The Determination of

BUTTERFAT IN ICE CREAM AND ICE CREAM MIX

A Modified Babcock Test
Employing Mixed Perchloric Acid and Glacial Acetic Acid
Using Standard Babcock Equipment

SPEED -- ACCURACY -- WIDE RANGE -- CONVENIENCE

By
G. FREDERICK SMITH, Ph.D.
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FOREWORD

The Babcock determination of butterfat in milk, skim milk, and cream, is, beyond comparison, the most frequently employed method for a routine analytical application, ever developed. The number of individual determinations following this procedure applied annually has been conservatively estimated to be in excess of 50 million.

The unmodified Babcock test is not applicable to the determination of butterfat in ice cream or ice cream mix. The presence of sugar, vanilla, and excessive milk solids and other ingredients interfere.

Many procedures designed to overcome the difficulties have been devised. Still a better method was in demand. The present described process is thought to answer this need.

It would be almost folly to describe any new method which did not employ equipment customarily used in the unmodified Babcock test. The present described procedure meets this requirement.

Essentially but one alteration is a prerequisite. This involves the replacement of sulfuric acid by a mixture of perchloric and glacial acetic acids. We employ the same equipment. Most of the manipulations are duplicated.

The authenticity of the new procedure has been tested by two recognized authorities in dairy technology. The originators of the new technique are the present author, Prof. James Fritz, now of the chemical staff at Iowa State College, and Doctor Harry Pyenson, formerly engaged in dairy research at the University of Illinois.

The acid mixtures employed are commercially available or may be compounded by technicians applying the method. The individual acids of the mixed reagents are commonly available in regular trade channels.

The new method has already attained widespread adoption. The present purpose is to extend popular acceptance through the publication of all available data previously published in current scientific journals on this subject.

G. Frederick Smith
Professor of Chemistry
University of Illinois
Urbana 1954

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

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AND
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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, accurary 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 of 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 applies to both procedures.

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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.

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 ($HClO_4 \cdot 2H_2O$) 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 Roese-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.

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

(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 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.

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

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 satisfactory 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.

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BUTTERFAT IN ICE CREAM[1]

The Perchloric-Acetic Acid Test for Determining Butterfat in Ice Cream

A. H. WHITE[2]

In the dairy industry, the analytical test most extensively used is the Babcock test for determining the butterfat content in various products. As the usual procedure for milk and cream is not applicable to ice cream, a number of modifications have been proposed in order to minimize the charring effect of sulphuric acid on the sugar in ice cream.

One of the more commonly used modifications of the Babcock test for ice cream involves the use of 95% glacial acetic acid in combination with sulphuric acid. With careful procedures, this modification gives reasonably accurate results with plain ice cream, but it is not recommended for chocolate ice cream, and clear readings are not always obtained with fruit and nut ice creams.

Recently Smith *et al. (1)* at the University of Illinois have proposed a new modification using a 1:1 mixture of 72% perchloric acid and 95% glacial acetic acid. The authors state that sugar and milk proteins are soluble in 72% perchloric acid without charring while butterfat is as insoluble in the aqueous perchloric acid as it is in aqueous sulphuric acid. It was reported also that such ingredients in ice cream as stabilizers, flavors, egg products and chocolate do not interfere with the test. The authors also claim for the test "marked improvements in the speed, accuracy and simplicity," as it is not necessary to add water and there is only one two-minute centrifuging period.

The recommended procedure for the test is as follows:

Reagents and Equipment

The only reagent used is a mixture of equal parts by volume of 72% perchloric acid and 95% glacial acetic acid. When the two acids are mixed very little heat is generated. Standard Babcock test equipment is used with the 20% ice cream test bottle graduated to 0.2%.

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[2] Associate in Dairy Research.

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Procedure

(a) Weigh a 9-m. sample of ice cream mix (or melted ice cream) into a 20% Babcock ice cream test bottle.

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

(c) Digest ice cream and acid mixture by immersion in boiling water for five minutes. No color forms at first, but gradually the mixture turns progressively tan, brown and finally a deep chocolate color. The curd is completely dissolved in one to two minutes. The mixture should be agitated two or three times during the digestion period. After five 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 balance pairs in a standard Babcock test centrifuge and revolve at the proper speed for two minutes. If the centrifuge is heated to 60°C. (140°F.), 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 five minutes before reading. The fat column is read 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 into 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 accumulates on the inner walls of the test bottle. All mineral salts present in cream are soluble in the acid mixture used.

The results reported by Smith *et al. (1)* showed that the maximum deviation between the new method and the Mojonnier method was +0.49% for 31 samples of plain vanilla ice cream, while the average algebraic difference was +0.07%. The average of the individual variations, however, was 0.106%. On eight samples of chocolate ice cream the maximum variation was —0.73% and the average of the individual variations was 0.21%.

The new test appeared to be of sufficient merit to warrant further investigation on various types of ice cream. The results of trials carried out in the laboratory of the Division of Bacteriology and Dairy Research, Science Service, are reported herein.

Materials and Methods

Samples of packaged and bulk commercial ice creams of various kinds and ice cream mixes were used. A number of the samples were collected before the butterfat standard was increased to 13%. The new test was compared with the Mojonnier method for all samples and with the acetic-sulphuric acid test for a number of the plain, fruit and nut ice creams. All Mojonnier tests were made in duplicate, while the tests by the perchloric acid method were at least duplicated, and for a number of samples from three to eight replicates were made. The perchloric-acetic acid tests were carried out in strict conformity with the procedure recommended by the authors.

The samples containing fruits, nuts or other solid flavoring ingredients were divided into equal portions, and, after melting, one portion was strained through cheese cloth to remove the solid ingredients; these are indicated by the letter "A." The other portion was mixed in a Waring Blendor for four minutes which thoroughly chopped the solid fruits, nuts, etc., into fine particles; these are indicated as the "B" samples.

Results

The results of tests on plain ice creams and ice cream mixes are shown in Table 1. Sample No. 6 was a maple ice cream, while sample 25 was flavored with licorice.

The maximum variation between the perchloric-acetic acid test and the Mojonnier method was +0.25% while the average of all variations was 0.12%. For 20 of the 22 samples, the fat test by the perchloric acid method was slightly higher than that by the Mojonnier test. The Illinois tests showed 21 of 31 samples with higher fat tests by the perchloric than the Mojonnier method. Four of the five tests by the sulphuric-acetic method checked closely with the perchloric acid tests and were higher than those by the Mojonnier method.

The analytical data for the samples of ice cream containing fruits, nuts, etc., are given in Table 2. These samples varied considerably in the content of solid flavoring ingredients. For the strained "A" samples, the fat tests by the three methods agreed reasonably well. The maximum variations of the perchloric acid and the sulphuric acid modified methods from the Mojonnier test were +0.59 and +0.47%, respectively, and the averages of all the variations were 0.22 and 0.24%, respectively. However, visual observations of the completed tests showed that the fat columns obtained with the perchloric acid method were always clear and easily read, but with the sulphuric-acetic acid method in practically every case there was a varying

TABLE 1

Butterfat Determinations on Plain Ice Cream and Ice Cream Mix by the Modified Perchloric-Acetic Acid, Sulphuric-Acetic Acid and the Mojonnier Methods

Sample No.	Perchloric-acetic acid	Sulphuric-acetic acid	Mojonnier test	Ave. diff. perchloric and Mojonnier	Ave. diff. sulphuric-acetic and Mojonnier
6	9.40	9.40	9.26	+0.14	+0.14
15	9.55	9.50	9.40	+0.15	+0.10
18	11.20	11.15	10.96	+0.24	+0.19
19	9.45	9.55	9.37	+0.08	+0.18
25	9.50	9.10	9.31	+0.19	−0.21
32	9.95	.....	9.94	+0.01	.....
33	10.40	.....	10.38	+0.02	.....
34	10.30	.....	10.33	−0.03	.....
38	13.15	.....	13.03	+0.12	.....
39	13.25	.....	13.11	+0.14	.....
40	13.40	.....	13.37	+0.03	.....
41	13.35	.....	13.19	+0.16	.....
42	13.20	.....	13.04	+0.16	.....
43	13.50	.....	13.35	+0.15	.....
44	13.55	.....	13.44	+0.11	.....
45	13.90	.....	13.90	0.0	.....
46	13.40	.....	13.27	+0.13	.....
47	13.60	.....	13.35	+0.25	.....
48	13.25	.....	13.11	+0.14	.....
49	13.60	.....	13.42	+0.18	.....
50	13.60	.....	13.44	+0.16	.....
51	13.65	.....	13.55	+0.10	.....
Average....	12.19		12.07	+0.12	

amount of curdy material at the bottom of the fat column. In a number of the samples the curdy plug of material had risen in the fat column during the tempering period, thus tending to increase the fat reading. In other cases the lower meniscus of the fat column was so uneven that it increased the difficulty in reading.

With the "B" samples which were mixed in the Waring Blendor, it was impossible to get satisfactory readings with the modified Babcock methods, especially when there were considerable quantities of solid flavoring ingredients present. The fat columns with the perchloric acid test were clear, but a plug of the finely chopped solid flavoring materials formed in the graduated neck of the test bottle acting as a mechanical obstruction to the rising of the fat. This is indicated by the lower fat readings as compared to the Mojonnier tests in a number of samples. With the sulphuric acid test, there was excessive curdy material in the neck of the bottle, so that it was useless in some cases to attempt to determine the fat.

In the majority of cases the fat determinations by the Mojonnier method on the "B" samples were somewhat lower than the strained

TABLE 2

Butterfat Determinations on Ice Creams Containing Fruits, Nuts and Other Solid Flavoring Ingredients

Lab.	Perchloric-acetic method		Sulphuric-acetic method		Mojonnier method		Diff. perchloric and Mojonnier methods		Diff. sulphric and Mojonnier methods	
No.	"A"	"B"	"A"	"B"	"A"	"B"	"A"	"B"	"A"	"B"
3	8.80	......	8.60	......	8.93	......	−0.13	......	−0.33	......
4	8.62	......	8.40	......	8.78	......	−0.16	......	−0.38	......
5	9.30	......	9.30	......	8.97	......	+0.33	......	+0.33	......
9	9.60	8.60	9.40	8.60	9.28	8.58	+0.32	+0.02	+0.12	+0.02
11	9.95	8.00	9.55	7.55	9.90	9.47	+0.05	−1.47	−0.35	−1.92
12	10.40	8.80	10.40	7.50	10.21	10.52	+0.19	−1.72	+0.19	−3.02
13	9.60	9.35	9.65	8.80	9.37	9.29	+0.23	+0.06	+0.28	−0.49
14	9.50	9.00	9.30	9.00	9.16	9.12	+0.34	−0.12	+0.14	−0.12
16	9.45	7.80	9.35	......	9.29	8.79	+0.16	−0.99	+0.06	......
17	9.60	9.80	9.40	......	9.36	10.20	+0.24	−0.40	+0.04	......
20	9.60	9.25	8.60	8.30	9.01	9.05	+0.59	+0.20	−0.41	−0.75
22	9.75	9.00	9.40	......	9.72	9.25	+0.03	−0.25	−0.32	......
24	9.85	......	9.80	......	9.87	......	−0.02	......	−0.07	......
26	10.40	......	10.40	......	10.06	......	+0.34	......	+0.34	......
27	9.95	......	10.00	......	9.53	......	+0.42	......	+0.47	......
31	9.55	......	9.65	......	9.55	......	0.0	......	+0.10	......
Average....	9.62	......	9.45	......	9.44	......	+0.18	......	+0.01	......
Average of Variations........							0.22		0.24	

"A" indicates strained samples. "B" indicates samples mixed in a Waring Blendor.

"A" samples due to the dilution factor. However, the fat content was slightly higher in a few "B" samples which contained a considerable quantity of nuts or shredded cocoanut.

The results for ten samples of chocolate ice cream and chocolate mixes are shown in Table 3. Samples 21 and 35 showed markedly

TABLE 3

Butterfat Determinations on Chocolate Ice Cream

Lab. No.	No. of tests	Perchloric test ave. B.F.	Mojonnier method ave. B.F.	Diff. between perchloric and Mojonnier
21	3	7.93	9.25	−1.32
28	4	13.80	13.59	+0.21
29	4	8.78	9.13	−0.35
35	2	10.50	13.54	−3.04
36	4	10.60	10.59	+0.01
53	4	11.55	11.67	−0.12
55	6	12.40	12.52	−0.12
56	4	12.65	13.20	−0.55
57	8	12.13	12.60	−0.47
58	4	12.28	12.76	−0.48
Average of variations........				0.66

lower tests by the perchloric acid test than by the Mojonnier method. In both tests, there was a heavy plug of material at the bottom of the column, probably due to undissolved cocoa used as a flavoring ingredient. There was also a slight plug of material at the base of the fat column in samples 56, 57 and 58, as indicated by the difficulty encountered in emptying the test bottles. This plug of material, due to the addition of cocoa no doubt, caused a mechanical obstruction to the rising of the fat during centrifuging, and resulted in lower fat readings. This difficulty was not overcome by increasing the centrifuging period. The same trouble was experienced by the Illinois workers *(2)* with some chocolate ice creams. The perchloric acid fat determinations for the other five samples, which did not contain cocoa so far as is known, agreed quite closely with those obtained by the Mojonnier method.

Testing Samples Containing Formalin

At times, samples of ice creams or mixes are preserved by the addition of formalin. In testing a sample of chocolate ice cream which contained a few drops of formalin to 50-ml. replicate samples, complications were encountered. Further tests were made by adding one, three and five drops of formalin to 50-ml. replicate samples of the same ice cream and testing after several days. The results are shown in

Table 4. Even one drop of formalin in 50 ml. ice cream appeared to decrease the fat reading by the perchloric acid test, while three and five drops made the test useless as very little fat appeared in the graduated neck of the bottle. The perchloric acid test, therefore, cannot be used for samples of ice cream which have been preserved with formalin. Bird *(3)* found that samples of ice cream preserved with formalin cannot be tested by the glacial acetic-sulphuric acid modification of the Babcock test.

TABLE 4

The Effect of Formalin on the Fat Test of Ice Cream by the Perchloric-Acetic Acid Test

Sample No.	Mojonnier method ave. B.F.	None	Perchloric-acetic method Drops formalin per 50 ml. ice cream		
			1	3	5
1	13.30	13.60	13.0	2.4	1.6
2	11.67	11.55	10.6	1.0	no reading
3	.....	11.10	11.0	no reading	no reading
4	13.55	13.65	.....	.....	2.3
5	13.35	13.60	.....	.....	2.4

Observations on the Perchloric Acid Test

The results obtained in these trials indicate that the perchloric acid test has some advantages over modifications of the Babcock method employing sulphuric acid. The new test can be applied to most types of ice cream, and the fat determinations, in general, agree closely with those obtained by the Mojonnier method. Clear fat columns are obtained consistently with a clear-cut demarcation at the lower meniscus and with no charring or curdy material which frequently occurs when sulphuric acid is used.

In testing ice cream containing fruits, nuts, etc., as flavoring ingredients, the solid materials should be strained from the melted ice cream before sampling to obtain the best results. Chocolate ice creams containing some types of cocoa cannot be tested with accuracy due to mechanical obstruction to the rising of the fat during centrifugation.

The test can be completed rapidly as there are fewer manipulations and only one two-minute centrifuging period. Good lighting assists greatly in reading the tests as the acid-serum mixture under the fat column is blackish in color.

With certain limitations, therefore, the new perchloric-acetic acid modification of the Babcock test for fat appears to be a useful addition to plant testing methods. It should prove to be a valuable aid to the ice cream manufacturer who does not have Mojonnier equip-

ment and who is interested in keeping a close check on the fat standardization of mixes and frozen ice creams.

Cost of the Test

Perchloric acid of the required strength is considerably more expensive than sulphuric acid and the test requires 22 to 23 ml. of perchloric acid as compared to about 9 ml. of sulphuric acid. At the price of perchloric acid in 7-lb. quantities, the cost per test would be approximately 10 cents for the perchloric acid alone as compared to about 0.5 cents per test for sulphuric acid. However, the saving in time and the wider applicability of the new test would partially compensate for the increased cost.

Handling Perchloric Acid

The originators of the new test claim that only the usual precautions and care are necessary in handling perchloric acid as with any strong mineral acid. Very little heat is generated in mixing perchloric and acetic acids or when the acid is added to the sample of ice cream or mix. However, perchloric acid is rather inflammable when excessive quantities come in contact with organic materials such as wood or paper, so that care should be taken to prevent spillage on wooden table-tops or floors.

Summary

A new modification of the Babcock butterfat test for ice cream, in which the reagent is a mixture of equal parts by volume of 72% perchloric acid and glacial acetic acid, has been compared with the Mojonnier method and the acetic-sulphuric acid modification on various types of ice cream.

The new test is rapid, gives clear fat columns, and readings that are in close agreement with those by the Mojonnier method for most types of ice cream. More accurate results were obtained on ice creams containing fruits, nuts, etc., when the solid flavoring ingredients were strained from the melted ice cream. Low inaccurate results were obtained on some chocolate ice creams containing cocoa due to the formation of a plug of solid material in the neck of the bottle which causes a mechanical obstruction to the rising of the fat. The new test cannot be applied to ice cream or mixes which contain even a few drops of formalin as a preservative.

The cost per test by the perchloric acid method is considerably more than for the sulphuric acid modification, but the saving in time compensates somewhat for the increased cost.

The new test should prove to be a useful addition to plant methods of determining the fat content of most types of ice cream and mixes.

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PERCHLORIC ACID TEST FOR FAT *

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There have been numerous attempts to modify the Babcock test so as to make it applicable to the determination of fat in ice cream. Most all such modifications involve the substitution of either organic or inorganic acids for a portion or all of the sulfuric acid used in the regular Babcock procedure. The sample is weighed into an ice cream test bottle or an eight or ten per cent milk test bottle. Where chemicals less active than inorganic acids such as sulfuric are used, a digestion period in hot water usually precedes centrifuging. In some procedures glymol is used. There are two constituents of ice cream that make modification of the regular Babcock test necessary—one is the sugar and the other the added condensed milk solids.

One of the more recent modified Babcock tests for ice cream is that presented by Smith *et al. (1)*. This test involves the use of thirty ml. of equal parts of seventy-two per cent perchloric acid ($HClO_4 \cdot 2H_2O$) and glacial acetic acid, a digestion period of five minutes in boiling water, filling the bottle with the acid mixture, centrifuging for two minutes, tempering the fat by placing the bottle in a 135° to 140°F. water bath, the reading being made after the addition of glymol.

The Smith Report

Smith *et al.* reported thirty-one comparisons between the results obtained on ice cream mix using their method and the Mojonnier method, in which the average variation from the Mojonnier was +.07 per cent. The individual differences in tests varied from +.49 to a —.26 per cent. The mixes studied varied in per cent fat from 9.03 to 10.05.

At the suggestion of several industry laboratories, a study was made to determine the suitability of the test for mixes of low and high fat content. A preliminary survey revealed that while the original test as recommended by Smith *et al.* gave satisfactory results for vanilla mixes of approximately twelve per cent fat, low results were obtained for mixes testing four to six per cent fat, and high results were obtained when the test was applied to mixes containing approximately sixteen per cent fat.

* *Ice Cream Field*, November, 1951, page 60.

Various modifications of each step in the procedure recommended by Smith *et al.* were made in an attempt to change the test so as to make it more accurate for the low and high fat mixes. It was not found possible to obtain a single modification suitable for all mixes. Procedures satisfactory for low fat (four to six per cent) mixes and one satisfactory for medium fat (ten to twelve per cent) and high fat (fourteen to sixteen per cent) mixes were evolved.

TABLE 1

Results Using Modified Test on Medium and High Fat Mixes

Sample No.	% fat by modified Babcock	% fat by Mojonnier
121	16.4	16.38
	16.4	16.33
122	16.3	16.27
	16.3	16.29
123	15.5	15.41
	15.5	15.45
124	11.4	11.43
	11.4	11.38
125	12.3	12.22
	12.3	12.23
126	11.9	11.95
	11.9	11.92
127	16.4	16.38
	16.4	16.33
128	11.95	12.01
	11.95	12.00
129	16.3	16.27
	16.3	16.29
130	12.2	12.18
	12.2	12.15
131	11.9	11.84
	11.9	11.85
132	11.4	11.45
	11.4	

The modified procedures and typical results obtained when using these modified tests on plain ice cream or mix are as follows:

Procedure for high and medium fat mixes.

1. Weigh a 9-gram sample of the mix (or melted ice cream) into a twenty per cent Babcock ice cream test bottle.

2. Add thirty-three ml. of the acid reagent (forty parts by volume of seventy-two per cent perchloric acid and sixty parts by volume of glacial acetic acid) to the test bottle, rinsing the adherent mix free from the neck of the bottle as the acid is added. Place bottle in mechanical shaker and mix contents for at least one minute. The

ice cream mix and acid should be at room temperature at time mixing takes place.

3. Digest the mix and acid mixture by immersion of the bulb of the test bottle in water at 206° to 208°F. for five minutes. During digestion the mixture turns progressively tan, brown and finally a deep chocolate color. After five minutes the fat will occur as an immiscible supernatant layer.

4. Add enough of the acid mixture to bring the fat into the neck of the test bottle.

5. Place the test bottles in balanced pairs in a standard Babcock centrifuge and revolve at proper speed for two minutes.

6. If an unheated centrifuge is used place bottles in water bath (130° to 140°F.) for five minutes.

TABLE 2

Results Using Modified Test on Low Fat Mixes

Sample No.	% fat by modified Babcock	% fat by Mojonnier
1120	3.6	3.73
	3.6	3.71
1121	3.6	3.67
	3.6	3.67
1122	6.1	6.13
	6.1	6.12
1123	3.6	3.57
	3.6	3.59
1124	7.7	7.67
	7.7	7.65
1125	4.6	4.57
	4.6	4.59

7. Add glymol and read to the nearest half division on the test bottle.

Procedure for low fat mixes.

The procedure is the same as for medium and high fat mixes except:

1. Use thirty-five ml. of acid mixture instead of thirty-three ml.

2. Use ten minutes in the bath (206° to 208°F.) instead of five minutes.

The results presented are plain or vanilla flavored mixes only.

Using the procedures given produced clear tests that checked closely with results obtained with the Mojonnier method. It is important, however, to adhere closely to the specific details of each test

in order to obtain satisfactory results. This is particularly true of the proportion of the two acids and the amount of the mixture of acids used, as well as the temperature of the bath water.

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