The Lipeometer Fat Test

A New Rapid Method for Determining Fat and Oils in Fat Bearing Materials, Press Cake or Refuse

By ROBERT SCHWARZ*

OIL chemists and superintendents of oil pressing plants have for a long time felt the need for and the value of a method whereby it would be possible to rapidly and accurately determine the fat or oil content of their raw materials, press cakes and other waste or by-products. Their discussions have laid stress not only on the rapidity of such a desired method but also on the safety, and particularly the accuracy thereof. When large quantities of seeds are handled, small variations become very important.

The purpose of this short paper would not be served by presenting a detailed bibliography or discussion of the various methods which have been presented. It is, however, important to note that in 1908, Chas. H. Herty presented a method which, like our own, based the estimation of cotton seed oil in meal or cake on the drop in specific gravity of carbon tetrachloride when the same was used to dissolve the fat in a weighed sample. Herty used a Westphal balance, prepared detailed tables for the conversion of specific gravity to percentage of cotton seed oil and prescribed methods for standardizing new batches of carbon tetrachloride whose variations in gravity had always to be taken into consideration. This method required considerable manipulation and involved the use of a solvent which is quite volatile, offering the possibility of considerable error due to evaporation of solvent, particularly in warm climates.

Early in 1920 David Wesson proposed a new optical method for determining oil in oil mill materials. The physical property upon which the determination was based, is the refractive index, the solvent halowax, and the measuring instrument, a butyrorefractometer. The Wesson method was studied in detail in the Grain Research Laboratory of the Bureau of Agricultural Economics, U. S. Department of Agriculture, and the results of this research published by Coleman & Fellows, U. S. Department of Agriculture, Bulletin #1471, March 1927. This method unquestionably gives accurate results. It, however, involves considerable preparatory work, the use of a rather elaborate temperature regulating device to be attached to the refractometer, and the standardization of the solvent each time that a new batch is used. An analytical balance is required, and as the determination is made on a 2 gram sample, considerable skill in manipulation is required.

A number of laboratories have developed methods based on shaking out various quantities of material with volatile solvents, filtering off an aliquot into tared fat flasks, evaporating off the solvent and weighing the fat recovered. These methods require about one hour per determination, and from the point of view of practicability present the requirement of analytical balances and volatile, generally inflammable, solvents. These objections have mitigated against their wide adoption for plant work. These methods furthermore have not shown themselves sufficiently superior to that of the American Oil Chemists' Society to warrant their consideration as alternates for official procedure.

A study of the methods available readily disclosed that a rapid, simple and safe procedure could not be based upon an extraction and subsequent separation of the fat to be determined. A physical property of the oil to be determined had to be used. As the specific gravity of freshly pressed oils in general varies between fairly narrow limits, and as specific gravity spindles which are sufficiently delicate to be accurate to one unit in the fourth place can be obtained, there was sought a solvent of high specific gravity which was non-poisonous, non-inflammable and at the same time very active as regards dissolving fats from raw materials. The basis for such a solvent was found in ortho-dichlorbenzol, which, with its high boiling point of 179 Deg. C. and its specific gravity of over 1.30 is well suited for this pur-

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Presented before the Twenty-first Annual Meeting of The American Oil Chemists Society, New Orleans, La., May 9, 1930.

pose. Of course, commercial ortho-dichlorbenzol invariably contains some of the para-isomer and frequently small amounts of trichlorbenzol. It therefore became necessary to develop a new analytical solvent of definite composition and specific gravity. The solvent has an unusual affinity for practically all fats and oils. The change in gravity when different amounts of the various fats, particularly cotton seed oil, are added, presents a straight line graph.

In order to develop a method that would be practical for plant control, all delicate balances and fine measurements were eliminated. The first work was done with samples of 33⅓ grams and later the 100 gram sample was adopted. A Torsion balance with dash pot is amply sensitive for weighing out the sample. Using 100 grams of sample, 1% of oil becomes 1 gram of oil. Therefore, the minimum amount of solvent which could be used to float an average-sized specific gravity spindle or hydrometer was determined and a chart prepared by adding to this quantity of solvent from 1 to 13 grams of pure cotton seed oil. The specific gravities at 20° C. were carefully noted, checked and these gravities then used as the basis of a hydrometer whose scale readings are given in percentages of cotton seed oil instead of specific gravities. The inevitable factor of change in specific gravity with variation in temperature required automatic compensation in order to make this method simple and certain. The curves for variations in specific gravity due to changes in temperature above and below 20 deg. C. were carefully prepared and correction scale prepared for a thermometer in the body of the hydrometer which for simplicity and identification has been named the *Lipeometer*.

Early tests showed that when 100 grams of material are treated with about 600 grams of solvent and the mixture heated until the solvent fumes slightly, the mixture then cooled and after filtration through a Büchner Funnel, tested with the Lipeometer, the fat content found in most instances agreed closely with the results obtained by the A.O.C.S. method. When, however, the material under examination was rather finely ground, the results were found to be from 0.3 to 0.5% high. A further study showed that if the meats or meal were ground to from 60 to 80 mesh and the heating of the mixture and solvent and sample entirely omitted, the results agreed very closely with those of the official method used by the American Oil Chemists' Society. Table I gives results on 11 samples which were tested by both the Official and by the Lipeometer method.

TABLE I

SAMPLE	Method of A.O.C.S.	"Lipeometer" Rapid Method Ground to 60-80 Mesh
A	4.05 %	4.1 %
D	8.2 %	8.1 %
C-12	6.25 %	6.15%
C-16	6.02 %	6.1 %
55	5.981%	5.9 %
C-13	6.21 %	6.2 %
B	4.55 %	4.4 %
50	6.0 %	6.0 %
51	5.65 %	5.75%
C-14	6.25 %	5.95%
C-15	6.17 %	6.1 %

The maximum difference between A.O.C.S. and Lipeometer method is 0.3%, the minimum 0.00% and the mean error 0.09%. The Lipeometer tests were carried out on samples that had been previously ground through a Straub laboratory mill.

The final procedure developed is described briefly as follows: An aluminum beaker plus stirring rod of standardized weight, plus the sample are balanced against a tare weight, using the Torsion balance. This procedure automatically weighs out a 100 gram sample. The tare is changed for one which balances the beaker, the rod, the sample and the required quantity of solvent. As soon as solvent needed to balance the new tare has been added, the mixture is thoroughly stirred, allowed to stand 5 minutes, stirred again and then filtered through a Büchner funnel into a special filtering cylinder, using suction. The filtration requires but a few minutes. The filtrate is thoroughly mixed with an aluminum stirrer and the Lipeometer then inserted and after the solution has come to equilibrium, the reading is taken, using the upper meniscus. At times stratification of the solvent containing the dissolved oil requires that the Lipeometer be moved up and down in the solution several times before reading is taken. In the event that the temperature of the solvent, or particularly the room temperature is materially above 20 deg. C. the filtering cylinder is placed in a cooling jar and the temperature of the filtered solution reduced to about 20° C. by circulating cold water from a faucet. This requires but a few moments and does not materially add to the length of time required for the complete test, which, including the grinding of the sample, should not exceed 20 minutes. Where several samples are taken at a time, it is possible to have three and possibly four results within one-half hour.

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Cottonseed Samples

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The curved plate "F" (Fig. 1.) in the rear of the hopper beside preventing any back dropping of seed, apparently tends to cause a rolling of the seed in the hopper and possibly results in some mixing of the seed. As to size, the belts are ⅞ inch in width, the pulleys 3 inches in diameter and 7½ inches from center to center. The tacks protrude ⅛ inch through the belts. We operate the crank at a fairly constant rate of about 75 rpm. The apparatus is exceptionally simple and was constructed in this office by one of my assistants, Mr. F. S. Hubbard, from scrap material with the use of hack saw, a metal drill, pair of tin snips and a soldering iron.

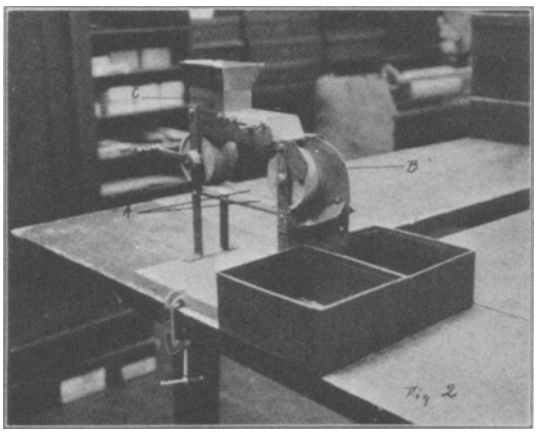

Fig. 2. COTTON SEED SAMPLE REDUCER.

Side view—showing machine in position for operation.

The machine is peculiarly adapted to the division of samples of cotton seed sent in under the revised rules of the National Cottonseed Products Association. These samples under the new rules are to weigh 1,000 grams. One-half or 500 grams is to be retained as a referee sample, a single division on the machine. Again dividing one half of the original sample gives two parts of 250 grams, a second division of one of these portions results in two parts of 125 grams each. One of these is available for Free Fatty Acid determinations and dividing the other results in two portions of approximately the size required under the rules for moisture and oil determinations.

The Lipeometer

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The method as presented, has been thoroughly tested not only on a number of samples but on various portions of the same gross sample. The results are within 0.1 of 1% in the hands of various analysts. It is perhaps needless to point out that the simplicity of the manipulation permits the work to be done by a trained technician.

Acknowledgment is hereby made to Dr. C. P. Harris, of our Research Laboratory, for his work in the development of the process, and to his assistant, Mr. Manuel Horwitz, for his co-operation.

Fat Composition and Uses

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China wood (tung oil) trees, rubber seed, perilla and some other plants are being systematically tested. Yet, bearing in mind the wide climatic and agricultural resources of the Empire and the opportunity for opening up fresh tracts of cultivated land on these lines, progress within the Empire is all too slow, and the efforts already made by the Government Departments concerned to promote these developments deserve encouragement, but also require to be intensified and fortified by the close co-operation of chemists, biologists and agricultural experts.

Tentative Official Oil Trier

THE Tentative Official Oil Trier (sampler) adopted at the meeting in New Orleans this year can be obtained complete from the Refinery Supply Company, Tulsa, Okla. at the price of $35.00.

The committee suggests that all companies who have any use for oil sampling devices, purchase at least one of these Tentative Official Triers in order to enable us to collect data and opinions during the coming season. At the next meeting of the Society, this matter will again come up for definite adoptions or recommendations.

We very much appreciate your cooperation in connection with the above and wish to refer you to the Sampling Committee's report printed in the May issue of *"Oil and Fat Industries,"* also the report of Uniform Methods Committee, *"Oil and Fat Industries,"* June, 1930. Further details concerning the sampler may be obtained from the chairman.

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