THE REFRACTOMETER AS AN AID TO THE RAPID ANALYSIS OF OIL BEARING MATERIALS

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Industrial enterprises are constantly in search of methods of analysis which will shorten their routine laboratory work in such a manner as will give more efficient plant control.

Typical of such rapid methods now in use are those of the steel chemist for determining carbon, phosphorous, etc., of the sugar chemist for ascertaining the sugar content of syrups and of the cereal chemists for denoting the percentage of moisture in cereal grains.

There can be no question but that a test of a similar character would be of great assistance to the vegetable oil industry, as the buyer of oil bearing seeds would be then in a position to know within a short time the composition of his raw materials, and in addition the plant could be more efficiently operated as frequent tests could be made at short intervals to determine just what the presses were doing in so far as leaving oil in the cake was concerned.

Early attempts have been made by different vegetable oil chemists at one time or another to simplify the ether extraction method for making accurate oil determinations or to develop some rapid test which while not so reliable, as far as extreme accuracy is concerned, would give results sufficiently accurate for routine use in testing raw and finished products. As a practical matter, however, only one of the proposed short methods has proven worth while for the reason that the time element was not sufficiently reduced, that the expense of making the test was too great, that the accuracy, as compared with the standard methods was not sufficient, or that the method was not simple enough for the average analyst to carry out.

Of all the proposed methods, Wesson's[1] observation that he could measure the quantity of oil in cotton seed meal and meats by noting the change in the refractive index of a solvent known as Halowax (which chemically speaking, is a substituted napthalene) as it became diluted with cotton seed oil extracted from the sample of cotton seed under the conditions of the test, appeared to be the most worth while for consideration as a general method of study for determining the oil content of many of the oil bearing substances. For this reason, the method has been the object of study for some time at the Research Laboratories of the Grain Division of the Bureau of Agricultural Economics, United States Department of Agriculture. Among the commodities to which the method has been applied are linseed (flaxseed), and linseed meal, soybeans, mustard seed, peanuts, cocoa beans, and cocoa products.

The research relative to the use of the Wesson method in relation to determining the oil content of linseed (flaxseed), as well as the application of the method to the determination of oil in linseed meal has been very fully described in United States Department of Agriculture Mimeographed Circular G. I. 33, entitled: *A Simple Test for Determining the Oil Content of Flaxseed and Linseed Meal.* Suffice it to say as a result of this study, that with linseed and linseed meal the method worked ideally. Of the 120 samples of flaxseed analyzed 48.7 per cent of the samples tested by the optical method varied less than one-tenth of a per cent from the ether extraction method, 37.8 per cent varied greater than one-tenth of a per cent and less than .19 of a per cent, 14.3 per cent varied by greater than two-tenths and less than twenty-nine hundredths of a per cent, whereas only 4.2 per cent varied by more than three-tenths of a per cent, the greatest variation with all the samples tested being only .33 of one per cent. The samples tested varied in oil content from 29.86 per cent to 43.56 per cent. Similar success as attanied from the analysis of a large number of linseed meal samples.

Considerable progress has been likewise made towards developing the method for peanuts, cocoa beans, and cocoa products and with each of these commodities, on account of their high oil content, the question of securing a representative sample seems to be one of the points on which the adaptation of the optical method to their oil analysis depends. Among other materials on which the method is to be tried are corn germ meal and fish meal, ~~crackling~~, mineral oil in parawax, and oiled paper.

Special Points for Consideration in Making the Test

In making the test several points of importance arise which should be watched in order to secure good results.

It is quite essential that the sample to be tested be thoroughly ground and in such a state of subdivision that intimate contact with the solvent is assured.

It has been found necessary to grind flaxseed with a small sized flouring mill, due to the fact that an attrition or burr mill mashes the flaxseed rather than cuts it, and likewise spreads the oil over the surface of the plates.

As usually received cocoa needs no grinding, and the cocoa bean can be successfully ground on a burr mill.

Due to the fact that the refractive index as well as the specific gravity of different lots of the solvent (Halowax) will vary, it is necessary to prepare standard conversion tables with each new lot of solvent and commodity tested. These tables must be made from a study of samples of Halowax and the oil under test of known composition.

The refractometer readings must be corrected for temperature changes because each degree change over or above a certain specified point will change the refractometric readings by approximately .00045, which value in the case of flaxseed is equivalent to three-fourths of a per cent of oil.

The Halowax vegetable oil mixture should be separated from the ground material by means of a small folded filter paper rather than by the cotton pellet method as recommended by Wesson.[1]

Care should be given to the refractometer and frequent use of the test slab should be made to keep the refractometric scale always adjusted to the same position.

Equipment Necessary and Time Consumed

The minimum amount of equipment necessary for carrying out the test by the refractometer method is as follows:

> Suitable grinding equipment, a refractometer capable of being read to the fifth decimal place, one pipette, one analytical balance, several mortars and pestles, small funnels, test tubes, folded filter paper, and absorbent cotton.

On the average it takes about 15 minutes to make a simple determination by the optical method. This time, of course, can be reduced if the tests are made in volume. This is in distinction to the 24 hours necessary to extract the linseed oil from flaxseed to the 16 hours necessary to extract cocoa butter from the cocoa bean and to the 3-4 hours necessary for the extraction of cottonseed oil from cottonseed meal.

The cost of making the test is also slight. After the initial expense of purchasing the equipment necessary for completing the test, the expense of reagents and so forth should not be over 2 cents per test.

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[1] Wesson, David "New Optical Method for Determining Oil in Oil Mill Material. Cotton Oil Press," Vol. 4, No. 3, July, 1920.