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A. D. MELVIN, Chief of Bureau.

A RAPID METHOD FOR THE DETERMINATION OF WATER IN BUTTER.[a]

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Having given the subject of moisture content in butter considerable attention, the writer feels perfectly safe in stating that none of the methods for determining water in butter have supplied the need felt by the average manufacturer and dealer. The most approved methods require skill and time for operation, expensive apparatus, and several hours to complete a moisture determination. Probably the most satisfactory rapid method is that described by H. Droop Richmond,[b] which, in substance, is as follows: Weigh out 5 to 10 grams of butter into a suitable tared receptacle (dish or beaker containing a glass stirring rod); heat over a direct flame or on a sand bath, while stirring constantly, until all foaming ceases; cool and again weigh, the percentage of loss being calculated and considered as water.

Other simple methods have been devised, but on account of not giving accurate results they have been of little value. Appreciating the need of a simple and accurate method for determining water content in butter, and believing that such a method would be of material value to the manufacturer, dealer, and exporter, whether handling butter on a large or a small scale, the writer has spent considerable time and effort in attempting to devise such a test. A number of schemes have been tried, some giving results more or less satisfactory, but only one has been perfected to such an extent as to give results which seem wholly satisfactory. The writer firmly believes that with this method (requiring apparatus costing but a few dollars) anyone of average intelligence, after a few trials, may make moisture determinations which will compare very favorably with the results obtained by the most approved method now in use.

[a] Application for United States patent on the apparatus and method herein described has been filed under the act of Congress of March 3, 1883, so that the method may be used by the Government of the United States, or any of its officers or employees in the prosecution of work for the United States, or by any person in the United States, without the payment of royalty.

[b] Laboratory Book of Dairy Analysis. London, 1905.

APPARATUS.

The apparatus required for making the test is as follows:

Balance.—Sensitive to 0.025 gram. A balance suitable for weighing samples of cream for the Babcock test should be satisfactory for weighing samples of butter for this test; however, there are many cream balances in use which are not accurate enough for weighing either cream or butter samples.

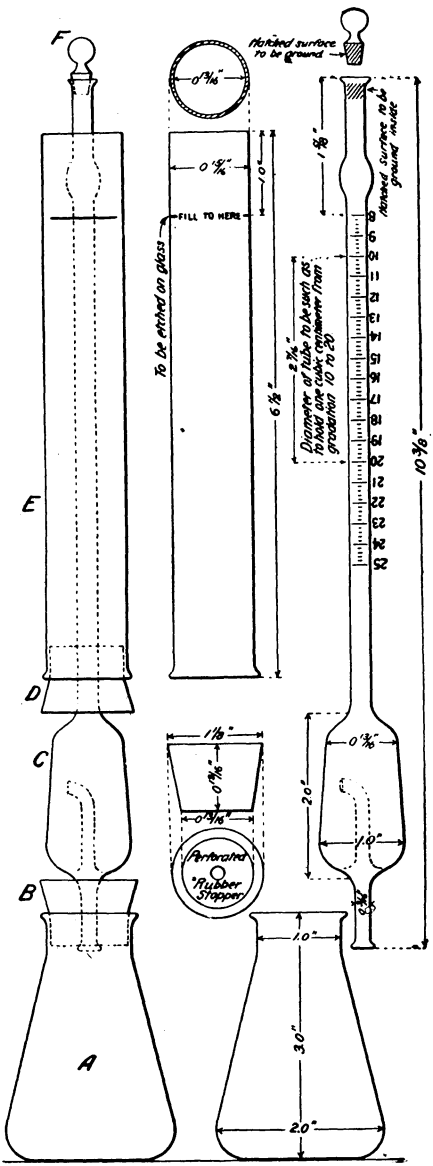

FIG. 1.—Special apparatus for a rapid method of moisture determination, a form giving most satisfactory results for determining water in butter.

Weights.—One 5-gram and one 10-gram.

Graduate.—For measuring 6 c. c.

Burner.—If gas is not readily available an alcohol lamp may be used.

Paper.—Parchment, 5 by 5 inches; must be perfectly dry.

Special apparatus.—As shown in figures. Referring to figure 1, A is a flask of a capacity of a little over 70 c. c. C is a graduated tube, which is connected with the flask A by means of a rubber stopper, B. F is a glass stopper ground into the tube C. Each glass stopper is ground to fit a particular tube and will not properly fit other tubes. Each stopper and tube should be marked, by the manufacturers, to eliminate the danger of using a tube with a stopper which has not been ground to fit. The tube C is graduated after this glass stopper F has been ground in, the zero mark being the end of the stopper. Each mark of the graduation represents one-fiftieth c. c., or when a 10-gram sample of butter is used each mark represents two-tenths of 1 per cent of water. E is a glass condensing jacket con-

nected to the graduated tube C by rubber stopper D, as shown in figure 1, or ground onto the bulb of the tube C at the point D, as shown in figure 2. The apparatus shown in figure 1, having the condensing jacket connected by means of a rubber stopper, is the form which seems most satisfactory for general use.

Rubber stoppers.—The rubber stopper B will be slowly decomposed by the heat and reagent during the process of making the tests. As a stopper is rendered unfit for use by making about 100 determinations, extra rubber stoppers should be obtained.

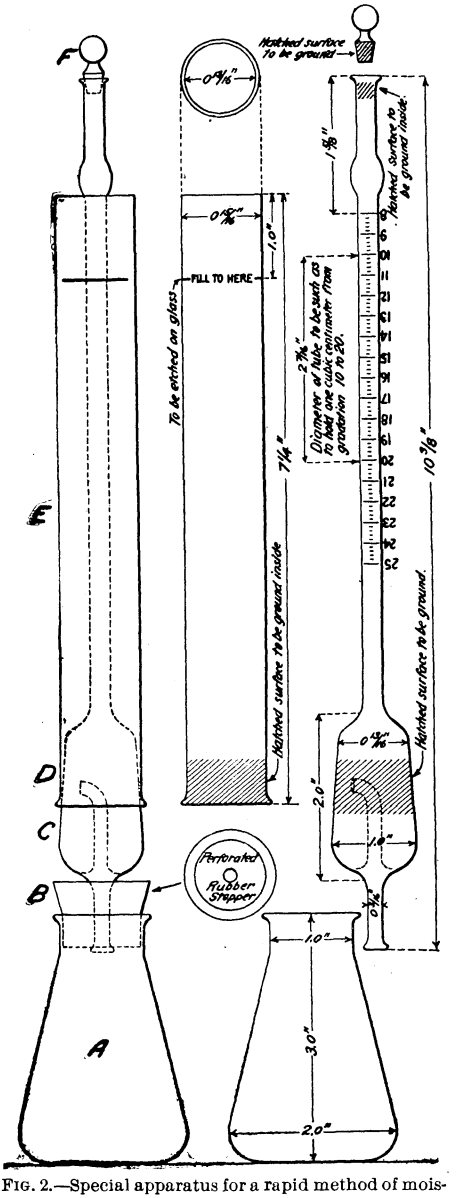

FIG. 2.—Special apparatus for a rapid method of moisture determination, showing different manner of connecting condensing jacket.

REAGENTS.

Amyl reagent.—A mixture of amyl acetate 5 parts and amyl valerianate 1 part. Must be free from water-soluble impurities in order to give accurate results. Users not in position to test this reagent for impurities should insist on a tested article.

Alcohol (for burning) when alcohol lamp is used.

MAKING A DETERMINATION.

Preparing the sample.—The sample of butter is placed in a suitable container (1-pint Mason jar or metal cup will be satisfactory). This container is placed in water at about 100° F. The butter is stirred with a spatula or spoon until it is about the consistency of thick cream and no free water can be seen. Samples of butter should not be left standing in open containers any length of time before making water determination, as some of the moisture will evaporate and the percentage of water shown when the determination is finally made will be too low.

Weighing the sample.—Place on each pan of the balance one sheet of parchment paper and balance accurately. Place the 10-gram weight on one pan and balance again by placing butter on the parchment paper on the opposite pan, placing the sample as near the center of the paper as possible.

Transferring sample to flask.—When exactly 10 grams are weighed out remove the sample from the pan and fold it in the parchment paper in such a shape that the paper and butter may be slipped into the flask A. Always use care that none of the butter is lost in transferring.

Adding amyl reagent.—Fill the graduate with amyl reagent to the mark 6 c. c., first being sure that the graduate is free from water. Place the 6 c. c. of amyl reagent in the flask with the butter.

Connecting the apparatus.—Connect the apparatus as shown in figure 1 and fill the condensing jacket E with cool water to within 1 inch of the top. Remove the stopper F.

Distilling off the water.—CAUTION: Be sure that the glass stopper F is removed.

Place the apparatus over the flame of the burner, applying heat to the bottom of the flask A. In a short time the butter will melt, running from the parchment paper into the amyl reagent. The water in the sample then boils and passes as steam into the tube C, where it is condensed and trapt. Watch the condensation in the graduated part of the tube C, and do not let the steam get higher than the 15 per cent mark. If it goes higher than this, remove the flame, as there is danger of water being lost. If there is any indication of liability of the mixture in the flask A foaming over, remove the flame. Foaming is usually prevented by 6 c. c. of amyl reagent, but some samples of butter, especially those of high moisture, require a trifle more than 6 c. c. In case of continued foaming, allow the mixture in the flask to cool, and add about 2 c. c. of the amyl reagent, and continue heating. After the water in the sample has boiled out, the temperature rises and the amyl reagent boils, driving the last traces of water and water-vapor from the flask and bottom of the stopper. Some of the amyl reagent is carried into the tube C with the steam, and some is boiled over after the water has been driven off. This amyl reagent in the tube is of no disadvantage.

Determining when all water has evaporated.—The time required for driving all water from the sample is not less than 5 minutes and with most samples need not be more than 8 minutes. When the mixture in the flask becomes a brown color and all the crackling noises in boiling cease, it is safe to conclude that all water has been driven from the flask.

Disconnecting the apparatus.—Disconnect the flask A from the stopper B, place the glass stopper F in the tube C, giving it a slight

turn to insure its being held firmly; invert the tube C, first being sure that the mouth of the small tube inside the bulb is held upwards; pour the water from the condensing jacket E, after which the jacket may be removed.

Separating amyl reagent from the water.—When the tube C is inverted the water and amyl reagent flow into the graduated part of the tube. To separate these and to get the last traces of water down into the graduated part, the tube C is held with the bulb in the palm of the hand and the stoppered end away from the body, raised to a horizontal position, and swung at arm's length sharply downward to the side. This is repeated a number of times until the dividing line between the water and amyl reagent is very distinct and no amyl reagent can be seen with the water or vice versa. The tube should then be held a short time with the stoppered end downward and the amyl reagent in the bulb of the tube agitated in order to rinse down any water that may be adhering to the sides of the bulb.

Reading the test.—The reading should not be taken until the tube and its contents have cooled so that very little warmth is felt. The water is in the bottom of the tube, and when a 10-gram sample is taken the percentage may be read directly. Read to the lower part of the meniscus.

Other than 10-gram samples.—With butter very low in moisture it may be desirable to take a 15-gram sample, and with butter extremely high 5-gram samples may be used. The reading multiplied by 10 and the product divided by the weight in grams of the sample taken equals the percentage of water.

Time required.—To make a determination, including weighing sample, requires from fifteen to twenty minutes.

CLEANING THE APPARATUS.

Flask.—The flask may be cleaned by washing with soap, washing powder, or washing soda in hot water. It is not absolutely necessary to wash the flask after each determination; the residue may be poured out and the flask wiped with a cloth or thin paper. The flask must always be dry (free from water) before making a determination.

Graduated tube.—After making the test, empty the tube C by holding the stoppered end downward, removing the stopper and allowing the contents to flow out quickly. In this way the amyl reagent runs out after the water and carries with it practically all of the water, which might otherwise adhere to the tube. The tube, after emptying, should be swung in the manner described for separating water from amyl reagent, which will almost completely empty it. Following this plan it is not necessary to dry the tubes after each determination. Occasionally the tubes should be washt carefully with a hot solution of sodium carbonate (sal soda) and thoroly dried before using.

THIS METHOD COMPARED WITH THE OFFICIAL METHOD.

In order to give some idea as to how results obtained by the method just described compare with results obtained by the method adopted by the Association of Official Agricultural Chemists[a] for determining water in butter, the following table is presented. This table is prepared from results of determinations made by Mr. C. W. Fryhofer, Scientific Assistant in Dairying, Dairy Division.

Comparison of results of determinations by the new method and by the official method.

Butter sample No.—	Per cent of water.			
	Official method.		New method.	
	A.	B.	A.	B.
1	12.60	12.62	12.7	12.7
2	12.51	12.52	12.6	12.6
3	10.52	10.45	10.5	10.55
4	9.93	9.95	10.0	10.0
5	11.46	11.44	11.4	11.4
6	11.67	11.68	11.65	11.6
7	12.49	12.43	12.4	12.3
8	11.46	11.47	11.5	11.55
9	12.89	12.85	12.75	12.8
10	12.85	12.80	12.8	12.9
11	18.86	18.86	18.8	18.85
12	15.55	15.3	15.45	(a)
13	16.03	(a)	16.10	(a)
14	11.73	(a)	11.65	(a)
15	12.0	12.10	12.0	12.10
16	21.50	21.56	21.60	(a)
17	11.47	11.36	11.4	11.4
18	12.14	11.95	11.9	12.0
19	12.22	12.32	12.15	12.20
20	11.56	11.67	11.5	11.55
21 (renovated)	11.5	11.44	11.6	(a)

[a] Only one determination made.

NOTE.—Since devising this method the writer has learned that Dr. J. F. Hoffmann (Zeit. f. Angewandte Chemie, Jahrg. 1902, p. 1193; Grain Dealers' Journal, Chicago, 1906, p. 526) has described a method for determining moisture in grain, based on the fundamental principle of driving moisture from the sample by heating, condensing the water, and measuring the same; however, accomplishing these results with apparatus and reagents quite different from those used in this method. The apparatus and the use of amyl reagents as described herein are believed to be original and new.

Approved:

JAMES WILSON,
Secretary of Agriculture.

WASHINGTON, D. C., *November 22, 1906.*

[a] Bulletin No. 46, revised edition, Division of Chemistry, U. S. Department of Agriculture, page 43.

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