Determination of Refractive Indices of Oils¹

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The usual methods for the determination of the refractive index depend upon the bending of a beam of light on passing through a portion of the substance having flat surfaces. The angle of total reflection is measured. This is illustrated by the Abbé refractometer and the immersion refractometer. Other methods depend upon the optical effects produced when light passes through portions of the substance having curved surfaces. This is the principle involved in methods for obtaining refractive index by means of the microscope.

The method to be described is based upon this latter principle and may be called a "refractoscopic" method to distinguish it from methods using the refractometer, since the observed optical effect is not measured.

It requires more oil than the Abbé refractometer, but has two distinct advantages over the latter:

1—It is cheaper. The Abbé refractometer costs about \$300 and therefore cannot be purchased by every laboratory. The cost of the method to be described is negligible.

2—The oil may be totally recovered.

We are all familiar with the fact that if we look at an object through a transparent medium there is no alteration in the size of the object, provided the medium has flat, parallel sides. If we look through a convex lens of glass, the object is magnified if we are within the focal length. Similarly, a concave lens makes the image smaller than the object.

If the substance of which the lens is composed has a smaller refractive index than the surrounding medium, the phenomenon will be reversed. Thus, for any given shape of lens, its ability to magnify or reduce depends upon its refractive index with respect to the surrounding medium. A convex lens of crown glass immersed in carbon bisulfide would not magnify, but would give an image smaller than the object.

The same principle may be applied to oils. If a spherical bulb of oil is immersed in a medium of another oil it will magnify or reduce, depending on whether it has a greater or smaller refractive index than the medium. If it has the same refractive index there will be no effect (disregarding the small effect of the glass in the bulb).

The formula for the focal length of a lens is:

$$\frac{1}{f} = (n - 1) \left(\frac{1}{R} - \frac{1}{R'} \right)$$

where f = the focal length,
 n = the refractive index with respect to the medium,
 R and R' = the radii of curvature of the lens.

Since the two radii are equal to each other and equal to one-half the diameter of the bulb, the formula which applies here is:

$$\frac{1}{f} = \frac{4(n - 1)}{D}$$

where D = the diameter of the bulb.

Hence, with a given bulb the focal length of the

lens produced would be inversely proportional to $n - 1$. The value of the refractive index is given by:

$$n = 1 + \frac{D}{4f}$$

It would be possible to obtain the focal length of the bulb of oil immersed in another oil, but this would be a difficult method for determining the refractive index.

A more practicable method is to observe qualitatively whether the lens is magnifying or reducing, by comparing the height of a distant building with the image produced by looking through the bulb of oil immersed in another oil. The effect is more pronounced when the bulb is held at arm's length away from the eye, but the size of the bulb makes it difficult to judge the size of the image, hence another more delicate method is desirable.

In this method the bulb is raised up and down while looking through it at a distant object. If the image rises as the bulb is raised and sinks as the bulb is lowered, the refractive index of the oil is *less* than that of the medium. This is represented in the bottom line of Fig. 3. The bulb is acting as a concave lens in air (shown on the right). Likewise, if the bulb is filled with an oil having a *greater* refractive index than the medium, the effect is produced which is represented in the top line of Fig. 3. On raising the bulb the image goes down, and on lowering the bulb the image rises. The bulb is acting as a convex lens in air.

APPLICATION OF METHOD

The method of making the bulbs is shown in Fig. 1. It is essential that they be as thin as possible. The bulbs are filled with oil by sucking through one stem. The lower stem is sealed off and the top one bent into a hook (Fig. 2) without sealing it, thus leaving the oil at atmospheric temperature.

At first, square receptacles, made by cutting the tops off square bottles, were used to contain the oil which acted as a medium. It was later found that test tubes have many advantages over these in that they are more convenient to handle, have no irregu-

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larities in the glass, may be stoppered readily, require less oil and, being thinner, may be more easily seen through.

The bulb is suspended in the top of the test tube, as shown in Fig. 2. The tube is held in the hand at arm's length, in such a position that some distinct horizontal line in the distance may be seen through the bulb. This may be, for instance, the horizon or the border line between a grass lawn and some distant buildings. The tube is tipped forward or backward slightly until it is vertical, as is shown by the fact that the line as seen through the center of the bulb appears straight. Then by slowly raising and lowering the bulb about an eighth of an inch it may be readily observed whether the oil in the bulb has a greater or lower refractive index than the surrounding medium, according as it shows the phenomenon represented in the upper or the lower line in Fig. 3.

The refractive index of an unknown oil in the bulb may be obtained by comparing it with a series of known oils contained in test tubes. A series of such oils was prepared and arranged in order so that with a bulb of an unknown oil it was a simple matter to obtain the refractive index.

These, together with a solution of glycerol (1.4545) and a sample of toluene (1.496), constitute a series with which the refractive index of oils may be determined with an accuracy greater than the normal variation between different specimens of the same oil.

This method was proved to be accurate to 0.0005.

The exact values for these oils were obtained on the Abbé refractometer, and were as follows:

Sperm.....	1.4655
Olive.....	1.4703
Olive-cottonseed mixture.....	1.4713
Cottonseed.....	1.4735
Corn.....	1.4758
Rape-seed.....	1.4778
Castor.....	1.4796
Linseed.....	1.4820

(Corrected to 15° C.)

The Abbé refractometer is said to be accurate to 0.0002, but does not always check up as closely as that. The lard oil and olive oil with which the author was working were very close to each other in refractive index. The lard oil is usually listed below olive oil, but was shown to be higher by the above-described method. Values obtained on the Abbé refractometer showed this to be correct, the difference being 0.0005.

There is one difficulty in the use of this method which should be mentioned. The glass of which the bulbs are made produces a slight effect similar to that of a concave lens in air. So for a bulb filled with an oil and immersed in a tube of the same oil there is a slight effect, as shown in the middle line of Fig. 3. With a little practice one can tell how much of the effect is due to the oil and how much is due to the glass in the bulb. This effect is reduced to a minimum by the use of bulbs with extremely thin walls blown as shown in Fig. 1, which is drawn a little smaller than natural size. The bulbs should have a diameter between three-eighths and one-half inch.

Microanalytical Methods in Oil Analysis¹

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Although much work has been done on perfecting the methods for identifying oils, little attention has been paid to reducing the quantity required for analysis. Occasionally, as in extracting oils from leather, the oil chemist is called upon to identify a quantity of oil so small in amount as to handicap him in obtaining accurate results. The purpose of this paper is to show that an accurate proximate analysis may be made upon an oil when only a few drops are available, and with an accuracy comparable to that of the usual methods.

For the present work attention has been focused on four oils, selected because of their widely differing properties. These were olive, lard, cottonseed, and raw linseed. It is safe to assume that these oils represent in their properties all classes of saponifiable oils. Any adaptation of the general tests which would apply to them would apply equally well to others.

The tests to which most attention was given were the iodine number, saponification value, and specific gravity.

APPARATUS

The apparatus used in obtaining the iodine numbers and saponification values is shown in Figs. 1 to 7.

Fig. 1 represents the ordinary titration apparatus on a small scale. The bottle was a liter bottle and

the buret was an ordinary buret-pipet of 10-cc. capacity. For this purpose one 30 cm. long was selected. A ball or bead valve was used. It was of course necessary that the drops falling from the nozzle tip be as small as possible. To this end the tip was so drawn

out that the outlet was on the side of the tip about half way down, the lower half being a fine glass rod down which the solution would run and fall off in fine drops. (The same effect may be produced with a finely pointed tube smeared with a layer of grease.) Apparatus of this description was used for the sodium thiosulfate in the iodine number determinations

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