Oven Drying Method for the Determination of Moisture in Cottonseed Meal

By PAUL SHERRICK

RECENT investigations by the Moisture Committee of the American Oil Chemists Society comparing the loss of weight and distillation methods for moisture in cottonseed meal have again brought the technique of this important determination under a variety of criticisms. The attitude of the Committee is indicated by its report published in OIL & FAT INDUSTRIES, June, 1926, as follows:

"Your committee knows of no other analytical process in which we are concerned that needs greater special attention than moisture determination, as shown by our moisture results on ch ck meal work and on our past year's work using the Bidwell-Sterling method; and these results also show conclusively that many of our laboratories are not equipped with a uniform and constant temperature oven."

Vacuum oven drying as the official or published referee's method, dates back to the Tentative Standard Methods published in September, 1916 by the Committee on the Analysis of Commercial Fats and Oils, Division of Industrial Chemists and Engineers of the American Chemical Society (*Journal of Industrial and Engineering Chemistry*, Vol. 9, No. 11). At that time an alternative routine procedure using air ovens was also outlined. In the report of the Committee's adoptions, as drawn up by W. D. Richardson, it is stated, "The Committee has also considered the various distillation methods for the determination of moisture in fats and oils, but . . . . it was decided that the most desirable method was the vacuum oven method as described."

That Committee also submitted the design for a so-called standard vacuum oven which it was hoped would be capable of the required degree of control. This oven, however, has not allowed a sufficiently ready maintenance and duplication of conditions as shown by the inevitable small but significant discrepancies between reports of different collaborators on test samples at various times. A number of other commercial vacuum ovens subsequently produced have led to no reduction of this working variable. At the same time the air drying ovens used by control laboratories following the alternative recommended method have shown very poor temperature control and uniformity and caused occasional gross errors in moisture reports covering raw materials in transit or storage.

Out of this general situation has arisen the present interest in applying to cottonseed meal the distillation method recommended by Bidwell and Sterling for a variety of raw materials. This principle has been employed to some extent for the last twenty years in methods applied to grain by Brown and Duvel, to crude fibre by Schwalbe, to leather by Rogers, and to petroleum products by Dean and Starck.

The present method as developed by Bidwell & Sterling has some theoretical superiorities over any loss of weight method as pointed

out by Mr. Bidwell (*Journal of Industrial & Engineering Chemistry*, Vol. 17, No. 2, namely, (1) it is free from effects of variable humidity; (2) possible losses in weight from slight oxidation of the sample are avoided; and (3) it proposes to measure actual water rather than water and volatile matter. The last contention is, of course, not strictly true, since the distillate in the Bidwell-Sterling method is liable to contain some volatile matter miscible with water which could introduce a positive error in the volume reading. If the quantity of volatile waste included in the results secured by the drying method were appreciably greater than the similar error by the distillation method, the former would be expected to yield a higher moisture figure. Actually, however, oven drying reports are consistently lower than distillation reports on the same samples by an amount in the vicinity of 0.3 per cent of dry sample weight. This almost constant difference must be attributed either to a more extensive inclusion of substances other than water by the distillation method or to the retention of some mechanically held moisture by a dried sample even after it has reached apparent constant weight under the specified conditions.

In either case, a major consideration in establishing the more practical method in the hands of laboratory chemists would seem to be a decision as to which permits the closest possible duplication of results when used by different operators in various laboratories. In this connection data published in the report of the Moisture Committee, this Journal for June, 1926, appears to be somewhat in favor of the oven method. During the year 1925, six samples of cottonseed meal were submitted to a large number of collaborators for moisture reports by the Bidwell-Sterling and the oven drying methods. Successive samples were sent to the same chemists with the result that any unaccustomed to the Bidwell-Sterling method should have become familiar with its manipulation by the time the later samples of the series were sent out. For the purpose of estimating the uniformity of results by either method in the hands of numerous operators, the most significant figure which can be secured from this collection of results is the mean deviation from the mean. In other words, having determined the average value for moisture percentage from collected reports on a given sample, each individual variation from this average is computed and the average of all these deviations is a figure indicating the probable duplication of results on a given sample by different operators. The accuracy of this figure is, of course, a function of the total number of determinations from which it is calculated. These mean deviations from the means as computed from the third, fourth, fifth, and sixth samples of the Committee's series are as follows:

		Mean deviation from the mean in % of dry weight.	
Sample	No. of reports	Bidwell-Sterling	Oven Drying
3	25	0.47	0.28
4	17	0.48	0.24
5	14	0.37	0.23
6	13	0.26	0.24

In comparing these figures it should be remembered, of course, that the Bidwell-Sterling method was comparatively new in the hands of some operators.

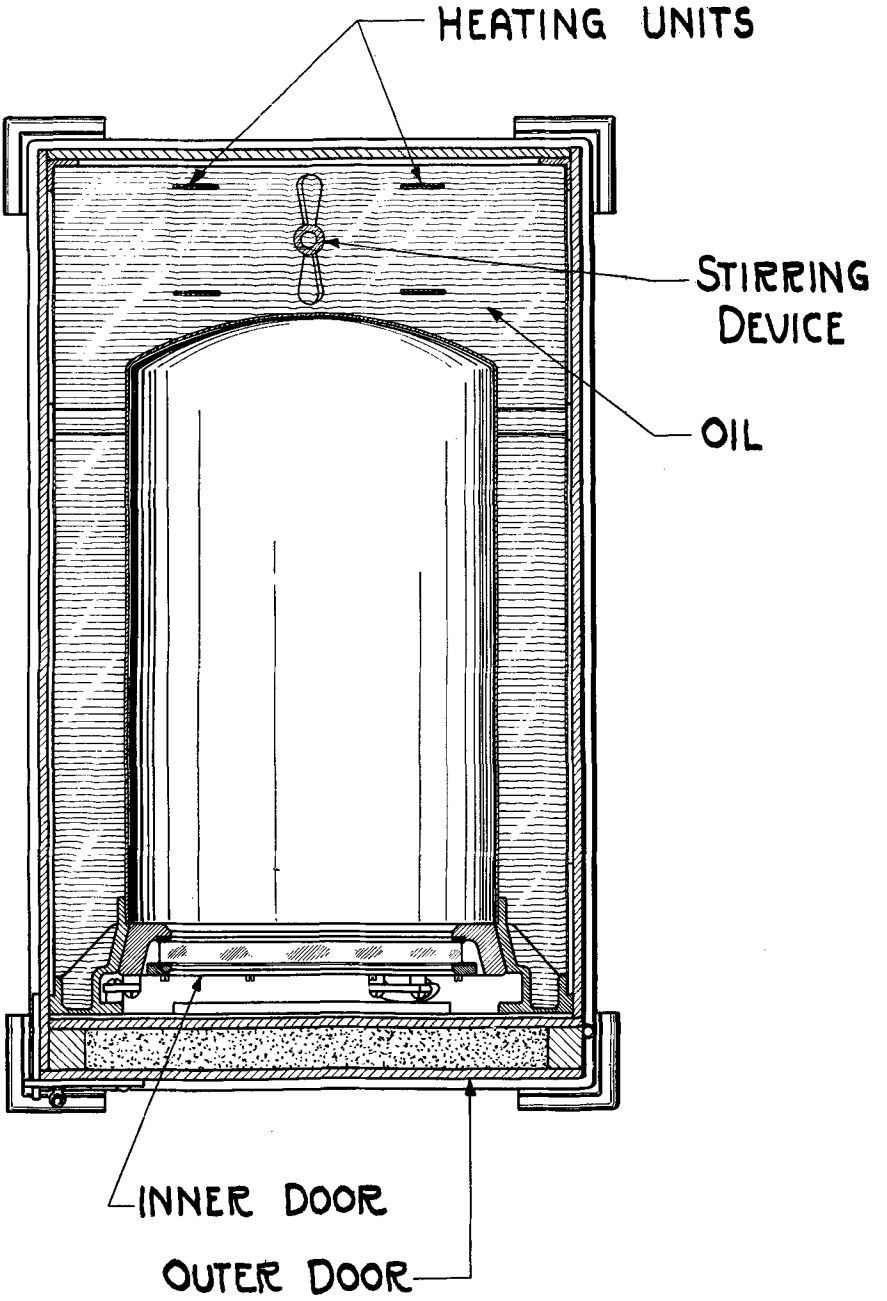
Variations in the oven method are accounted for to a large extent by the lack of temperature control

and uniformity which characterizes a large number of the air drying and vacuum ovens in use in control laboratories. Referee's results on moisture in flour indicate that temperature variations in oven drying begin to affect moisture results when such variations approach a magnitude of 5° C (*Journal of the Association of Official Agricultural Chemists*, Vol. 9, No. 4, page 404). Moisture results on cottonseed meal are probably affected to a similar extent. The actual magnitude of local temperature differences in many well-known and widely used air drying ovens which employ primarily a direct radiation system often reaches 30°C between the top and bottom shelves, or 15°C on one shelf and a similarly large variable is found in direct or air heated vacuum ovens. In the field of oven design, work of the past five or six years, carried on by the Central Scientific Company of Chicago, and directed upon the problems of heat control and distribution in closed chambers is of particular importance, inasmuch as it has produced an oven of each type, air drying and vacuum, whose working variations in temperature and pressure are well within the apparent significant limits for this determination. These are the DeKhotinsky air drying oven and the new Cenco vacuum oven.

Distribution of heat in the DeKhotinsky ovens is secured by properly balancing the effects of direct radiation and convection. These two elements are the important factors in the thermal characteristics of any oven. The former establishes a temperature gradient with its high at the bottom and its low at the top of the oven chamber. Convection currents tend to establish a reverse gradient with warmer air above being replaced by cool air below. By effectively increasing and distributing the convection factor, the DeKhotinsky oven balances these two gradients and at the same time maintains an equally effective lateral circulation. Exhaustive measurements of heat distribution have been carried out by loading the ovens with sample dishes of oil meal, each dish containing its own thermo-element. All these measurements show a probable maximum temperature difference between any two points in the oven of 3°C. It is highly improbable that this variation could ever appear in any moisture determinations reported in the usual way to 0.01 per cent of dry weight. Under these conditions routine tests may be conducted in large numbers simultaneously by loading the oven to a reasonable capacity, which is obviously one of the strong appeals of the air drying method to the control chemist.

Experience with a variety of former vacuum ovens has shown that the necessary temperature control and uniformity cannot be obtained by the direct application of intermittent heating to the chamber proper. Several manufacturers have used a thermostatic air bath as the heat transmitting medium, but this method, up to the present time, has not produced a reasonably close control in the drying chamber. The third logical plan has been employed with excellent results in the new Cenco vacuum oven, namely, immersion of the vacuum chamber in a circulating oil thermostat.

The oil bath itself is controlled to variations less than ± 0.5°C, by means of the well-known DeKhotinsky bi-metallic thermo-regulator, Bureau of Standards form Knife Type heaters, and motor driven stirrer. The drying chamber

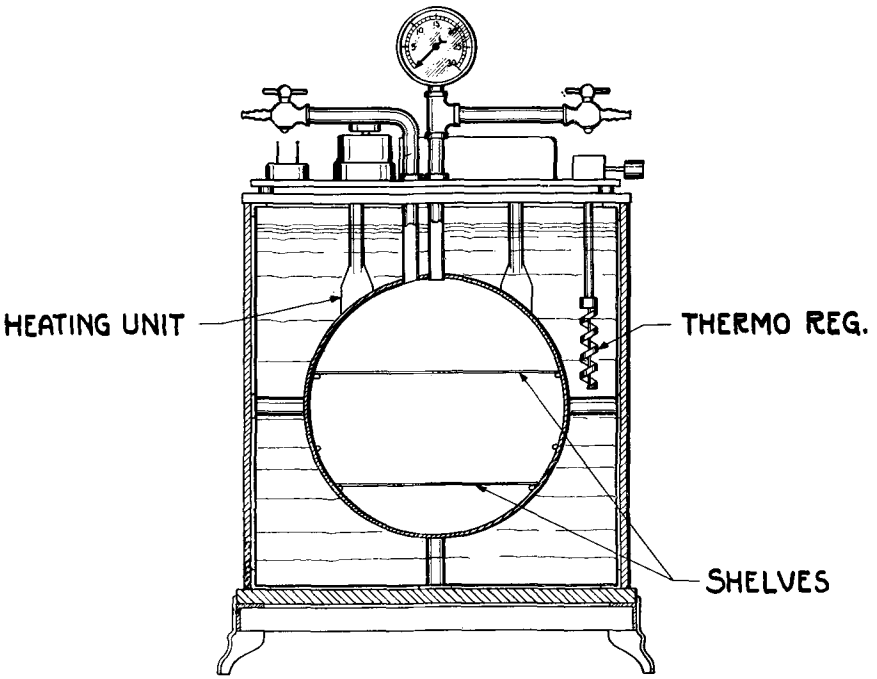


A Cross Section View of the Vacuum Oven

proper is cylindrical in form, 9″ in diameter, and 14″ long, allowing the use of three shelves. The door consists of a circular bronze casting with a pane of heavy Pyrex glass and fits into a tapered brass seat. An effective seal is achieved by properly grinding the door into its seat. The accompanying diagrams, illustrate the construction in detail.

A great number of exhaustive tests have been made on these vacuum ovens to establish the exact performance that may be expected from them. Uniformity of distribution has been repeatedly measured by placing sample dishes of sand at the extreme ends and sides of the three shelves, each containing its own thermo-element, and each compared with a central dish at the thermometer bulb as reference standard. In none of these tests has the average total difference between two widely separated samples been more than 1°C, and the largest total variation on record is 1.4°C. It is undoubtedly safe to assume a general working limit of uniformity to be ± 0.5°C. Figure 3 is a three-hour section from a characteristic test curve, showing simultaneous temperatures in the oil bath and in the oven chamber at its axis. With reasonably constant room temperature, the oven temperature, as read from the thermometer, seldom varies as much as 0.5°C. This curve was taken when the chamber was at a pressure of 3 mm. of mercury.

The use of ovens, either air or vacuum, for the determination of moisture in cottonseed meal has its greatest value in the convenient handling of a considerable number of samples with the least demand on the attention and time of the



Another Cross Section View of this Vacuum Oven

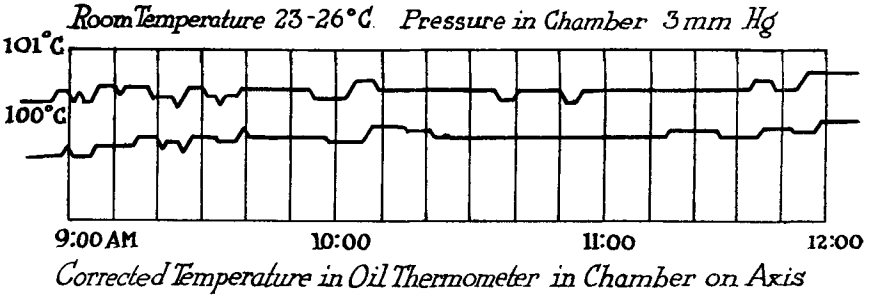


Fig. 3. A three-hour section from a characteristic test curve, showing simultaneous temperatures in the oil bath and in the oven chamber at its axis.

chemist. Wherever this method is employed it appears highly possible, by the use of ovens similar to those that were described above, to achieve a generally uniform measure of moisture in routine control in which probable variations among different laboratories should be reduced to a quantity of no commercial significance whatever.

Examination of Mixtures

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mixture of oils has been deduced a new mixture should be made having this composition, and the values obtained therefrom compared with those of the original. In this way a very near approximation to the truth may be obtained.

The detection of small amounts of butter in margarines, which is sometimes required, may be carried out by this method supplemented by the method of Gilmour (*Analyst*, 1920, 45, 2; 1925, 50, 272). Experiments along these lines have been carried out, and it is hoped to publish the results obtained.

It may not be out of place at this stage to remind workers that practically the whole of our knowledge of the examination of margarine is due to the pioneering work of Cribb and Richards and Bolton, and of Revis and Richmond. The present authors are glad to acknowledge their indebtedness to these workers, and to state that, at best, they can only claim to have confirmed and possibly somewhat extended the earlier work.

Alkali Absorption in Crude Oil Refining

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indicated by analysis. This would indicate that the globules of caustic solution do not come in such contact with the oil that all the caustic is taken up till after a long time of stirring. It also indicates the effect of excess caustic in producing excess saponification.

Adam Hilgar, Ltd., Publishes Bulletin

Adam Hilgar, Ltd. manufacturers of instruments for scientific research, have recently issued a new 32-page bulletin covering the work of their research and sales departments for the year ending December 31, 1926. The bulletin describes new instruments as well as changes and improvements in the standard Hilgar line.