California Olive Oil Studies

*Proposing Tests and Standards for Quality Control of a Valuable Domestic Edible Oil Product**

By P. F. NICHOLS[1] *and* B. E. LESLEY[2]

WHILE tests and standards for the purity of all oils of commerce are well established and generally reliable, tests for quality have not found so nearly universal acceptance. Particularly is this true in the case of California olive oils, for which the time-honored and the only test for quality of an exact, impersonal, laboratory nature is that of free fatty acid content. The tasting test for flavor and rancidity is of course of great value and gives at least reasonably consistent comparisons when applied by experts. No difficulty should be experienced with this test in distinguishing between oils that are perfectly satisfactory and those that are distinctly bad or off-flavor. Nevertheless the test is so largely a matter of personal taste and judgment that gradations in quality cannot be exactly defined and it is impossible to establish by its means standards that can be rigidly adhered to by all persons. For this reason it permits a great deal of difference of opinion as to intermediate grades and fine distinctions.

An important factor in quality in olive oil is the relative freedom from rancidity. It is known that the free fatty acid content of oil is not a direct measure of rancidity, since rancid flavors and odors may remain after all free acid has been neutralized. The use of the free fatty acid determination as an index of rancidity is likely to be very misleading, therefore, particularly if the oil has undergone any sort of refining. This fact has been realized by investigators in the past, and several laboratory tests for rancidity have been proposed ([1,2,3,4]).

Other factors in quality are the color, which in several other oils is determined by the Lovibond tintometer and color glasses, and the "body" which appears to be measurable by various tests for viscosity.

These tests have been applied by the writers to oils kindly furnished and described by California olive oil manufacturers, to California and imported olive oils bought on the open market, and to a few olive oil substitutes also bought on the open market. While the work so accomplished certainly does not warrant the adoption by the industry, or perhaps even the recommendation to the industry of standards at this time, the results are of interest in that they point out certain possibilities which could be tried out in a preliminary way now, if desired, and they also show some of the difficulties yet to be overcome.

Description of the Tests

THE tests used, described briefly, are as follows:

1. Color was determined by use of a tintometer designed by one of the writers (P. F. N.) in which the color of a standard depth of oil (5¼") in a flat-bottomed clear glass tube illuminated by a transmitted white light was matched by the interposition of standard yellow, red and blue Lovibond color glasses over a similar depth of distilled water illuminated in the same manner.

2. Relative viscosity was determined by observing the time in seconds required for a standard pipet held in a stationary vertical position to discharge the amount of oil contained between two marks on the stem respectively above and below the bulb. This number of seconds, corrected for temperature to 20 deg. C., was divided by the number of seconds required for the similar discharge of distilled water at 20 deg., and the quotient used as an index of relative viscosity.

3. Free fatty acid was determined as per cent oleic acid by bringing to boil, with shaking, approximately 20 grams of oil mixed with 50 c.c. of neutralized 95 per cent ethyl alcohol, and titrating with N/10 Na OH, using phenolphthalein as an indicator.

4. The Kreis test (1) was performed by shaking approximately 10 c.c. of oil with a similar amount of concentrated HCl, shaking for 30 seconds in a large, rubber-stoppered test tube, and adding 10 c.c. of 0.1% solution of phloroglucin and shaking again. A red color

* *Contribution from Fruit Products Laboratory, University of California.*

[1] Associate in Fruit Products.

[2] Formerly Assistant in Fruit Products.

developing on addition of the phloroglucin solution was considered positive, and gradations in the intensity of the test are shown in the Table 1 with the other tests.

5. The Issoglio test was run in two ways, namely, the oxidation following the digestion recommended by R. H. Kerr[1] and the oxidation following steam distillation as recommended by Issoglio[2].

6. The von Fellenberg test was run by adding to approximately 1 c.c. of oil about 1 c.c. petroleum ether and ½ c.c. Fuchsin sulfurous acid reagent as recommended by von Fellenberg[3]. An immediate blue color was considered as positive, the gradations in rate of color appearance being classified as shown in Table 1.

7. The Vintilesco and Popescu test was run by mixing 10 gms. oil with 4 to 5 drops of defibrinated horse blood, 10 drops of tincture of guaiac, and 10 c.c. hydrogen peroxide as recommended by Vintilesco and Popescu[4]. A blue color was considered as positive, the gradations in intensity being classified as shown in Table 1.

Results Obtained

THE results of these tests are summarized in Table 2, in which are shown the types and number of samples studied, together with averages, maxima, and minima.

TABLE I

Test	Kreis		Von Fellenberg		Vintilesco and Popescu	
Positive	+ + +	= 7	+ + +	= 5		
	+ +	= 6	+ +	= 4	+ +	= 4
	+	= 5	+	= 3	+	= 3
Doubtful	+ (pink)	= 4	+ (weak)	= 2	+ (weak)	= 2
	—(orange)	= 3				
Negative	—	= 2	—	= 1	—	= 1
Negative	— —	= 1				

TABLE II

	Color—Lovibond Y	R	B	Relative Viscosity	Free Fatty Acid	Kreis Test	Issoglio Number Digestion	Distillation	von Fellenberg Test	Vintilescu and Popescu Test
					21 California Olive oils—Edible					
Ave.	80.8	4.8	2.0	15.5	1.00	— Orange to —	16.4	7.0	+ (weak +	+(weak)to +
Max.	90.	17.5	6.7	16.4	6.24	+	42.0	14.2	+ +	+ +
Min.	60.	.5	—	14.5	0.16	— —	5.4	2.7	—	—
					4 California Olive oils—Doubtful					
Ave.	86.7	2.6	1.3	15.7	1.48	+ (pink)	9.7	5.1	+	+
Max.	90.	3.2	2.0	16.4	4.30	+ +	22.4	7.5	+ +	+ +
Min.	85.	1.3	0.0	15.2	0.23	— (orange)	2.9	2.7	+ (weak)	+ (weak)
					9 California Olive oils—Technical					
Ave.	87.	5.1	8.6	16.3	3.85	+ +	16.2	8.2	+ to + +	+ to + +
Max.	90.	10.0	15.0	17.2	7.38	+ + +	21.1	10.2	+ + +	+ +
Min.	85.	2.3	0.0	15.9	0.90	— (orange)	8.3	5.3	—	—
					4 Imported Olive oils—Edible					
Ave.	62.5	2.5	.5	15.7	.83	—	9.7	6.3	+ (weak)to +	+ (weak)
Max.	75.	4.3	1.5	16.1	1.83	+ (pink)	10.6	8.0	+	+ +
Min.	40.	.5	—	15.1	.40	— —	8.6	5.3	+ (weak)	—
					2 Olive Oil Substitutes					
Ave.	37.5	2.2	—	14.1	.06	+ (pink)	5.8	6.6	+ (weak)to —	—
Max.	60.	3.5	—	14.4	.09	+ +	8.0	8.4	+ (weak)	—
Min.	15.	1.0	—	13.8	0.3	—	3.5	4.9	—	—

Summary

FROM Table 2 it is seen that, in the samples studied, color was lowest in olive oil substitutes, then in imported oils, then in California edible, doubtful, and technical oils respectively.

Viscosity was lowest in olive oil substitutes, highest in California technical oils, and medium in all the rest. Imported oils appeared to have the same "body" as California edible oils.

Free fatty acid was lowest in substitute oils, and next lowest in imported olive oils.

With respect to the Kreis, Issoglio, von Fellenberg, and Vintilesco and Popescu tests for rancidity, the latter two tests appear unreliable. The Issoglio test, using the digestion method, seems to give too high results especially with unwashed oils; using the distillation method it gives too low results and fails to distinguish bad oils. The Kreis test appears to give reliable results more closely correlated with rancidity than do any of the others, but occasionally fails to distinguish bad oils or condemns good oils.

Conclusions and Recommendations

IT APPEARS from the study here reported that, on the average, California olive oil has very similar characteristics of color, relative viscosity, and free fatty acid to those of the imported olive oils some of which command a premium in price. The deviations from the average, however, are greater.

As an aid to the development of greater uniformity, therefore, it is recommended: first,

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tube and place a layer of absorbent cotton on the plate. Place the meats in the prepared tube and pour sufficient portions of petrolic ether on the meats to extract at least 5 grams of oil. Receive the extract in a tared flask. Evaporate the ether from the oil on a steam bath. Care must be taken to see that all the ether is removed from the oil. Weigh the oil, add 30 cc. neutralized alcohol (Formula 30) and titrate the free fatty acid of the oil with standard alkali, using phenolphthalein as the indicator. (0.1 N alkali is used if f. f. a. if low, but 0.25 N is used for oils with f. f. a. above 3 per cent). The addition of a small amount of petrolic ether before titration makes the end point sharper. The titration is performed in a flask which is shaken vigorously during the titration, the end point being taken when a permanent pink is obtained which persists for at least one minute.

$$\text{Per cent F. F. A.} = \frac{28.2 \times \text{Normality of alkali} \times \text{cc. used}}{\text{weight of oil}}$$

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that California oils be further studied along these lines, with the idea of calling attention to cases of considerable variation from the average; second, that the average figures of 80 yellow—5 red—2 blue for color, 15-16 for relative viscosity, and 1.0 per cent. for free fatty acid be considered as tentative standards for the purpose of such investigation; and third, that a slight but distinct positive Kreis test be considered tentatively as maximum permissible rancidity for the purpose of such investigation.

References

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[3] Fellenberg, T. von Mitt. Lebensm, Hgy. *15*: 198-208 (1924); Chem. Abs. *18*: 3731 (1924). (Original not seen.)

[4] Vintilesco, I., and Popescu, A. Bul. Acad. Sci. Roumaine, *4*: 151-157 (1915); J. Pharm. Chim. *12*: 318-323 (1915); Chem. Abs. *10*: 646 (1916). (Original not seen.)

Soap Chemists' Report

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The committee and cooperating laboratories follow:

Ralph W. Bailey, Stillwell & Gladding, Inc., New York City; Chas. J. Gundel, Works Chemist, Fels & Co., Philadelphia; L. F. Hoyt, Manager, Research Dept., Larkin Co., Inc., Buffalo; Martin H. Ittner, Chief Chemist, Colgate-Palmolive-Peet Co., Jersey City; H. J. Morrison, The Procter & Gamble Co., Ivorydale; W. D. Richardson, Chief Chemist, Swift & Co., Chicago; M. L. Sheely, Chief Chemist, Armour Soap Works, Chicago; M. L. Sheely, Chief Chemist, Armour Soap Works, *Babbitt Lab.*, Jersey City; H. P. Trevithick, Chief Chemist, New York Produce Exchange, New York City; R. B. Trusler, Industrial Fellow, Mellon Institute, Pittsburgh; H. C. Bennett, Chief Chemist, Los Angeles Soap Co., Los Angeles; V. K. Cassady, Chief Chemist, The Palmolive Co., Milwaukee; Curtis & Tompkins, San Francisco; M. R. Dickson, Chief Chemist, Colgate - Palmolive - Peet Co., Berkeley; M. M. Durkee, The A. E. Staley Mfg. Co., Decatur; F. E. Joyce, Haskins Bros. & Co., Omaha; A. J. Harvey, Technical Director, Lever Bros., Ltd., Toronto; John Ornfelt, LaFrance Mfg. Co., Philadelphia; Foster D. Snell, Brooklyn; W. J. Reese, Chief Chemist, Colgate - Palmolive - Peet Co., Kansas City; Wm. A. Peterson, Chief Chemist, Kirkman & Son, Brooklyn, Secretary, Soap Section, A. O. C. S.; A. K. Church, Chief Chemist, Lever Brothers Co., Cambridge, Chairman, Soap Section, A. O. C. S.

The Institute for Commercial Expansion of the Ministry of Agriculture, Industry and Commerce of Brazil has issued a very complete discussion entitled "The Babassu Nut," published in Portuguese and English, which gives extensive information on the occurrence in Brazil of the Babassu palm, the products which may be developed from the palm and the conditions surrounding its exploitation. The booklet is well prepared and attractively printed. It is profusely illustrated with half-tones and contains maps of the babassu-producing areas, together with many beautifully designed colored charts setting forth exports of babassu products in recent years and imports of various commodities which may be affected by the development of the babassu industry.

Referee Applicant

Mr. Clinton Morris, of Morris-Flinn Company, Macon, Georgia, has applied to the American Oil Chemists' Society for Referee Chemist certification on all products covered by the Rules of National Cottonseed Products Association. (*First Publication*)