_{\mu}$  difference between any two adjacent scores. Purity increased and brightness decreased as the color score increased.

USDA color scores and Hunter Color Difference Meter alues for reconstituted frozen concentrated orange juices.—Color scores, Hunter Color Difference Meter values and spectral characteristics for 21 samples of reconstituted frozen concentrated orange juice are listed in Table 2. Color scores ranged from 32 to 37 points when the OJ set of USDA plastic color comparator tubes were used. The Rd values ranged from 20.3 to 27.1; the a values from -7.5 to -2.8; and the b values from 28.1 to 29.8. In general, the Rd values decreased and the a values increased as the color scores increased.

Spectral curves for reconstituted frozen concentrated orange juices.—Some typical spectral curves for reconstituted orange juices, ranging in score from 32 to 37, are shown in Figure 2. Spectral curves for the orange juices had higher dominant wavelenghs (Table 2) than those for the USDA color comparator tubes (Table 1), indicating more redness in the juices as compared to that in the plastic-in-glass tubes. The dominant wavelengths of the spectral curves for these comparator tubes ranged from 571 to 574 m $_\mu$  while those for the juice curves ranged from 576 to 581 m $_\mu$ . This shift in dominant

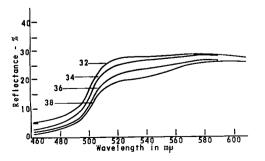


Fig. 1.—Spectral curves for color of trial set of plastic-inglass color comparator tubes with score point range of 32-38.

Table 1. Dominant wavelength, purity, and brightness of the color of USDA plastic-in-glass color comparator tubes

Color	DWL	Purity	Brightness		
score	mμ	<u></u> %	%		
32	571	70	25.5		
34	572	74	24.6		
36	573	81	22.3		
38	574	84	21.0		

wavelengths was probably due to the smaller area presented to the instrument by the round comparator tube as compared to the larger area exposed when the flat cell was used to hold the juice. The dominant wavelength range of 578 to 580  $m_{\mu}$  included those for 78% of the juice samples.

The slope of a spectral curve (Figure 2) indicates the dominant wavelength. The height of the portion of a spiral curve on the right side of the slope is indicative of the brightness of the color in orange juice. As the height of this portion of the curve becomes greater, the brightness increases but the color score decreases, as is shown in Figure 2.

When brightness and purity, calculated from the spectral curves for juices, are compared with the color scores (Table 2), only brightness showed any relation to the score in that it decreased as the score increased. As mentioned previously, the Hunter a value become greater as the score increases (Table 2). When the Hunter a values and the brightness of the color of orange juices are plotted, as in Figure 3, parallel diagonal lines can be drawn which will separate the scattered points into groups to which visual color scores may be assigned. Ideally, each of the parallel lines, separating the different score groups, would be equi-distance apart. Since this is not true, perfect correlation between the Hunter a values and the brightness of the color of the reconstituted juices and the color scores does not exist.

In conclusion, subjective USDA color scores, objective Hunter Color Difference Meter values and spectral curves for 21 samples of reconstituted frozen concentrated orange juice were obtained. As the color score increased, the Hunter a value increased and the Rd value decreased. Based on special curve data, the brightness of the color of the reconstituted juices decreased as the color score increased. However, there was

Table 2.	Color	scores,	Hunter	Color	Difference	Meter	values,	and Spectral	

characteristics for reconstituted frozen concentrated orange juices

Sample Color number scores <sup>1</sup> , <sup>2</sup>		HCDM values <sup>3</sup>			DWL <sup>4</sup>	Purity <sup>4</sup>	Brightness <sup>4</sup>
	$R_{d}$	a	ъ	mµ	%	%	
1	32	27.1	-6.7	28.5	578	72	33.8
2	33	25.4	-6.9	28.3	577	76	31.3
3	15	26.2	-7.4	28.3	578	75	29.4
4	11	25.5	-7.5	28.1	577	73	29.8
5	<b>!</b> 1	25.0	-7.1	28.2	578	74	29.1
6	34	26.0	-6.2	29.2	578	74	31.0
7	35	22.8	-5.4	29.0	579	76	26.4
8	11	24.2	-5.8	29.0	580	80	26.5
9	11	23.6	-6.5	28.9	579	80	26.3
10	11	23.8	-5.4	29.3	579	81	26.5
11	11	22.8	-4.6	29.4	580	80	25.5
12	11	22.4	-4.8	29.1	576	90	25.8
13	11	23.6	-4.9	29.2	579	80	26.9
14	11	23.8	-4.3	29.8	580	78	28.0
15	11	23.7	-5.0	29.1	579	74	28.3
16	36	20.9	-3.8	28.9	581	75	24.2
17	*11	21.8	-4.0	29.3	580	80	25.0
18	11	21.9	-3.0	29.7	576	88	25.1
19	11	21.4	-3.8	28.3	580	76	24.4
20	37	21.7	-2.8	29.6	580	79	25.2
21	11	20.3	-3.1	29.4	580	82	20.3

 $^{
m l}$ Color scores determined visually using USDA plastic comparator tubes designated as 0J3, 0J4, 0J5, and 0J6.

 $2_{
m The}$  color scores were determined by personnel of the Florida Citrus Commission and the Florida Citrus Experiment Station.

 $^3$ Hunter Color Difference Meter values.

no trend evident between either the dominant wavelength or the purity of the color of the reconstituted orange juices and the USDA color scores. On the basis of the data reported in this paper, the Huner Color Difference Meter values, a and Rd, provide the best indicator for determining color score for orange juices.

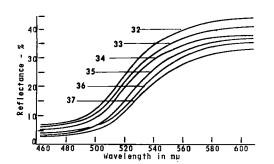


Fig. 2.—Spectral curves for color of reconstituted frozen concentrated orange juices with USDA score point range of

### REFERENCES

1. Huggart, R. L. and F. W. Wenzel. 1954. Measurement and Control of Color of Orange Cencentrate. Proc. Florida State Hort. Soc. 67, 210-216.
2. Huggart, R. L. and F. W. Wenzel. 1955. Color Differences of Citrus Juices and Concentrates Using the Hunter Color Difference Meter. Food Technol. 9, 27-29.
3. Kramer, Amihud and Bernard A. Twigg. 1962. Fundamentals of Quality Control for the Food Industry. The Avi Publishing Company, Inc., Westport, Connecticut.

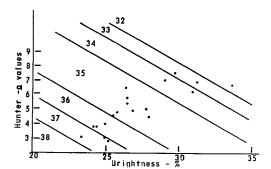


Fig. 3.—Relation of score of reconstituted orange juices to brightness and Hunter a values.

<sup>4</sup>Computed from spectral curves.

4. MacKinney, Gordon and Angela C. Little. lor of Foods. The Avi Publishing Company, 1962. The Avi Publishing

Color of Foods. The Avi Publishing Company, Inc., Westport, Connecticut.

5. Maerz, A. and M. Rea Paul. 1930. A Dictionary of Color. McGraw-Hill Book Company, Inc., New York, New

6. USDA. 1963. Scoring color of orange juice products with USDA-1963 orange juice color standards. U.S. Dept. Agr., Agr. Marketing Service, Washington, D. C.

7. USDA. 1964. United States Standards for Grades of Frozen Concentrated Orange Juice. U.S. Dept. Agr., Agr. Marketing Service, Washington, D. C.
8. Wenzel, F. W. and R. L. Huggart. 1962. Relation Between Hunter Color—Difference Meter Values and Visual Color of Commercial Frozen Concentrated Orange Juice. Proc. Florida State Hort. Soc. 75, 331-336.

# A RAPID PROCEDURE FOR EXTRACTION OF NARINGIN FROM GRAPEFRUIT RIND

#### PAUL L. DAVIS<sup>1</sup>

### Introduction

The extraction of naringin, the principal flavanoid of grapefruit rind, as described by Kesterson and Hendrickson (3), is a long procedure. A rapid Soxhlet extraction is described which saves considerable time in routine analyses. When extracts from the two procedures were compared, the percent of naringin extracted was almost identical. The Soxhlet extraction requires 3 hours, the other procedure, 20 hours. The Soxhlet extraction requires fewer steps and is thus less subject to error.

#### EXPERIMENTAL METHODS

Samples of grapefruit rind were removed with a cork borer; 40.0 grams were ground in a Waring Blendor with 100 ml of ethyl alcohol for 1 minute. The mixture was filtered, and filtrate and residue were each divided into two equal portions. To insure equal portions, the filtrate was made up to 200 ml before division; the residue was air dried to remove alcohol and was weighed into equal portions.

The two extraction procedures were compared in seven separate tests:

A-Soxhlet extraction. One portion of the residue was placed in an extraction thimble; one portion of the filtrate was placed in an extraction flask with 50 ml of ethyl alcohol, and the extraction was carried out for 3 hours. The filtrate in the flask was then made up to 250 ml and then diluted 1 to 100 for analysis.

B-Kesterson-Hendrickson extraction. The other portions of the filtrate and residue were combined and allowed to stand for 16 hours with

<sup>1</sup>Market Quality Research Division, Agricultural Research Service, U.S. Department of Agriculture, Orlando.

occasional stirring. The residue was further extracted for 2 hours with water containing calcium oxide and then with water heated to 95° C immediately and allowed to stand for 2 hours. The three filtrates were combined, made up to 500 ml, and diluted 1 to 50 for analysis.

The Davis (1) method of analysis depends upon the production of a yellow color on the addition of alkali in the presence of diethylene glycol. A Bausch and Lomb Spectronic 20 Spectrophotometer was used to measure color development. Although small amounts of materials other than naringin, which form a yellow color under these conditions (4), may be present, this method has been found suitable for routine assay of citrus flavanoids (2).

In each test, two aliquots of each diluted extract were taken, and measurements of color development were averaged.

### RESULTS AND DISCUSSION

Separate analyses of extracts from each step in the Kesterson-Hendrickson procedure showed that about 72% of the naringin was extracted in the first extraction (alcohol); 18% in the second (water-calcium oxide); and 10% in the third extraction (water).

When extracts from the two procedures were compared, naringin contents were almost identical (Table 1). The slightly higher naringin content of the Soxhlet extract probably indicates more complete extraction. The Soxhlet extraction requires 3 hours, the other procedure, 20 hours. The Soxhlet extraction requires fewer steps and is thus less subject to error.

For routine analyses, samples of rind are weighed, ground for 1 minute in alcohol, transferred to a Soxhlet apparatus, and extracted for