The Rapid Determination of Total Fat

Variation in Specific Gravity of a Solvent Made Basis of Analysis of Seeds or Press Cake for Oil Content†

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THE importance of the determination of total fat in fat- or oil-bearing materials has long been recognized by the converters of these products into marketable materials. The residues remaining from the expression or extraction of oils and fats are almost invariably of lower value than the oil which is usually the prime product. The value of the oil-bearing raw product is, of course, dependent upon its oil content, and this may be determined within a reasonable period without causing delay or loss to the vegetable oil producer. The amount remaining in the residue (press cake) indicates the efficiency of the expression process and tells the operator whether any of the product is being needlessly lost. It is obvious, however, that the efficiency of a pressing must be known as quickly as possible in order to prevent avoidable loss. Many methods have been devised for the estimation of "total fat." Some are very slow, a few are quite rapid, but all are subject to the objection that a skilled chemist is required to obtain significant results. It, therefore, was suggested that methods which were rapid, accurate, and above all, sufficiently simple that non-technical persons could operate them, would meet with welcome by oil and fat producers.

Such methods have been devised. The time required for their operation varies from ten minutes with cottonseed meal to thirty-five minutes with the more finely ground chocolate products. The accuracy is at least as great as that claimed for the Official methods and the simplicity is such that a laboratory boy or factory girl can become adept after a few hours practice. Before describing one of the new methods it might, perhaps, be of interest to recall some of the older methods for this determination. The most widely used method for chocolate products, an Official method, consists in mixing the sample with an equal weight of sand, transferring to an extraction thimble, and extracting with ether (petroleum ether is preferred to ethyl, as the latter extracts substances other than fats) into a weighed extraction flask for five hours, distilling off the ether, drying the extract first in an oven, then in a desiccator, and weighing the fat. This procedure in one form or other has been, and still is the standard procedure. With only two exceptions the idea has always been to *isolate* the actual fat present and weigh it. Welman's[1] variation consists in suspending the material in water before the ether extraction. Hanus[2] makes use of a dilute alcoholic and ammoniacal suspension before the ether extraction. He prefers a mixture of the petroleum fraction and the ethyl ether. Kreutz[3] melts the sample with chloral hydrate to facilitate extraction. The most exact results are supposed to be given by Heller's[4] method, which consists of a twenty-four hour extraction in a Soxhlet apparatus.

Gephardt speeded up these extraction methods, finding that good results are obtained by the use of only 1 gram of sample and by a very short extraction and washing of the material on a Gooch crucible. The method requires a skilled operator. Gerber and Kooper[5] modified the Babcock method for milk. Salts of hydroxybenzoic (salicylic) acid are used to free the fat from extraneous material, and butanol is used as a "clarifying agent." This method seems to give excellent results with milk, cream and other dairy products, but, possibly because the proteins are different in chemical nature from the casein of milk, thus having an altered solubility in the salicylates, the method seems inapplicable to chocolate products. At any rate the results are neither consistent nor reliable with this sort of material. A method devised by Hughes[6] is popular in some circles because of its rapidity. It consists of successive extractions with ether of the fat-bearing substance, centrifuging the ether layer after each extraction. Low results are obtained if the extraction has not been complete, and, on the other hand, high results are likely if the centrifuging of the

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finely divided particles is less than perfect. A variation of the extraction method consists of the S. B. Phillips[6a] procedure, using trichlorethylene. A larger sample (8-10 gm) is taken and an aliquot of the extract is evaporated and weighed in the usual manner. The method is fairly rapid, but the technique is involved. Lepper and Waterman have recently modified the Official method described above and use a Knorr extraction apparatus. The sample is introduced into the Knorr tube, stirred with petroleum ether, and washed ten times. The method has been made official and appears to be more rapid than the earlier official procedures. Heiduschka and Muth[7] have devised glass apparatus for extraction and evaporation methods which shorten the number of manipulations and transfers of solution. The Gooch crucible is designed to serve also as a weighing flask for the sample, and the distilling flask is also fitted with covers and is sufficiently small to act as a weighing flask for the extracted fat.

Richter[8] and Herty[9] broke with tradition so far as total fat estimation is concerned and devised methods which do not depend upon weighing the fat actually present in the sample. They recognized that solution of a second substance in a liquid alters the physical properties of the liquid, and usually in direct proportion to the amount of substance dissolved. Therefore, if the change in that property can be sufficiently accurately measured, the amount of added substance can be determined. Richter's method depends upon the change in refractive index, Herty's upon the change of specific gravity. Richter used as dissolving liquid a mixture of ether, alcohol and trisodium phosphate. The substance whose fat content was to be determined was extracted with this mixture, the clear solution, obtained after warming, adjusted exactly to a definite temperature (17.5°C.) and examined under a Zeiss refractometer. From the refractive indices of solvent, solution and the pure fat, the amount of fat present in solution and, consequently, the amount present in the original sample may be calculated. One difficulty with this method lay in the very great volatility of the ether and alcohol of the solvent. Evaporation of these substances during the operation will render this method inaccurate. To overcome this objection, Wesson[10] proposed the use of "Halowax" a trade name for chlorinated naphthalenes. As certain of these products, impure monochlor compounds, are liquids, they can be used instead of the original mixture of Richter. Halowax is non-volatile and otherwise suitable for this method, but the commerical product varies in composition and properties. The refractometer method has been further elaborated by Coleman and Fellows[11] to include a number of fat-bearing materials. Before using this method the Halowax must be standardized, i.e., a table showing the variation of refractive index with increasing percentages of the oil or fat must first be developed before the particular batch of solvent may be used. According to Coleman and Fellows "It is necessary to prepare for each new lot of the solvent a standard conversion table for each kind of oil-bearing material on which the test is to be made." Furthermore, for certain materials, such as chocolate liquor, an involved process of preparation is necessary. This substance must be grated, melted, chilled, and grated twice before the analysis can be run. The temperature of the solution must be carefully adjusted before the refractometer reading is taken. However, when all these precautions are taken, a skilled operator can obtain results in very short intervals.

Herty's[9] method consists of the solution of the fat by means of carbon tetrachloride, and the determination of the fat by the use of a Westphal balance on the resultant clear solution. Due to its volatility, this solvent will inevitably cause great difficulty, as evaporation of the solvent during the operation will give high results. Here again, the specific gravity of the solvent will vary over an appreciable range, and in contact with various materials will hydrolyze and form various degradation products which very much alter the gravity. Consequently the solvent requires frequent checking and standardization and the operation requires considerable skill to avoid a change in specific gravity during the testing interval. The Westphal balance is not without objection.

REFERENCES

1. Welman, *Zeitschr. Offentl. Chemie 1900,* p. 304.
2. J. Hanus, *Zeit. Unters. Nahr. Gen. 1906* 11 p. 738.
3. A. Kreutz, *Zeit. Unters. Nahr. Gen. 1908* 15 p 680; *16,* p. 584.
4. Heller *Apoth.-Zeit.* 1916 *31,* p 330.
5. W. D. Kooper, *Zeit. Unters. Nahr. Gen.* 1915 *30,* p. 461.
6. Hughes, *Chemical News* Aug. 1919, CXIX, 1041.

6a. S. B. Phillips, *Analyst* 1916, p. 122.

7. A. Heiduschka and F. Muth, *Chemiker-Zeitung* 1928, No. 90.
8. Richter, *Zeit. Unters. Nahr. Gen.* 1912, *24,* p. 312.
9. Herty et. al. *J. Ind. Eng. Chem.* Vol. 1, 1908.
10. David Wesson, New Optical Method for Determining Oil in Oil-Mill Materials. *Cotton Oil Press* 4(3) : 70-73, 1920.
11. D. A. Coleman and H. C. Fellows, *U. S. Dept. Agriculture Tech. Bull.,* No 71, 1928.

(*To be continued*)