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A NEW METHOD FOR DETERMINING FAT AND SALT IN BUTTER, ESPECIALLY ADAPTED FOR USE IN CREAMERIES.

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Numerous attempts have been made to modify the Babcock test so that it can be used in testing butter for butter fat. To the author's knowledge none of these have been even partially successful. The reasons for this are obvious. Butter contains a fairly large percentage of salt, and the sulphuric acid used in the test reacts with the salt, setting free gaseous hydrochloric acid. The latter chars the fat and produces a large amount of foam, which is sufficient unless great care is used to force part of the contents of the bottle out through the neck. One modification suggests avoiding this feature by adding the sulphuric acid dropwise with continuous agitation.

Again, in estimating fat by volume we must assume that a definite weight always occupies a definite volume at the same temperature; in other words, that butter fat has a definite specific gravity. This is not true. Butter fat is composed of a number of fats in varying proportions and the specific gravity varies in consequence. This variation is not much, to be sure; but in a substance containing more than 80 per cent of fat it is enough to play a very important part when the fat is read by volume.

In practical operation another factor enters which will produce a greater error than the variation in specific gravity; this factor is the temperature of reading. It is not easily possible in a creamery to control the temperatures at which the readings are made, and a small variation here from the temperature at which the bottle is standardized will make a large error in the estimated percentage.

Early in the investigation of which the test herein described is the outcome the author became convinced that any test for butter fat in butter, based on reading the percentage of fat by volume, was not feasible. Any test to be of value in a creamery must be (1) accurate,

(2) it must not take too long to operate, and (3) it must be capable of being used by those untrained in the use of scientific instruments.

Several satisfactory moisture tests are already in use in creameries. The test brought out herein, which embraces a test for salt, as well as for fat, will complete the analysis of butter by rapid and simple methods.

In the new test for fat the salt and part of the curd are first removed with hot water, the remaining curd is dissolved in dilute sulphuric acid, the acid solution is removed from the fat, and the latter weighed.

DETERMINATION OF FAT ALONE.

APPARATUS REQUIRED.[a]

A Babcock centrifuge.

A special separatory funnel.

A balance which is sensitive to 0.01 gram. (A torsion balance such as is used in the moisture test will answer if it is in good condition.)

An accurate set of metric weights.

A 10-cubic-centimeter graduated glass cylinder.

A 100-cubic-centimeter glass beaker.

Special separatory funnel.—This is essentially a separatory funnel with a capillary stem. The capacity of the funnel should be about 75 cubic centimeters and its weight when empty should not exceed 70 grams. The stopper may be dispensed with if desired. It is a convenience in the final weighing, but not a necessity. Figure 1 shows the form and dimensions of the funnel. It is so simple in construction that a further description is hardly necessary.

Special socket.—This is a double socket for holding the above funnel while centrifuging, and is made of heavy sheet copper with hangers of steel. Each socket will hold two funnels. The cut shows the construction and dimensions. It differs in no material way from the socket ordinarily used on the Babcock centrifuge, except for the opening in the side. If the dimensions given fail to fit the centrifuge at hand, they may be changed to suit so long as the dimensions of the barrels are not altered. Care must be taken that the capillary stem of the funnel does not project far enough through the hole in the socket to strike against the side of the centrifuge when being whirled. It is best to fit a disk of rubber gasketing to the bottom of the socket.

SAMPLING THE BUTTER.

In the determination of fat in butter, great care must be taken in securing a representative sample and in preparing this for the test. Errors introduced by improper sampling are far greater than those in the actual test.

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[a] Applications for United States letters patent on the special apparatus and on the method for fat herein described have been filed under the act of Congress of March 3, 1883, so that the method may be used by any person in the United States without the payment of royalty.

Samples are best taken with a butter trier, and one should always take several plugs from different parts of the tub or churn. These are placed in a suitable container, such as a 1-pint Mason preserve jar or a cup, which is placed in water at about 100° F. The sample is then mixed with a spatula or spoon until about the consistency of thick cream. The sample must not be left any length of time in open containers, since some of the moisture will evaporate. Should

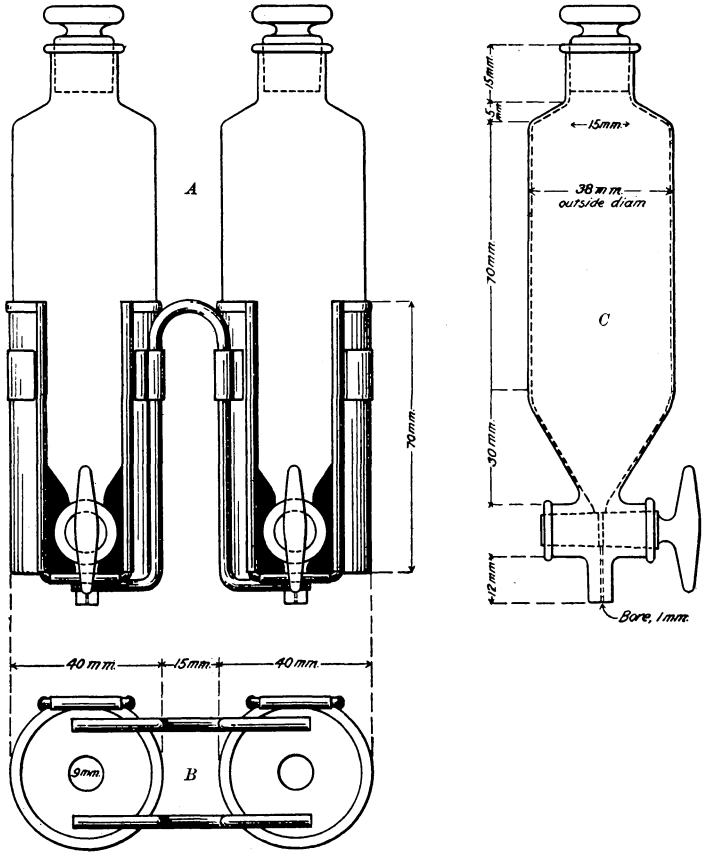

FIG. 1.—The special apparatus for determining fat and salt in butter. *A*, the socket with funnels in position; *B*, view of socket from below; *C*, the separatory funnel with capillary stem. (Reduced one-half.)

the sample be kept for any reason for a day or two before it is mixed, it should then be placed in warm water (with the cover on the container) until melted, and then cooled while being vigorously shaken until it solidifies. The reason for this is that on standing some of the water will ooze out and can not be reincorporated except by emulsifying and cooling while in this condition. Too much stress can not be laid on careful sampling and mixing the sample, for upon this the accuracy of any determination in butter very largely rests.

It will be found more economical in some cases if 4 or multiples of 4 determinations are made at once. In this case the 2 double sockets containing the funnels will balance when placed opposite in the centrifuge. If but 1 or 2 determinations are to be made it will be necessary to balance the centrifuge by putting weights in the opposite socket. First of all the weight of the clean and dry separatory funnel must be ascertained, and this as well as the other weighings involved must be done with care. This weight once found will suffice for all determinations made with that particular funnel unless by accident some of the glass should be chipped off. A slight scratch made with a file can serve to identify the funnels. A paper label can not be used.

I. Weighing the charge.—Counterpoise the small beaker on the balance and carefully weigh out 20 grams of the sample mixed as directed.

II. Transferring the charge to the separatory funnel.—Place the beaker containing the charge on a radiator or steam pipe until the butter is melted. (This may also be accomplished by adding a small quantity of boiling water.) Next pour the charge into the funnel, which must be maintained in an upright position, and no part of the charge must be lost in transferring. With a fine stream of hot water rinse down the sides of the beaker and pour the rinsings into the funnel. Repeat this, using not more than a teaspoonful of water at a time until the funnel is full to within one-quarter of an inch of the shoulder. The rinsing can be done very conveniently with the arrangement on many steam centrifuges for filling the Babcock test bottles, i. e., the rubber tube ending in a glass or metal point and connecting with a water tank heated by steam. The point must be fine, however. Should it be larger than three-sixteenths of an inch it can be replaced with the tip of a small oil can. Should this arrangement not be at hand one can easily be improvised from a tin can, a rubber tube, and an oil-can tip. In transferring the melted butter and rinsings the last drop may be prevented from running down the outside of the beaker by touching the lip of the beaker to the neck of the separatory funnel.

III. Centrifuging.—Insert the separatory funnel in the special socket, allowing the stem to project through the hole in the bottom and the handle of the stopcock through the open side. (Caution: The socket must always be placed in the centrifuge with the open side facing the direction in which the wheel revolves. This is very important, for if the opening faces the reverse direction the stopcock will be thrown out and broken.) Whirl one minute at the same speed used in testing milk with the Babcock test. The centrifuge must be kept warm.

IV. Removing the water.—Remove the separatory funnel from the socket and allow the water to flow through the stopcock until the fat (or curd) is within one-eighth of an inch of the stopcock. In this and subsequent operations involving the stopcock one must be sure it does not stick. It must always be under control, and it is best to give it frequent slight movements when the water or acid is running through it to be sure that this control is maintained; otherwise it might stick at a critical moment and the determination be lost. The most of the salt and part of the curd are taken out by the water. The remainder of the curd and all of the fat stays in the funnel.

V. Dissolving the curd.—Measure out 9 cubic centimeters of cold water, preferably condensed steam, with the glass graduate and pour into the beaker. Add to this 11 cubic centimeters of sulphuric acid of the same strength used in testing milk and cream (specific gravity, 1.82–1.83) and mix by gently shaking. (Caution: Always add acid to water and not water to acid, or a serious accident may result.) While still very hot add the mixture to the contents of the separatory funnel. Now dissolve the curd by giving the funnel a circular motion with the hand grasping the neck. Centrifuge one minute, as before. Draw off the acid solution till the fat layer is within one-fourth of an inch of the stopcock and repeat the operations in this paragraph.

VI. Freeing the fat from the acid solution.—The fat will now be in a clear transparent layer free from curd, and the solution below it will be practically colorless. To separate these two, draw off the latter until the fat nearly reaches the stopcock and centrifuge another minute. Now allow the fat to come down through the stopcock till it just reaches the end of the capillary stem. This last step offers no difficulties, providing the stopcock is kept in control, but it requires care.

VII. Determining the percentage of fat.—Carefully dry the separatory funnel on the outside with a clean soft towel and weigh it. The weight thus obtained minus the weight of the empty funnel represents the weight of butter fat in 20 grams of the sample. The percentage is obtained by dividing this weight by 2 and multiplying by 10.

Sometimes it is possible to obtain a clear fat layer with but one addition of acid, but in the majority of cases it will be found necessary to add it the second time, as directed. The proportion of acid and water selected is the outcome of a number of experiments, and is the one that yields the best results. The test for fat alone involves 4 centrifugings of 1 minute each. The centrifuge should be kept warm and the contents of the funnel in a melted state when the acid is added. The time consumed should not be much longer than it takes to test cream with the Babcock test, and the operations involved are simple and easily learned. No difficulty has been

experienced in obtaining a clear fat. Occasionally there will appear a slight emulsion at the bottom of the fat layer when the fat is drawn into the stem. This is so small in amount that it does not seem to affect the accuracy of the test to any considerable extent. The emulsion should be weighed as fat and considered as such.

CLEANING THE SEPARATORY FUNNELS.

The separatory funnels should be washed after each determination, but it is not necessary to dry them before use providing their weight, when clean and dry, has been found. The cleaning is easily done with hot water and either soap or cleansing powder. They should be well rinsed off with clean water and drained.

DETERMINATION OF SALT.

ADDITIONAL APPARATUS REQUIRED.

A 50-cubic-centimeter burette graduated in tenth cubic centimeters.

A 250-cubic-centimeter volumetric flask.

A 25-cubic-centimeter pipette.

A 250-cubic-centimeter beaker or white cup.

CHEMICALS REQUIRED.

An aqueous silver-nitrate solution containing 14.525 grams pure silver nitrate per liter. This solution may be obtained from a chemical supply house. A 10 per cent aqueous solution of potassium chromate, which may be obtained at a drug store.

METHOD.

To determine the percentage of salt the wash water, obtained as previously directed in Paragraph IV, is allowed to run into the 250-cubic-centimeter flask, and the operations in Paragraph IV conducted 3 times instead of but once, the water each time being allowed to run into the flask.

After the washings have become cool the flask is filled to the mark with cold water and the contents mixed. Twenty-five cubic centimeters, which represents 2 grams of the original sample, are then measured with the pipette into the beaker or cup and titrated with the silver-nitrate solution from the burette, using 2 or 3 drops of the potassium-chromate solution as the indicator. The first appearance of a permanent red is the end point. The silver-nitrate solution is of such strength that 2 cubic centimeters represent 1 per cent of salt if a 1-gram charge is used. In the above test where 2 grams are represented $\left(\frac{25}{250}\times 20\right)$ the number of cubic centimeters divided by 4

gives the percentage of salt in the original sample. As an example, if the burette reading showed that 10.6 cubic centimeters of the silver-nitrate solution were consumed in reaching the end point, then 10.6 divided by 4, or 2.65, would be the percentage of salt in that particular sample of butter.

ESTIMATING THE PERCENTAGE OF CURD.

If the moisture is determined in a separate charge by one of the reliable methods, the percentage of curd may be found by subtracting the sum of fat, salt, and moisture from 100.

A COMPARISON OF THE NEW METHOD WITH THE OFFICIAL LABORATORY METHOD.

Tables 1 and 2 show comparisons of the new method with the official laboratory method for both fat and salt. Two determinations of fat were made on each sample by both methods, and both determinations are included in Table 1 in order that the agreement of the duplicates may be seen. It will be observed in Table 2 that the new method gives a slightly lower figure for salt than the official method, the reason, of course, being that a trace of salt is left in the separatory funnel after the third washing. It is believed, however, that this is quite close enough for practical purposes. Should more exact results be desired, it is only necessary to increase the number of washings, as described in Paragraph IV.

All of the analyses by the official method and a part of those by the new method were made by Mr. R. P. Norton, assistant chemist, and acknowledgment is hereby made to him.

TABLE 1.—*Comparison of results for fat obtained with the official and with the new method.*

Sample No.	Official method.			New method.		
	Determination I.	Determination II.	Average.	Determination I.	Determination II.	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	87.60	87.56	87.58	87.66	87.75	87.70
2	85.73	85.67	85.70	85.52	85.54	85.53
3	84.87	84.91	84.89	84.87	84.87	84.87
4	84.67	84.62	84.65	84.52	84.55	84.54
5	86.04	86.31	86.18	86.36	86.24	86.30
6	87.13	87.07	87.10	86.91	87.06	86.99
7	84.03	84.17	84.10	84.11	84.39	84.25
8	86.82	86.84	86.83	86.80	86.77	86.79
9	84.48	84.58	84.53	84.57	84.33	84.45
10	85.11	85.15	85.13	84.96	85.20	85.05
11	82.76	82.86	82.81	82.73	82.67	82.70
12	80.01	80.16	80.09	80.09	79.89	79.90
13	79.53	79.59	79.56	79.38	79.42	79.40
14	86.35	86.48	86.42	86.29	86.12	86.21
15	80.86	80.87	80.87	80.80	80.73	80.76
16	79.93	79.90	79.92	79.81	79.79	79.80
17	81.27	81.32	81.30	81.08	81.26	81.17
18	81.18	81.21	81.20	81.16	81.37	81.27

Comparison of results for salt obtained with the official and with the new method.

Sample No.	Official method.	New method.	Difference.	Sample No.	Official method.	New method.	Difference.
	Per cent.	*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
1	2.15	2.10	0.05	5	2.05	2.00	0.05
2	2.33	2.27	.06	6	2.51	2.46	.05
3	2.32	2.27	.05	7	2.23	2.17	.06
4	1.71	1.63	.08	8	2.63	2.57	.06

Approved:

JAMES WILSON,

Secretary of Agriculture.

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