Olive Oil Analytical Method

PART II

The Use of the Ultraviolet Ray in the Detection of Refined in Virgin Olive Oil

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THE first report[1] has emphasized the present need for a means of conclusively distinguishing between virgin and refined olive oils and mixtures of the two. It was also pointed out that although this problem has been successfully attacked in Europe by the use of the ultraviolet, with the exception of a short investigation at the Bureau of Standards[2], nothing has been done in this country to assist in its solution.

The main purpose of this investigation was, therefore, to confirm the work which has been conducted in various European laboratories[3] in connection with the use of the ultraviolet lamp in detecting "refined" in "virgin" olive oil.

The experimental procedure consisted in observing the oils under the light from a quartz-mercury lamp in conjunction with a Corning ultraviolet filter.

The mercury lamp was completely enclosed in a wooden box, the entire inner surface of which was stained a dull black. The radiation was obtained horizontally by fitting the Corning filter into one side of this box—care being taken to eliminate stray radiations. All this work was conducted in a "dark room" so as to facilitate the observations.

The samples of oils were placed in Pyrex glass test-tubes of 15 mm. diameter. These tubes were suspended vertically about eight inches from the Corning filter, and the characteristic fluorescence of each oil was observed against a white background. A white background was employed thruout because it was found to be far superior to any other in detecting very slight differences in fluorescence. (It is understood that by fluorescence we refer to the light reflected from the sample, and not that transmitted.)

The olive oils examined were obtained from every point of production in the Mediterranean districts which constitute the field of production of fully ninety-five percent of the world's olive oil crop. Because of the facilities extended to us through our buying organization, we were able to absolutely authenticate the qualities and treatments of the oils obtained. The virgin olive oils were obtained in most instances directly from the subterranean tanks of the co-operative farm presses, and these samples were checked with original shipments arriving at Baltimore. The samples of refined olive oils were obtained from practically every large edible oil refinery in southern Europe, and we have every proof that the "French refined" and "pulp refined" samples were accurate.

By "French refined" we refer to the ordinary methods of refining and deodorizing treatments given the third and fourth pressings of olive oils which have not been solvent extracted. By "pulp refined" we refer to the oil which has been extracted with the use of carbon disulphide from the "marc" or hulls of the olives after the usual steam extraction, and subsequently refined and deodorized in the ordinary manner.

It was observed that in some instances of fluorescence the oils were opalescent in appearance under the ultraviolet; otherwise, they were perfectly clear.

"Virgin" Oil Results

The fluorescences observed in the examination of virgin olive oils are shown in the following table:

Type Oil	Origin	Age	Fluorescence
Aragon	Spanish	8 months	Deep orange
Bari	Italian	10 "	" "
Sousse	Tunisian	7 "	Canary yellow
Sfax	Tunisian	7 "	" "
Infigiable	Tunisian	7 "	" "
Bari	Italian	7 "	" "
Andria	Italian	2 "	Deep yellow
French Blend	—	19 "	Yellow (oil opalescent)
Virgin*	Unknown	14 years (Approx.)	Yellow (oil opalescent)
Puglia	Italian	1½ months	Canary yellow
Basilicata	Italian	1½ "	" "
Bitonto	Italian	1½ "	Yellow (oil opalescent)

* Authentic *virgin*, packed about 1914; rancid; free fatty acid 3.74%.

Each virgin oil upon analysis in the laboratory conformed to the physical and chemical requirements for a pure olive oil.

The fluorescence exhibited by a refined olive oil is characteristically bluish-violet; this color

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is the same irrespective of source, and this fluorescence is absolutely incomparable with that of any virgin olive oil.

It was found very difficult to approximate the percentage of refined oil in virgin olive oil in mixtures containing more than 65% of the former, as the bluish-violet fluorescence was observed to have reached a maximum intensity at this concentration of refined oil. However, Stratta and Mangini[4] report the utilization of the spectrophotometer to detect the presence of virgin oil to 1% in mixtures with refined oil. This is possible from an examination of the fluorescence spectrum which, when virgin oil is present, is characterized by a red band of 669 millimicrons.

On the other hand, it was found that the presence of a refined oil could be easily detected in mixtures down to 5%. It was difficult, however, to clearly distinguish even a slight bluish-violet tinge in the fluorescences exhibited by mixtures containing under 5% of refined oil. This observation was repeatedly verified and was facilitated by comparison of the mixture in question with the virgin oil contained therein. We attribute this difficulty to the intensity of the fluorescence from the virgin oil which obscures that of the refined oil.

Stratta and Mangine[4], however, report that with their method of analysis, they can detect a change in the fluorescence of virgin oil upon the addition of only 1% refined oil.

"Pulp refined" olive oil was identical in color under the lamp with ordinary refined oil.

We feel justified in offering as an explanation of the fluorescences observed of the different types of untreated and treated oils that the distinguishing feature is a direct function of the chlorophyll content. We base this assumption upon the following facts:

Other Oils Examined

Authentic samples of crude cottonseed oil were examined under the lamp. Crude, unfiltered cottonseed oil showed no fluorescence and appeared very dark brown in color; crude cottonseed oil filtered thru paper (unbleached) possessed a deep yellow fluorescence exactly comparable with a virgin olive oil; crude cottonseed oil filtered thru earth (bleached) showed the same fluorescence as a refined olive oil.

We have found that all available refined vegetable oils, including cottonseed, peanut, and sesame oils, cannot be distinguished under the lamp from a refined olive oil. It is understood, of course, that all refining treatments include the use of a bleaching agent and subsequent deodorization, which necessarily modifies the original chlorophyll content.

A sample of virgin olive oil which originally showed a canary yellow fluorescence was heated to 300°C., maintained at this temperature for thirty minutes and then allowed to cool to room temperature. This oil in ordinary daylight had darkened considerably in color and under the lamp it took on a fluorescence of a 5-10% mixture of refined in virgin olive oil.

Numerous samples of second pressing natural olive oils showed under the lamp a deep reddish-orange fluorescence not comparable with any fluorescence noted above. The oils obtained from second pressure have naturally a higher chlorophyll content, as is evidenced by the fact that they are of a much deeper hue after this extraction than those of the first pressure.

The California virgin olive oils show a fluorescence which is very similar to that of the second pressure oils of European origin. We infer from this that the methods of extraction of the California oils have progressed to the point where a much greater percentage of the oil may be extracted from the fruit through first pressing than can be obtained in Europe. Therefore, on this basis we can account for the difference between the fluorescence of a European virgin oil and that of a California virgin oil. This is presumably the main issue that has prevented the continuation of the short investigation which was conducted at the United States Bureau of Standards[2].

We have found the ultraviolet lamp of value in examining our shipments of olive oil as to purity, and we are certain that if other manufacturers and importers will substantiate our results, they, too, will benefit.

Summary

1. The ultraviolet lamp has proven successful in the detection of refined in virgin olive oil down to a limit of 5% of the former. The results, however, are doubtful in mixtures containing under 5% of refined oil.

2. It is our belief that the chlorophyll content determines the characteristic fluorescence of an oil, and that the bluish-violet fluorescence of a refined oil is due to a change in the chlorophyll constitutents brought about by the refining process.

BIBLIOGRAPHY:

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[4] R. Stratta and A. Mangini: "Sulla Fluorescenza degli olii d'oliva italiani alla luce di Wood". Giornale di Chim. Ind. ed Appl. 10, 205-7, 1928.