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THE RAPID DETERMINATION OF MOISTURE

With Especial Reference to Oil Seeds and Their Products

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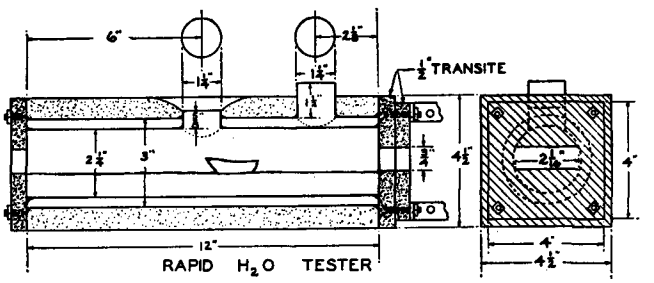
Abstract

The principles underlying the evaporation of moisture from oil bearing materials are discussed with especial reference to rapid testing. Two forms of rapid moisture testing apparatus employing relatively high temperatures and strong induced draft, are described. Data are given to show how the results compare with determinations made in a conventional oven at the usual drying temperature, as well as a tabulation comparing the results obtained by an untrained plant operator using the rapid tester with checks on the same samples made in the laboratory. The advantages inherent in rapid moisture testing from the control standpoint are discussed. Finally, the application of the apparatus as a controlled-heat hotplate is suggested.

THE American Oil Chemists' Society's concern in moisture testing, officially, is to have a method that will yield uniform results in different laboratories, and require only a reasonable time for the determination. It is also desirable that the results approximate as closely as possible the actual moisture content of the material tested. If our requirements in this latter respect should be too exacting; that is, should we insist that the method show absolute moisture content, then the question of uniformity of results would be completely satisfied, but at the sacrifice of the second requirement —reasonably short testing time. Absolute moisture content of organic materials like seed and seed meals may be indicated only by slow desiccation at low temperatures or moderate temperatures under vacuum. So for practical reasons we vote to content ourselves with empirical results and adopt testing conditions that minimize the loss of volatile matter not water and the decomposition of the dry material. When we admit a certain amount of empiricism into our results, as we so often must do, it becomes necessary to know what conditions cause any deviation of the results from the absolute and to so state the method and its alternates that uniformity of results is preserved, with particular respect to the allowance of alternate methods. Thus, in view of the effect on the rate of drying of the vapor pressure of the air over a material being dried, we are careful to consider maximum oven load and oven ventilation. Of especial importance in this connection is the forced draft principle. Through its employment the layer of air directly over the test material is continually removed, facilitating and accelerating the evaporation of moisture; so that at a given temperature a sample tested in a forced draft oven will lose its moisture in a shorter time than in a conventional type of oven. Moreover, on the reasonable assumption that decomposition of solid matter is slower than and lags behind the evaporation of moisture, we would expect that the shorter test for the same temperature would be more nearly in agreement with the true moisture content. In other words, the forced circulation has somewhat the effect of a moderate vacuum.

As to how much of the weight loss during drying is due to the loss of matter not water, we may form a qualitative idea from the shapes of the drying curves; and some of these curves for reasonably low temperatures indicate that while moisture is still present little or no loss of other matter occurs, but that after the material has completely dried, further weight loss begins and continues, the rate of loss being greater of course at more elevated



END PLATES OF BRASS (18 GAGE) AND ½ INCH TRANSITE HAVE OPENINGS CUT AS SHOWN. BRACKETS ARE BOLTED ON ONE END FOR FASTENING ON SIDE OF DESICCATOR BOX (SHOWN) WHICH IS TO BE MADE FROM ¼-INCH MASONITE-PRESSBOARD. BOTTOM TO BE GLUED ON AND TOP TO BE REMOVABLE ¼-INCH PLATE GLASS WHICH IS TO BE FLUSH WITH LARGER TRANSITE END BLOCK. 2¼-INCH AND 3 INCH (18 GAGE) BRASS TUBING ARE STOCK SIZES. FOR BRAZING USE SILFLOSS 1/16-INCH RODS AND FLUX.

FIGURE 1

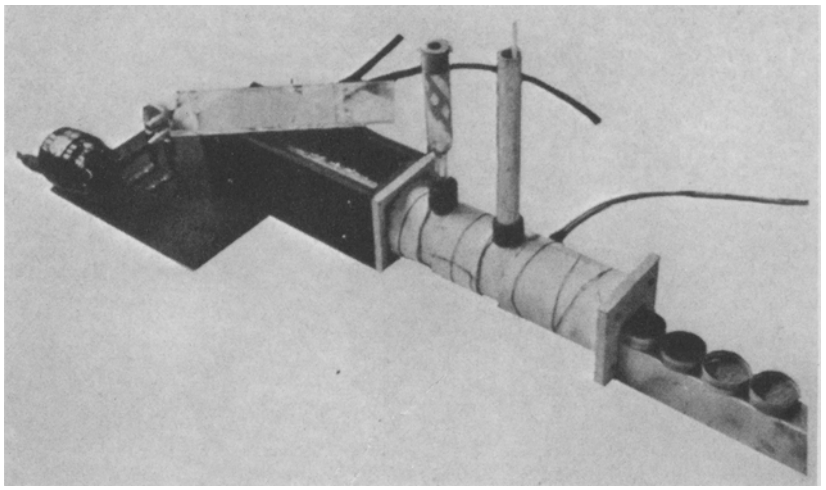


FIGURE 2

Fig. 2. Rapid moisture tester. High capacity, semi-automatic model. Method of use—non-automatic. Two dishes are shoved just inside and left for five minutes; then these two are shoved into the center of the oven by the next two tests, which also remain in the first position five minutes; finally, a third pair of dishes is pushed in behind the other four, for another five-minute period of time. Proceeding in this way, each test passes through the apparatus in fifteen minutes and occupies, relative to the center and ends, exactly the same positions as all other tests. The dishes remain in the water-cooled, glass-covered desiccator attached to the discharge end until ready for weighing. The motor-drive was designed to draw a charge of 6 tests through the apparatus at the required rate so that it should require no attention during the 30-minute period involved. The volume of work for which this was intended, however, was not sufficient to justify perfecting this convenience.

temperatures. In instances where this inflection of the drying curve does not occur we cannot determine the point at which the last moisture left. This is illustrated in Figure

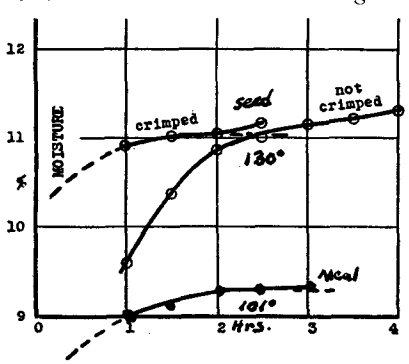


FIGURE IV.

4 by the curve for uncrimped cottonseed as compared with the crimped seed, in which latter there is a horizontal portion representing thirty minutes of almost no weight loss. This comparison shows that the condition favoring rapid drying (breaking the seed coats) permits a determination of the drying end-point, whereas at the same temperature the slower drying of whole seed evidently permits the beginning of decomposition of soild matter before drying has ceased, with the result that we do not know where true drying ends and decomposition begins. The curve for meal run at 101° C. in the forced draft oven is one of six on which the moisture contents indicated at two hours' drying differed from the official test indications (3 hours in jacketed oven at 101° C.) by — 0.02 + 0.03, + 0.01, — 0,02, — 0.07 and +0.18%, the values ranging from 6.5 to 9.6 per cent. The last difference was for the meal containing 6.50 per cent moisture the drying curve for which showed a slight flattening in the range 1½ to 2 hours, suggesting that drying was complete and that the 0.18 per cent difference represents decomposition of solid material in the case of the 3 hour official test.

RAPID TESTING

Advantage may be taken of the accelerating effect of strong air circulation in connection with higher than usual oven temperatures, in devising a rapid method that is free from most of the limitations and objections of the general run of rapid moisture testing methods—and one which is more accurate than most of them. The writer borrowed the principle for this tester from the Carter-Simon Apparatus, one of English manufacture and obtained with difficulty in this country. What the writer considers to be an improved form was described in *Industrial and Engineering Chemistry, Analytical Edition* 9, Page 434 (1937). This is shown in Figures 1 and 2. It is essentially a small jacketed oven heated to 135° C. by a constant boiling liquid, whereas the imported device employs electric coils and thermostatic control. The particular advantage of this apparatus over its prototype is the doubled capacity and the design for use with official AOCS moisture dishes. However, having occasion subsequently for another apparatus for use in the plant where single tests only were required, a much simpler design was built, shown in Figure 3. The principle of operation is the same, except that the test needs no attention during the 15 minutes' drying period. The box was made up from 1/16-in. brass plates, except for the top, which is ⅛-in. stock (to withstand warping under the heat of the brazing torch). All were cut on order to the proper length from the proper width strap brass. The housing was bent from ⅛x3-in. copper busbar stock; a plate was brazed over the rear, then openings filed in it near the bottom to admit air. The stack is 1-in. brass tubing; the air-cooled reflux condenser, a Butt tube. The heating liquid should be chosen so that when it boils, the air temperature at the base of the stack is 136° to 142° C., and does not fall below 120° C. when the oven is loaded. The writer uses a petroleum fraction boiling at 240° to 250° C.

The drying time is determined by

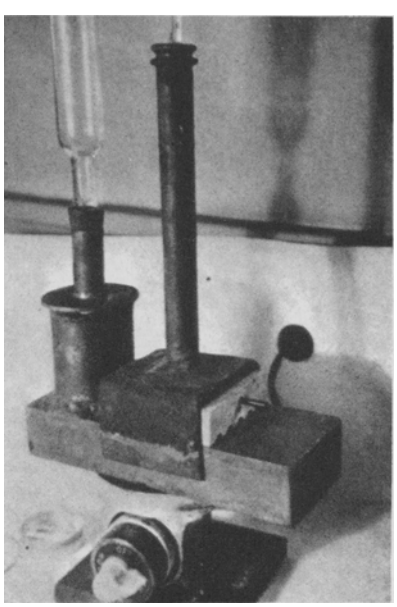


FIGURE 3

Fig. 3. Rapid moisture tester. Model designed for alternate use as controlled temperature hot-plate, using a boiling liquid or an oil bath. Length 8", width 4".

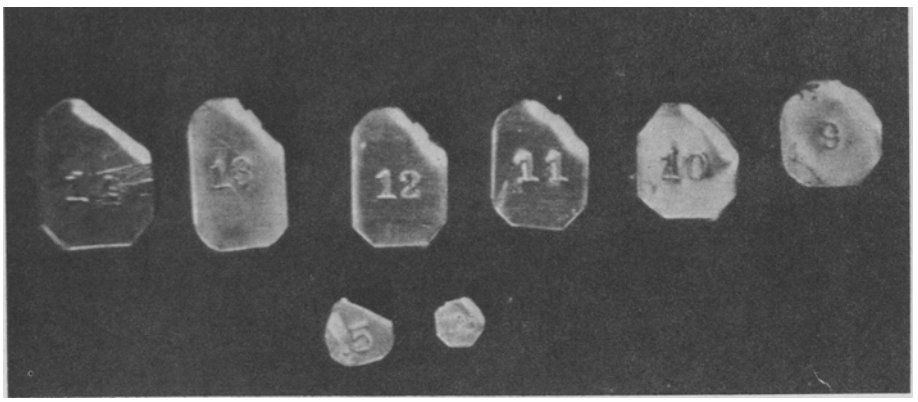


FIGURE 5

Fig. 5. Weights made from stainless steel sheet for use with 10-gram sample in plant rapid moisture tester by unskilled operator. The numbers represent per cent moisture. (Actual size.)

the combination of a number of conditions. In preventing local scorching on the bottom, better accuracy was obtained by having a thin asbestos pad beneath the dish. This, however, increased the drying time to 25 minutes using ten grams in two-inch dishes; but by substituting three-inch dishes the time was reduced to 13 minutes as a result of doubling the heating surface. These relations are shown in Figure VI.

Designed primarily for use in the plant by an untrained operator, the technique was simplified to the utmost limit consistent with the degree of accuracy desired, 0.25 per cent. The conditions were adjusted to yield results on a 10 gram sample agreeing with tests run 1½ hours in a Freas oven at 105-110° C. By using 10 grams the accuracy of weighing becomes less important, so a so-called pulp balance accurate to 0.01 gm. (with care, 0.005) is used. Use of counterpoised dishes permits simplification of the weighing. A series of weights ranging from 0.8 to 1.4 gms. were cut from light stainless steel sheet; one corner of each was bent up for the forceps, the result being a series of weights corresponding to whole number moisture percentages. The numbers 8, 9, 10, etc., were stamped on them. Fractional weights representing 0.5 per cent and 0.25 per cent moisture were made from aluminum. Thus, after cooling his dried test between copper slabs (requiring about 3 minutes), the operator places it on the left-hand balance pan and balances the dish counterpoise and 10 gm. weight by adding the proper moisture weight or weights to the test; that is, he replaces the water lost by its equivalent in weights, and reads from them the percentage directly. Table I shows results obtained under laboratory conditions. Table II represents the testing of soybean meal in the plant.

TABLE I—CHECK RESULTS

Material	1.5 Hours at 110° in Freas Oven %	15 Min. at 135-140° C. in Freyer Tester %
Linseed meal	8.28	8.36
	11.13	11.19
	10.72	10.54
	10.88	10.86
Castor beans	5.0	5.0
Fuller's earth	9.01	9.16-9.20*
Meal	9.1	9.1
	8.3	8.3
	9.5	9.55

*Same material put through second time.

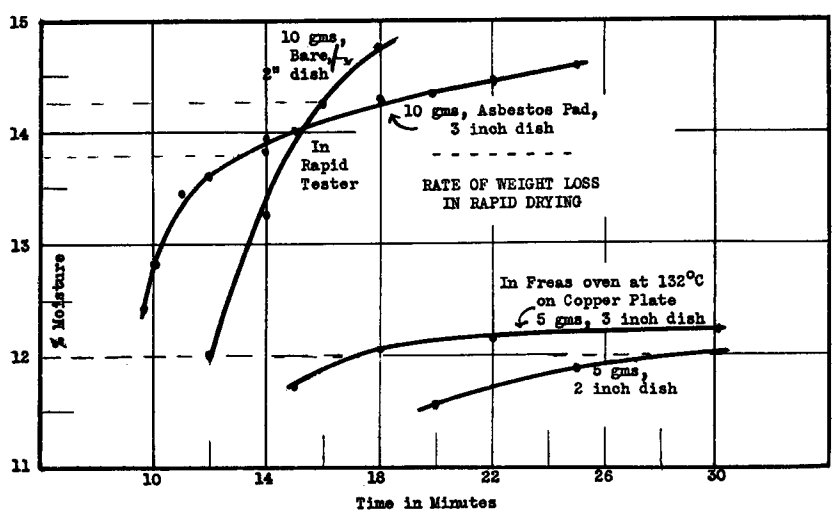


FIGURE 6

MOISTURE LOSS FROM SOYBEAN MEAL

Relation of drying time to sharpness of the break in the drying curve. Dotted lines represent moisture content indicated by the standard oven test. It is noted that in each case the set of conditions giving the most rapid drying also produces the flatest curve after the drying end-point has been passed. Thus in the Freas oven the end-point is reached in 17 minutes when drying 5 grams on a copper plate in the large dish, whereas 28 minutes is required when the smaller 2" dish is used, showing that in rapid moisture testing one of the most important factors is bringing the material up to temperature initially.

USE IN CONTROL

The ability to complete a moisture determination in twenty minutes in the factory and not require more than five minutes of the operator's time makes possible an accuracy of moisture control not generally realized by using ordinary methods. Moreover, in unloading and storing cottonseed from cars in which there are widely varying moisture contents, a rapid test is of use in permitting the wettest seed to be segregated and run through the mill before it becomes seriously damaged. Where tests are desired at thirty minutes or hourly intervals the time lag inherent in using the ordinary oven test precludes close control; and even if the plant laboratory has a rapid tester, the conveyance of samples to it and reporting back results add delays—to say nothing of interrupting a chemist's routine, if he has duties in addition to moisture testing, as is usually the case. Consequently, it is the writer's impression that moisture control is frequently regarded as "not worth the candle"—it is skimped over, with only a few daily tests made, and these in the day shift often twenty hours after

TABLE NO. II

Agreement of Plant Operator's Rapid Moisture Tests with Laboratory Tests Made in Freas Oven, 1.5 Hours at 105-110° C.

Remarks: 10 gram samples in 3″ moisture dish were used on thin asbestos pad. The moisture values ranged between 10 and 13%. The apparatus and method is capable of much better accuracy than these figures show, as indicated by the results given in Table I. The plant operator who obtained these results was not of the type to take pains; his use of the interval timer was even rather inaccurate.

Date	First few days of training	May 10	May 11	May 12	May 14	May 23
Testing time	14-15 min.	14 min.	13 min.	13 min.	13 min.	13 min.
Difference in % moisture reported. + indicates plant result was high..	+ .65	+ .22	— .01	+ .55	+ .06	+ .21
	+ .30	+ .60	— .23	+ .22	+ .08	+ .38
	+ .22	+ .65	+ .29	— .01	+ .80	+ .35
	+ .35	+ .28	— .15	+ .38	+ .22	— .06
	+ .54	+ .29	+ .30	— .07	...	+ .43
	+ .30	...	— .13	+ .36	...	+ .38
	+ .35	...	+ .05	...	...	...
	— .04	...	+ .18	...	...	...
	— .03	...	— .10	...	...	...
	— .02	...	...	...	...	...
	— .02	...	...	...	...	...
	— .05	...	...	...	...	...
	— .28	...	...	...	...	...
	+ .32	...	...	...	...	...
	+1.00*	...	...	...	...	...
	— .40	...	...	...	...	...
	+ .20	...	...	...	...	...
	+ .35	...	...	...	...	...
	+ .40	...	...	...	...	...

the taking of some of the samples, with the consequence that there is no control at all in the real sense of the word—only periodic checking of values; whereas by going to a little trouble to find a suitable method of testing, many oil mill and refinery operations, especially those of a continuous nature, can be conducted with enhanced efficiency, freeing the laboratory at the same time from a rather annoying burden of testing.

Notes on Conditions—Moisture in Oil

The apparatus was designed with the additional view of providing a hot-plate of definite controlled temperature for use in making moisture determinations on oil. The A.O.C.S. official hot-plate method specifies a temperature of 130° C. for this test but gives no hint of how this temperature might be measured. The use of a thermometer in the oil being tested is impractical because of weighing complications. The table given below shows one condition under which the test might be held at 130° C., using the apparatus described. Alternately, the boiling liquid in the tester might be replaced with oil and the temperature regulated at any desired value by use of a burner, the temperature being indicated by a thermometer placed in the *bath* oil.

The writer has seen unpublished data, which he has confirmed, showing that the requirements of our hot-plate method of bringing the oil to incipient smoking may cause high results to be obtained on Soybean Oil. The cause of accuracy would apparently be better served by changing the method so as to provide a definite maximum temperature to which the oil should be heated, even if momentarily; but should this be done, some such hot-plate of *controlled* and *indicated* temperature, as described, would be required.

RELATION OF VARIOUS TEMPERATURES

Boiling Liquid	194° F.	382° F.
Vapor	182	360
Air over Test	134	273
Oil in Dish on Bare Heater		
Outside of housing	154	310
Inside of housing	175	348
Oil in Dish on Asbestos Pad		
Outside of housing	130	266
Inside of housing	164	327

REPORT OF THE UNIFORM METHODS AND PLANNING COMMITTEE*

COLOR COMMITTEE:

The Color Committee have undertaken some very interesting work in the past year, which has not as yet been entirely completed. It is the recommendation of the Uniform Methods and Planning Committee that this work be continued.

COLOR GLASS DEVELOPMENT COMMITTEE:

This Committee had no recommendations to make, but the Uniform Methods and Planning Committee noticed that our methods call for the testing of standard glasses only by the Bureau of Standards. We, therefore, recommend that on page 16-e of our methods the statement be made that the Lovibond Color Glasses should be standardized either by the Bureau of Standards or the Electric Testing Laboratory.

*Reported at New Orleans, La., May 13, 1938.

CRUDE MILL OPERATIONS COMMITTEE:

This Committee made no formal report, but it was suggested by the chairman that a committee of this nature works under considerable difficulties, owing to the fact that developments in the crude mills are usually of a confidential nature. The Uniform Methods and Planning Committee, therefore, recommend that this committee be discontinued for the coming year.

FATTY ACID SOAP STOCK COMMITTEE:

This Committee made the following recommendations:

"1. That the methods for analyzing soap stock and accidulated soap stock, except from copra or palm kernal oils, as outlined, be suggested to replace the ones now given in the Methods of the American Oil Chemists' Society;

2. that the methods be designated as 'Dry Extraction Method for Total Fatty Acids of All Soap Stock and Acidulated Soap Stock, except from Copra or palm Kernel Oils,' and 'Wet Extraction Method for Total Fatty Acids of All Soap Stock and Acidulated Soap Stock, except from Copra or Palm Kernel Oils';

3. that the term 'petroleum ether' be adopted throughout all methods of the Society for the solvent variously referred to as petroleum and petrolic ether."

The Uniform Methods and Planning Committee approve these recommendations and suggest that the methods be included during the coming year as tentative.