

THE DETERMINATION OF BUTTERFAT IN ICE CREAM EMPLOYING MIXED PERCHLORIC AND ACETIC ACIDS

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The Babcock determination of butterfat in milk, cream and certain milk products such as skim milk (2) has been an established procedure for over 50 years. Probably no method of analysis has ever had a record remotely approaching the frequency with which the Babcock test has been applied in the dairy industry.

The unmodified Babcock butterfat test cannot be applied successfully to dairy products containing added sugar due to the charring action of the sulfuric acid. It was the purpose of the present work to show that the use of perchloric and acetic acids in place of sulfuric acid modifies the standard Babcock test to make it applicable to ice cream mix for the determination of butterfat. It can be applied without alteration of existing equipment and with marked improvements in speed, accuracy and simplicity. It diminishes the number of required manipulations per determination, as it is not necessary to add water and the bottle is centrifuged for only a 2-minute period. The increased cost of the mixed perchloric-acetic acid which it employs is more than justified by the saving in time and the abbreviation in operative details. Moderate variation in the amount of acid mixture used does not affect the accuracy of the test.

A mixture of 72 per cent perchloric acid and glacial acetic acid react to form two possible compounds (8), one with the ratio one molecule of perchloric acid to two molecules of acetic acid and the other compound with the molecular ratio of 1 to 1. Such mixtures are not hazardous to mix and may be stored without deterioration. At the boiling point, the acetic acid is evolved and may be thus separated from the perchloric acid. By the process to be described, no precautions other than those applied to the unmodified Babcock test are required. The usual care in the handling of strong mineral acids apply to both procedures.

Sugar is soluble in 72 per cent perchloric acid without charring. Butterfat is as insoluble in aqueous perchloric acid as it is in aqueous sulfuric acid. The proteins of milk and cream are soluble in perchloric acid. Since butterfat in the presence of 72 per cent perchloric acid tends to darken at temperatures near 100° C., thus making reading of the test difficult, it was found desirable to use a mixture of equal parts of 72 per cent perchloric acid and glacial acetic acid as a substitute for concentrated sulfuric acid in the application of the Babcock procedure to the testing of ice cream. The presence of sugar, ice cream stabilizers, flavors and egg products or chocolate does not interfere with the test.

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It is beyond the scope of the present work to give in any detail reference to former procedures which have been developed as substitutes for the original Babcock test as applied to the testing of ice cream. The literature on the subject is very voluminous (1, 2, 4, 5, 6, 7). In no published procedure was found any record of the use of perchloric acid for the purpose of modifying the Babcock test as applied to fat determination in ice cream or ice cream mix.

PROCEDURE

Apparatus and reagents. The perchloric acid-acetic acid mixture, which is the only reagent employed in this modification of the Babcock test, consists of equal parts of volume of 72 per cent perchloric acid ($\text{HClO}_4 \cdot 2\text{H}_2\text{O}$) and 95 per cent glacial acetic acid. Little heat is evolved from the mixing of these chemicals.

Standard Babcock equipment was used to measure the butterfat content by this method. Babcock 20 per cent ice cream test bottles graduated in 0.2 per cent were used throughout this study.

The Mojonnier test, a commercial adaptation of the official Roesse-Gottlieb method (3), was employed to carry out control determinations described in this work. All samples tested were evaluated both by the Mojonnier method and the perchloric-acetic acid process simultaneously and the results compared.

The perchloric acid-acetic acid process. The procedure of the test is as follows:

(a) Weigh a 9-g. sample of ice cream mix (or melted ice cream) into a 20 per cent Babcock ice cream test bottle.

(b) Add approximately 30 ml. of the acid reagent (equal parts by volume of 72 per cent perchloric acid and glacial acetic acid) to the test bottle, rinsing the adherent mix off the graduated stem of the test bottle into the body of the bottle as the acid is added. The ingredients should all be at room temperature during mixing.

(c) Digest the ice cream and acid mixture by immersion in boiling water for 5 minutes. No color forms at first, but upon heating in boiling water the mixture turns progressively tan, brown and finally a deep chocolate color. The curd is completely dissolved in 1 to 2 minutes. The mixture should be agitated two or three times during the digestion period. After 5 minutes, the fat will be found as an immiscible supernatant layer.

(d) Add enough of the acid mixture to bring the fat into the calibrated stem of the bottle.

(e) Place the test bottles in balanced pairs in a standard Babcock test centrifuge and revolve at proper speed for 2 minutes. If the centrifuge is heated to 60° C., the per cent of fat can be read as soon as the sample is removed from the centrifuge. If an unheated centrifuge is used, the test bottles should be tempered by immersion in a water bath (130°–140° F.) to the top of the fat column for 5 minutes before reading. The reading of the fat column is made in the customary manner after the addition of glymol.

(f) Contents of the test bottles should be poured into a reservoir of water and then emptied in the sink drain for disposal. The test bottle is rinsed with hot water and is ready for a second test. No coating of insoluble calcium salts ever accumulates on the inner walls of the test bottle. All mineral salts present in cream are soluble in the acid mixture used.

RESULTS

Experimental results on plain vanilla ice cream as compared with the Mojonnier test. Thirty-one different samples of plain vanilla ice cream were subjected to test. These samples were from a wide variety of commercial sources or were

experimental ice creams prepared in the University of Illinois Dairy Technology laboratory. No attempt was made to record their composition. The results are shown in table 1. The maximum deviation between the new method and the

TABLE 1

The analysis of plain ice cream and ice cream mix by the perchloric acid-acetic modified Babcock test and comparison with Mojonnier values

Sample no.	Perchloric acid method		Mojonnier method		Maximum variation from Mojonnier	Average variation from Mojonnier
	No. of analyses	Av. B.F.	Av. B.F.			
		(%)	(%)	(%)	(%)	(%)
1	10	11.33	11.22	+0.23	+0.11	
2	17	12.37	12.22	+0.23	+0.15	
3	18	9.05	9.03	+0.12	+0.02	
4	15	15.15	15.05	+0.13	+0.10	
5	7	12.03	12.00	+0.17	+0.03	
6	8	13.41	13.46	-0.26	-0.05	
7	15	11.80	11.89	-0.19	-0.09	
8	4	12.60	12.61	-0.11	-0.01	
9	4	12.15	12.22	-0.12	-0.07	
10	6	12.78	12.82	-0.22	-0.04	
11	4	11.78	11.69	+0.11	+0.09	
12	2	13.60	13.61	-0.01	-0.01	
13	5	10.56	10.51	+0.09	+0.05	
14	5	12.20	12.08	+0.22	+0.12	
15	6	10.13	10.17	+0.13	-0.04	
16	8	11.20	11.08	+0.22	+0.12	
17	4	10.68	10.65	+0.15	+0.03	
18	4	12.18	11.89	+0.31	+0.29	
19	37	12.42	12.49	-0.19	-0.07	
20	8	12.42	12.30	+0.26	+0.12	
21	8	12.48	12.44	+0.16	+0.04	
22	6	12.37	12.29	+0.11	+0.08	
23	12	12.51	12.21	+0.49	+0.30	
24	10	12.62	12.28	+0.37	+0.34	
25	10	12.70	12.43	+0.37	+0.27	
26	10	12.77	12.54	+0.36	+0.23	
27	8	12.91	12.64	+0.36	+0.27	
28	4	12.33	12.31	-0.11	+0.02	
29	4	11.98	12.02	-0.12	-0.04	
30	4	11.63	11.60	+0.10	+0.03	
31	4	11.13	11.20	-0.20	-0.07	
Summary	267				+0.07	

Mojonnier process was +0.49 per cent. The average algebraic difference was +0.07 per cent.

Eight analyses of the same sample gave 11.2 per cent for six determinations, 11.1 for one determination and 11.3 for the remaining test. The Mojonnier test for this sample was 11.08 per cent.

The determination of butterfat in chocolate ice cream. The procedure as described was applied to the determination of butterfat in eight samples of chocolate ice cream with the results given in table 2. Control analyses were carried out using the Mojonnier method. Results of the test of chocolate ice cream samples indicate that the perchloric acid-acetic acid procedure is satis-

factory for use in the determination of butterfat in chocolate ice cream. The average variation between the two methods was -0.11 .

SUMMARY

A new reagent has been described for use in a modified Babcock butterfat analysis of plain ice cream and chocolate ice cream. The reagent consists of a mixture of equal parts by volume of 72 per cent perchloric acid and glacial acetic acid. The test requires only one centrifugation and a complete analysis can be accomplished in about 8 minutes. The results are in close agreement with those obtained by the Mojonnier method.

TABLE 2
*The determination of butterfat in chocolate ice cream
by the perchloric-acetic acid procedure*

Sample no.	Perchloric acid method		Mojonnier method	Maximum variation from Mojonnier	Average variation from Mojonnier
	No. of analyses	Av. B.F.	Av. B.F.		
		(%)	(%)	(%)	(%)
1	7	14.36	14.37	-0.17	-0.01
2	12	13.28	13.44	-0.54	-0.16
3	2	20.05	20.14	-0.14	-0.09
4	5	11.00	11.07	-0.17	-0.07
5	4	10.10	10.01	+0.19	+0.09
6	5	15.42	15.12	+0.48	+0.30
7	7	10.89	11.10	-0.30	-0.21
8	7	12.53	13.26	-0.86	-0.73
Summary	49				-0.11

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