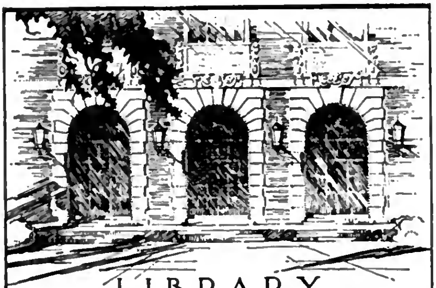

A Nonacid Babcock Method for Determining Fat in Ice Cream

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CONTENTS

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A Nonacid Babcock Method for Determining Fat in Ice Cream

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AN ACCURATE and rapid method for the determination of fat in ice cream is necessary in order quickly to standardize and to control ice-cream mixes. Most ice-cream plants have the regular Babcock equipment for testing milk and cream. An accurate and inexpensive method which will utilize the regular Babcock testing apparatus will be invaluable in the ice-cream industry.

This bulletin describes an attempt to devise a method for the determination of fat in ice cream in which the Babcock apparatus can be used and which it is believed will prove more satisfactory than the rapid methods now in use.

Review of Literature

Many procedures have been developed for the determination of fat in ice cream. Some of these have used Babcock apparatus. Most of the methods have depended on the action of an acid or a mixture of acids for the liberation of the fat. The most serious objection to the use of acid is that it reacts with the cane sugar to form a fluffy, charred material which rises into the neck of the test bottle and interferes with the reading of the fat column. This is especially true of sulfuric acid. Attempts have been made to use acids other than sulfuric, but the results obtained have not been very satisfactory. Probably the most successful of the acid methods have been those in which sulfuric acid was used in mixture with some other acid or acids. Certain alcohols, also, have been used with the acids. Some of these methods have given fair results with plain ice cream but have been unsatisfactory when used on fruit and chocolate ice creams.

There are a number of modifications of the Babcock method for the determination of fat in ice cream. One of the early methods used a mixture of equal parts of hydrochloric and acetic acids. White[25*] reported a method in which he used sulfuric acid after diluting the ice cream with water to three times its volume. The methods developed by Grigsby, Brinsmaid (both reported by Hortvet[7*]), Utt,[23*] Ross,[18*] Lichtenberg,[12*] and Halverson[5*] used mixtures of sulfuric and acetic acids. Halverson's method required a special bottle with a stopcock at the bottom for drawing off the sugar solution. Francis and Morgan[3*] modified the sulfuric-acetic acid method by adding a few drops of nitric acid. This method seems to give very good results with plain and vanilla ice creams. Le Cointe[10*] reported a method in which a small amount of amyl alcohol was added to the mixture of ice cream and sulfuric acid. In another method,[2*] used rather extensively, ethyl alcohol was mixed with the ice cream previous to the addition of sulfuric acid. It is doubtful if any of these methods have proved wholly successful on all kinds of ice cream.

Reagents other than acid have been used for the rapid determination of fat in milk, cream, and other dairy products. In 1888 Short[19*] reported a method in which two solutions were used. No. 1 was composed of 250 grams of caustic soda, 300 grams of caustic potash, and 1,809 grams of water. No. 2 was a mixture of equal parts of commercial sulfuric and acetic acids.

Thörner,[22*] in 1892, reported a method for the determination of fat in dairy products in which either an aqueous or an alcoholic potash solution could be used. Treatment of the milk with this solution was followed by treatment with acetic acid.

In the Nahm[15*] milk test, reported in 1894, milk was boiled with a mixture composed of potassium hydroxid, ethyl alcohol, amyl alcohol, and 30-percent ammonia solution, and the fat was read off on a scale.

In the lactanalyt[6*] test, milk was warmed with an alkaline solution, alcohol, and ether.

Lézé,[11*] in 1899, reported a method in which a mixture of caustic potash, ammonia, ethyl alcohol, and amyl alcohol were used.

The Sichler[20, 26*] sinacid method, first reported in 1903, which is patented and has been modified many times, has been used for testing milk and cream. Trisodium phosphate and sodium citrate were first used in this method, but these chemicals were replaced with sodium hydroxid, sodium sulfate, and sodium potassium tartrate (Rochelle salt). Isobutyl alcohol was used.

Gerber[4, 21*] developed and reported a method in 1906 known as the Gerber "Sal" method. Milk was mixed in a butyrometer with a solution consisting of sodium chlorid, sodium hydroxid, sodium potassium tartrate, and a small amount of coloring matter. Isobutyl alcohol was added. The mixture, after being heated in a water bath, was centrifuged and the fat read.

Wendler,[24*] in 1910, patented a nonacid method which was known as the "Neusal" method. A mixture of neutral salts consisting of sodium citrate and sodium salicylate with a small amount of methylene blue was used. Isobutyl alcohol was added separately.

In 1915 Pinkhoff[17]* reported a method for the determination of fat in milk without the use of sulfuric acid. Milk was warmed with amyl alcohol and a solution composed of sodium hydroxid, sodium phosphate, sodium citrate, and sodium chlorid. The butyrometers were centrifuged and the fat read.

Miwa[14]* patented a method in 1918 in which a mixture of isobutyl alcohol, a benzoate, a tartrate, a citrate, ethyl alcohol, a dye soluble in fats, and a water soluble dye was used.

Hoyberg[8, 9]* first reported his method in 1920, but it has been modified several times since. In this method milk was heated in a butyrometer with isobutyl alcohol and a solution of sodium hydroxid and sodium potassium tartrate. No centrifuge was used.

In 1920 Oshida[16]* reported a method in which the reagent consisted of 3.5 cc. ethyl alcohol, 5.6 cc. water, 3.5 cc. amyl alcohol, 1.0 cc. glycerol, .005 gram methylene blue, and .0025 gram sudan III.

Maghano and Porzio,[13]* in 1927, modified Hoyberg's method by using Fehling's alkali solution (60 grams sodium hydroxid, 173 grams sodium potassium tartrate, made up to 500 cc. with water) and a mixture composed of 45 parts methyl alcohol and 55 parts isobutyl alcohol.

In 1914 Bradbury[1]* adapted the Short method to the determination of fat in ice cream and condensed milk.

The Wendler method (reported by Hortvet[7]*) was tried on ice cream but did not seem to give consistent results. According to the directions for this method a 9-gram sample in a Babcock milk-test bottle was warmed with 19 cc. of a solution composed of 125 grams sodium hydroxid, 25 grams sodium potassium tartrate, and 25 grams sodium chlorid made up to 500 cc. with water. Ten to twelve drops of isobutyl alcohol were added to the mixture of ice cream and reagent.

Plan of the Investigation

In the present investigation a study of the acid methods was not attempted. A search was made for some nonacid reagent or reagents which could be used to replace acids. Alkali and salt solutions of varying concentrations and combinations were studied. The effects of various organic solvents with these solutions were also studied.

The general procedure followed in studying the different solutions was as follows:

A solution of the desired composition was prepared.

TABLE 1.—COMPOSITION OF SOLUTIONS GROUPED ACCORDING TO EFFICIENCY IN LIBERATING THE FAT FROM ICE-CREAM MIX

Sol.	Misc.	Trisod. phos.	Sod. ammon. phos.	Sod. pot. tartrate	Sod. citrate	Sod. acetate	Sod. salicyl.	Water	Ammon. hydrox.
Group 1—Solutions which failed to liberate the fat									
	grams	*grams*	*grams*	*grams*	*grams*	*grams*	*grams*	*cc.*	*cc.*
1	90 NaOH	...	...	...	...	150	...	Made up to 1 000	...
2	80 NaOH	...	...	200	...	...	...	"	...
3	....	200	...	...	...	...	...	"	...
4	....	150	...	...	...	...	...	"	...
5	....	100	...	...	...	...	...	"	...
6	300 Na_2HPO_4	...	...	...	...	...	...	"	...
7	....	...	100	...	200	...	...	1 000	...
8	....	...	100	...	200	...	...	700	300
9	....	...	200	...	...	200	...	1 000	...
10	....	...	200	200	...	...	...	1 000	...
11	....	...	100	...	100	200	...	1 000	...
12	....	...	100	100	...	200	...	1 000	...
13	....	100	100	200	...	...	...	1 000	...
14	....	100	100	200	...	...	...	700	300
15	....	...	100	200	100	...	...	1 000	...
16	....	...	100	200	100	...	...	700	300
17	....	...	100	200	...	100	...	1 000	...
18	....	...	100	200	...	100	...	700	300
19	....	200	...	200	...	...	...	1 000	...
20	....	200	...	200	...	...	...	700	300
21	....	...	...	200	200	...	...	700	300
22	....	...	...	200	...	200	...	700	300
23	....	...	...	...	...	400	...	1 000	...
24	....	...	...	...	...	400	...	700	300
25	....	...	...	400	...	...	...	700	300
Group 2—Solutions which liberated an appreciable amount of fat									
26	90 NaOH	...	...	150	...	...	...	Made up to 1 000	...
27	90 NaOH 20 Na_2SO_4	...	...	150	...	...	...	"	...
28	80 NaOH	...	...	140	...	...	...	"	...
29	70 NaOH	...	...	130	...	...	...	"	...
30	60 NaOH	...	...	120	...	...	...	"	...
31	50 NaOH	...	...	110	...	...	...	"	...
32	80 NaOH	...	...	120	...	...	...	"	...
33	70 NaOH	...	...	100	...	...	...	"	...
34	60 NaOH	...	...	80	...	...	...	"	...
35	50 NaOH	...	...	70	...	...	...	"	...
36	100 NaOH	...	...	170	...	...	...	"	...
37	90 NaOH 150 Borax	...	...	...	...	...	...	"	...
38	90 NaOH	150	...	...	...	...	...	"	...
39	100 NaOH 170 Borax	...	...	...	...	...	...	"	...
40	110 NaOH 200 Borax	...	...	...	...	...	...	"	...
41	.2 Methylene blue	300	...	...	80	...	80	"	...
42	.2 Methylene blue	...	...	...	100	...	100	"	...
43	....	500	...	...	...	...	...	"	...
44	....	400	...	...	...	...	200	"	...
45	....	200	...	...	...	...	200	.....	1 000
46	....	200	200	...	...	...	...	1 000	...
47	....	200	200	...	...	...	...	700	300
48	....	...	200	...	200	...	...	1 000	...
49	....	...	200	...	200	...	...	700	300
50	....	100	200	...	100	...	...	1 000	...
51	....	100	200	...	100	...	...	700	300
52	....	...	200	...	100	...	100	700	300
53	....	200	...	...	200	...	...	1 000	...
54	....	200	100	...	100	...	...	1 000	...
55	....	200	100	...	100	...	...	700	300
56	....	...	...	...	150	...	150	1 000	...
57	....	...	100	...	200	...	100	1 000	...
58	....	...	100	...	200	...	100	700	300
59	....	100	100	...	...	...	200	1 000	...

TABLE 1.—*Continued*

Sol.	Misc.	Trisod. phos.	Sod. ammon. phos.	Sod. pot. tartrate	Sod. citrate	Sod. acetate	Sod. salicyl.	Water	Ammon. hydrox.
	grams	*grams*	*grams*	*grams*	*grams*	*grams*	*grams*	*cc.*	*cc.*
60	....	100	100	...	...	...	200	700	300
61	....	...	200	...	...	200	...	700	300
62	....	...	200	200	...	...	...	700	300
63	....	100	100	...	...	200	...	1 000	...
64	....	100	100	...	...	200	...	700	300
65	....	...	100	...	100	200	...	700	300
66	....	...	100	100	...	200	...	700	300
67	....	100	...	...	100	200	...	1 000	...
68	....	100	...	...	100	200	...	700	300
69	....	100	...	...	...	200	100	1 000	...
70	....	100	...	100	...	200	...	1 000	...
71	....	100	...	100	...	200	...	700	300
72	....	...	100	200	...	...	100	700	300
73	....	100	...	200	100	...	...	1 000	...
74	....	100	...	200	100	...	...	700	300
75	....	100	...	200	...	100	...	1 000	...
76	....	100	...	200	...	100	...	700	300
77[1]	....	Variable	...	...	...	Variable	...	1 000	...
78	....	...	...	...	200	200	...	1 000	...
79	....	...	...	...	200	200	...	700	300
80	....	...	...	200	200	...	...	1 000	...
81	....	...	...	...	...	200	200	700	300
82	....	...	...	...	...	175	175	1 000	...
83	....	...	...	...	...	150	150	1 000	...
84	....	...	...	...	...	100	100	1 000	...
85	....	...	...	200	...	...	200	700	300
86	....	...	...	175	...	...	175	1 000	...
87	....	...	...	150	...	...	150	1 000	...
88	....	...	...	100	...	...	100	1 000	...
89	....	...	...	200	...	200	...	1 000	...
90	....	...	400	...	...	...	...	1 000	...
91	....	...	400	...	...	...	...	700	300
92	....	400	...	...	...	...	...	1 000	...
93	....	400	...	...	...	...	...	700	300
94	....	...	...	...	400	...	...	1 000	...
95	....	...	...	...	...	...	400	700	300
96	....	...	...	400	...	...	...	1 000	...
97[2]	....	Variable	...	...	...	Variable	Variable	Variable	Variable
Group 3—Solutions which appeared to liberate all the fat									
98	....	200	...	...	...	...	100	Made up to 1 000	...
99	....	...	...	...	100	...	200	"	...
100	....	150	...	...	50	...	150	"	...
101	....	200	...	...	...	...	100	700	300
102	....	300	...	...	...	...	200	Made up to 1 000	...
103	....	...	200	...	...	...	200	1 000	...
104	....	...	200	...	...	...	200	700	300
105	....	100	200	...	...	...	100	1 000	...
106	....	100	200	...	...	...	100	700	300
107	....	...	200	...	100	...	100	1 000	...
108	....	100	100	...	100	...	100	1 000	...
109	....	100	100	...	100	...	100	700	300
110	....	200	...	...	200	...	...	700	300
111[3]	....	Variable	...	...	...	...	Variable	Variable	Variable
112	....	200	100	...	...	...	100	1 000	...
113	....	200	100	...	...	...	100	700	300
114	....	200	...	...	100	...	100	1 000	...
115	....	200	...	...	100	...	100	700	300
116	....	...	...	...	200	...	100	1 000	...
117	....	...	...	...	200	...	100	700	300
118	....	...	...	...	175	...	175	1 000	...
119	....	100	...	...	200	...	100	1 000	...
120	....	100	...	...	200	...	100	700	300
121	....	...	100	...	100	...	200	1 000	...
122	....	...	100	...	100	...	200	700	300
123	....	100	...	...	100	...	200	1 000	...
124	....	100	...	...	100	...	200	700	300
125	....	...	100	...	...	200	100	1 000	...
126	....	...	100	...	...	200	100	700	300
127	....	100	...	...	...	200	100	700	300
128	....	...	100	200	...	...	100	1 000	...

TABLE 1.—*Concluded*

Sol.	Misc.	Trisod. phos.	Sod. ammon. phos.	Sod. pot. tartrate	Sod. citrate	Sod. acetate	Sod. salicyl.	Water	Ammon. hydrox.
	grams	*grams*	*grams*	*grams*	*grams*	*grams*	*grams*	*cc.*	*cc.*
129	....	100	...	200	...	...	100	1 000	...
130	....	100	...	200	...	...	100	700	300
77[1]	....	Variable	...	...	...	Variable	...	1 000	...
131	....	200	...	...	...	200	...	700	300
132	....	...	...	...	...	200	200	1 000	...
133	....	...	...	200	...	...	200	1 000	...
134	....	...	...	...	400	...	...	700	300
135	....	...	...	...	...	...	400	1 000	...
136	....	150	...	100	...	100	...	1 000	...
97[2]	....	Variable	...	...	...	Variable	Variable	Variable	Variable

[1]There are 19 aqueous solutions of mixturea of trisodium phosphate and sodium acetate, the variation in the amoun t of each salt being from 50 to 200 grams per liter of water. Five of the solutions belong in Group 2, and 14 in Group 3.

[2]There are 61 aqueous and aqueous-ammoniacal solutions of a mixture of trisodium phosphate, sodium acetate, and sodium salicylate. The phosphate varies from 50 to 350 grams, the acetate from 25 to 200 grams, and the salicylate from 10 to 100 grams per liter of solvent. The amount of ammonium hydroxid in the aqueous-ammoniacal solutions varies from 25 to 300 cc. per liter of solvent. Eighteen of these solutiona belong in Group 2, and 43 in Group 3.

[3]There are 46 aqueous and aqueous-ammoniacal solutions of mixtures of trisodium phosphate and sodium salicylate. The phosphate varies from 150 to 400 grams and the salicylate from 50 to 200 grams per liter of solvent. The amount of ammonium hydroxid in the aqueous-ammoniacal solutions varies from 25 to 300 cc. per liter of solvent.

Nine grams of the prepared sample of ice cream were weighed into a Babcock milk-test bottle.

The solution and the organic solvent were added, the latter being measured exactly.

The mixture was placed in a shallow water bath heated with a jet of steam, and was shaken occasionally while heating.

The bottle was centrifuged and the fat column read as in the regular Babcock procedure for milk.

The Roese-Gottlieb method as given by the Association of Official Agricultural Chemists was used for comparison. Three extractions with ether were made in each case. Duplicate determinations were made on each sample.

Eleven chemical compounds—sodium hydroxid, sodium sulfate, borax, disodium phosphate, trisodium phosphate, sodium ammonium phosphate, sodium potassium tartrate, sodium citrate, sodium acetate, sodium salicylate, and ammonium hydroxid—were used in making up 261 solutions. Table 1 shows the number and composition of the solutions studied in this investigation.

Effects that ten different organic compounds had on the separation of the fat were studied. The compounds were n-butyl alcohol, isobutyl alcohol, acetone, toluol, chloroform, carbon tetrachlorid, amyl acetate, glycerol, amyl alcohol, and ethyl alcohol. In addition, seventeen various mixtures of acetone with amyl alcohol, n-butyl alcohol, or isobutyl alcohol were tried. Unsatisfactory results were obtained with acetone, toluol, chloroform, carbon tetrachlorid, amyl acetate, glycerol, and the acetone-alcohol mixtures. The butyl alcohols and amyl alcohol gave the greatest promise of producing good results.

Results of the Investigation

The efficiency of the salt solutions in liberating the fat from the other components of the ice cream is indicated by the groupings in Table 1. Group 1 includes solutions which failed to liberate any appreciable amount of fat. In most cases the ice cream was curdled. Solutions 1 to 25 inclusive fall in this group. Group 2 includes solutions which liberated a considerable portion of the fat but failed to

TABLE 2.—FAT CONTENT OF ICE-CREAM MIXES AS DETERMINED BY USE OF SOLUTIONS NUMBERED 77 CONTAINING BOTH TRISODIUM PHOSPHATE AND SODIUM ACETATE IN VARYING CONCENTRATION

(Fat in sample 13.61 percent determined by the Official method; .8 cc. n-butyl alcohol was used in each test)

Solution	Composition of solution[1]		Tests			Description
	Trisodium phosphate	Sodium acetate	Orig.	Dup.	Aver.	
	grams	*grams*	*perct.*	*perct.*	*perct.*	
77a	200	200	13.8	13.8	13.8	Clear fat column
77b	200	150	13.6	13.6	13.6	Clear fat column
77c	200	100	13.6	13.6	13.6	Very slightly curdy
77d	200	50	13.2	13.4	13.3	Slightly curdy
77e	175	100	13.6	13.6	13.6	Very slightly curdy
77f	175	50	13.6	13.8	13.7	Slightly curdy
77g	150	100	13.6	13.6	13.6	Very slightly curdy
77h	150	50	13.6	13.6	13.6	Slightly curdy
77i	150	200	13.2	13.6	13.4	Slightly curdy
77j	100	200	11.6	9.8	....	Curdy
77k	50	200	8.0	8.0	....	Curdy
77l	100	175	13.2	13.2	13.2	Slightly curdy
77m	50	175	11.2	11.8	....	Curdy
77n	100	150	13.2	13.4	13.3	Slightly curdy
77o	50	150	12.8	12.8	12.8	Curdy
77p	150	200	13.6	13.6	13.6	Very slightly curdy
77q	175	175	13.6	13.6	13.6	Clear fat column
77r	100	100	13.0	13.0	13.0	Slightly curdy

[1]One liter of distilled water was used in making each solution.

produce complete liberation. In most cases a considerable amount of undissolved curd appeared at the bottom of the fat column. Solutions 26 to 97 inclusive are in this group. Group 3 includes all solutions which appeared to liberate the fat completely. Very little undissolved curd appeared and the fat columns were, in most cases, clear and sharply divided from the aqueous layer. Solutions 98 to 136 inclusive fall in this group. This group also includes some of Solutions 77 and 97.

In Group 3 Solutions 77 (trisodium phosphate and sodium acetate), 97 (trisodium phosphate, sodium acetate, and sodium salicylate), and 111 (trisodium phosphate and sodium salicylate) seemed most promising. They were given a thoro trial on various kinds of ice creams and ice-cream mixes.

TABLE 3.—COMPARISON OF FAT PERCENTAGES OBTAINED WHEN TESTING ICE-CREAM SAMPLES WITH SOLUTIONS 111 AND 97
(.7 cc. n-butyl alcohol was used in each case)

Sample	Official fat	Solution 111 without ammonia				Solution 111 with ammonia				Solution 97 without ammonia				Solution 97 with ammonia			
		Orig.	Dup.	Aver.	Dif.	Orig.	Dup.	Aver.	Dif.	Orig.	Dup.	Aver.	Dif.	Orig.	Dup.	Aver.	Dif.
	perct.	perct.	perct.	perct.		perct.	perct.	perct.		perct.	perct.	perct.		perct.	perct.	perct.	
37	12.05	12.4	12.4	12.40	+ .35	12.2	12.2	12.20	+ .15	12.0	12.1	12.05	......	12.4	12.5	12.45	+ .40
38	12.34	12.6	12.6	12.60	+ .26	12.6	12.6	12.60	+ .26	11.9	12.1	12.00	− .34	12.6	12.5	12.55	+ .21
39	11.80	12.3	12.3	12.30	+ .50	12.4	12.5	12.45	+ .65	12.0	12.2	12.10	+ .30	12.3	12.4	12.35	+ .55
40	11.84	12.3	12.4	12.35	+ .51	11.9	12.0	11.95	+ .11	11.4	11.4	11.40	− .44	12.1	12.1	12.10	+ .26
41	11.71	12.1	12.1	12.10	+ .39	11.9	11.9	11.90	+ .19	11.4	11.4	11.40	− .31	12.1	12.1	12.10	+ .39
42	10.61	12.5	12.6	12.55	+1.94	12.5	12.4	12.45	+1.84	12.4	12.4	12.40	+1.79	12.5	12.6	12.55	+1.94
43	11.89	12.4	12.4	12.40	+ .51	12.3	12.3	12.30	+ .41	11.6	11.2	11.40	− .49	12.4	12.4	12.40	+ .51
44	11.17	11.7	11.7	11.70	+ .53	11.6	11.6	11.60	+ .43	9.8	10.8	10.30	− .87	11.5	11.5	11.50	+ .33
45	11.53	11.7	11.6	11.65	+ .12	11.6	11.6	11.60	+ .07	11.0	11.0	11.00	− .53	11.7	11.7	11.70	+ .17
46	11.93	12.1	12.1	12.10	+ .17	12.2	12.2	12.20	+ .27	12.3	12.5	12.40	+ .47	12.2	12.2	12.20	+ .27
47	11.75	12.0	11.9	11.95	+ .20	12.0	12.0	12.00	+ .25	12.5	12.5	12.50	+ .75	12.1	12.1	12.10	+ .35
48	11.85	12.4	12.4	12.40	+ .55	12.1	12.3	12.20	+ .35	12.4	12.6	12.50	+ .65	12.3	12.3	12.30	+ .45
49	11.89	11.9	11.9	11.90	+ .01	11.9	11.8	11.85	− .04	12.1	12.1	12.10	+ .21	12.0	12.0	12.00	+ .11
50	9.63	9.9	9.8	9.85	+ .22	9.9	9.8	9.85	+ .22	10.2	10.2	10.20	+ .47	9.9	9.9	9.90	+ .27
51	9.94	10.0	10.0	10.00	+ .06	10.1	10.1	10.10	+ .16	10.4	10.4	10.40	+ .56	10.3	10.2	10.25	+ .31
52	11.85	12.0	12.1	12.05	+ .20	12.0	12.1	12.05	+ .20	12.2	12.2	12.20	+ .35	12.2	12.2	12.20	+ .35
53	11.84	11.9	12.0	11.95	+ .11	12.0	12.0	12.00	+ .16	12.4	12.4	12.40	+ .56	12.2	12.2	12.20	+ .36
54	11.21	10.4	10.8	10.60	− .61	11.5	11.4	11.45	+ .24	11.8	11.8	11.80	+ .59	11.6	11.5	11.55	+ .34
55	12.06	12.6	12.6	12.60	+ .54	12.2	12.2	12.20	+ .14	12.8	12.8	12.80	+ .74	12.2	12.1	12.15	+ .09
56	11.70	12.0	12.0	12.00	+ .30	11.7	11.7	11.70	......	12.2	12.2	12.20	+ .50	11.8	11.8	11.80	+ .10
Average..	11.5295	....	....	11.8725	+ .343	....	....	11.8325	+ .303	....	....	11.7775	+ .248	....	....	11.9175	+ .388

Table 2 shows the composition of the various solutions of trisodium phosphate and sodium acetate and the results obtained on a sample of unfrozen plain mix. The solution containing 200 grams trisodium phosphate and 150 grams sodium acetate (77b) was chosen as the best of the lot because it seemed as efficient in liberating the fat as the more highly concentrated solutions and it is more economical because of the smaller amount of the more expensive salt—sodium acetate.

Ammonium hydroxid was not added directly to the salt solution since it causes a large part of the salts to crystallize. However, ammonia seems to be necessary for the test. A second solution was made of 75 parts ammonium hydroxid, 35 parts n-butyl alcohol, and 15 parts ethyl alcohol by volume. Ammonium hydroxid and n-butyl alcohol are not miscible in these proportions, but on the addition of the ethyl alcohol a homogeneous solution is formed. A 2.5 cc. portion of this solution was used in each test.

A comparison between Solution 111 (200 grams trisodium phosphate and 200 grams sodium salicylate) with and without ammonia and Solution 97 (150 grams trisodium phosphate, 100 grams sodium acetate, and 25 grams sodium salicylate) with and without ammonia is shown in Table 3. In this comparison .7 cc. n-butyl alcohol was used in each determination.

A comparison between Solution 111 with ammonia and Solution 77b (200 grams trisodium phosphate and 150 grams sodium acetate) is shown in Table 4. In this comparison .7 cc. of n-butyl alcohol was used with Solution 111, while 2.5 cc. of the alcohol-ammonia mixture were used with Solution 77b.

Solution 77b was finally chosen as the best because it seems to give results most nearly approaching the correct values, does not change on long standing, remains in solution at ordinary temperatures, and is less expensive in that sodium acetate is a cheaper chemical than sodium salicylate.

The effect of the length of time of heating in the water bath before centrifuging is shown in Table 5. Fifteen minutes seems sufficient for most of the plain ice creams, but a longer time is usually required for the fruit and especially the chocolate ice creams.

Denatured grain alcohol may be used instead of 95-percent ethyl alcohol in making up the alcohol-ammonia mixture. Ethyl alcohol was denatured according to formulas 1, 2, 3, 4, 5, and 7 given in "Dena-

TABLE 4.—COMPARISON OF FAT PERCENTAGES OBTAINED WHEN TESTING ICE-CREAM SAMPLES WITH SOLUTIONS 111 AND 77b

(.7 cc. n-butyl alcohol was used with Solution 111 and 2.5 cc. of the alcohol-ammonia mixture were used with Solution 77b)

Sample	Official fat	Solution 111				Solution 77b			
		Orig.	Dup.	Aver.	Dif.	Orig.	Dup.	Aver.	Dif.
	perct.	*perct.*	*perct.*	*perct.*		*perct.*	*perct.*	*perct.*	
64	8.94	8.6	9.0	8.80	− .14	9.0	9.0	9.00	+ .06
65	9.96	9.8	10.2	10.00	+ .04	10.0	10.0	10.00	+ .04
66	10.84	11.0	11.0	11.00	+ .16	10.9	10.9	10.90	+ .06
67	11.92	12.3	12.0	12.15	+ .23	12.1	12.0	12.05	+ .13
68	13.25	13.8	13.4	13.60	+ .35	13.4	13.2	13.30	+ .05
69	15.13	15.2	15.4	15.30	+ .17	15.2	15.2	15.20	+ .07
70	16.27	16.4	15.6	16.00	− .27	16.4	16.4	16.40	+ .13
71	18.01	18.0	18.0	18.00	− .01	18.1	18.1	18.10	+ .09
72	19.02	19.2	19.2	19.20	+ .18	19.1	19.1	19.10	+ .08
73	23.44	23.6	23.6	23.60	+ .16	23.8	23.6	23.70	+ .26
74	11.20	10.4	10.4	10.40	− .80	11.0	11.0	11.00	− .20
75	11.83	11.8	12.0	11.90	+ .07	11.8	11.8	11.80	− .03
76	12.34	12.5	12.6	12.55	+ .21	12.5	12.6	12.55	+ .21
77	12.41	12.4	12.5	12.45	+ .04	12.6	12.6	12.60	+ .19
78	10.21	10.4	10.4	10.40	+ .19	10.4	10.5	10.45	+ .24
79	10.67	10.6	10.7	10.65	− .02	10.7	10.7	10.70	+ .03
80	11.10	11.0	11.2	11.10	......	11.2	11.2	11.20	+ .10
82	2.32	.8	.9	.85	−1.47	2.3	2.3	2.30	− .02
83	13.83	13.6	13.7	13.65	− .18	13.8	13.8	13.80	− .03
84	11.08	10.4	10.6	10.50	− .58	11.1	11.1	11.10	+ .02
85	10.29	10.0	10.0	10.00	− .29	10.4	10.3	10.35	+ .06
86	9.09	9.1	9.0	9.05	− .04	9.1	9.2	9.15	+ .06
87	10.70	10.6	10.5	10.55	− .15	10.8	10.8	10.80	+ .10
88	11.84	11.4	11.4	11.40	− .44	11.8	11.9	11.85	+ .01
89	10.28	10.4	10.5	10.45	+ .17	10.4	10.4	10.40	+ .12
90	14.08	14.0	13.9	13.95	− .13	14.2	14.2	14.20	+ .12
Average..	12.3096	....	....	12.2115	− .0981	....	....	12.3846	+ .0750

TABLE 5.—RESULTS OBTAINED ON SAMPLE OF ICE CREAM BY NONACID METHOD WHEN TIME OF HEATING VARIED FROM 10 TO 90 MINUTES

(Fat in sample 14.31 percent determined by the Official method)

Heating time	Original test	Duplicate	Average	Difference
minutes	*perct.*	*perct.*	*perct.*	
10	14.0	14.0	14.00	− .31
15	14.4	14.4	14.40	+ .09
20	14.4	14.4	14.40	+ .09
25	14.4	14.3	14.35	+ .04
30	14.4	14.4	14.40	+ .09
40	14.4	14.4	14.40	+ .09
50	14.4	14.4	14.40	+ .09
60	14.5	14.6	14.55	+ .24
70	14.6	14.6	14.60	+ .29
80	14.6	14.6	14.60	+ .29
90	14.6	14.4	14.50	+ .19

turization of Industrial Alcohol," Senate Document No. 195, 69th Congress, Second Session, Appendix to Regulations No. 61, Completely Denatured Alcohol Formulae.

Alcohol-ammonia mixtures were made up with the various denatured alcohols. The results obtained on an ice-cream mix are shown in Table 6. The denaturants seem to have no harmful effect on the

Table 6.—Results Obtained on Sample of Ice Cream by Nonacid Method When Denatured Grain Alcohol Was Used in the Alcohol-Ammonia Mixture

(Fat in sample 13.58 percent determined by Official method)

Denaturing formula No.	Original test	Duplicate	Average
	perct.	*perct.*	*perct.*
1	13.6	13.6	13.60
2	13.6	13.7	13.65
3	13.6	13.8	13.70
4	13.6	13.6	13.60
5	13.7	13.9	13.80
7	13.6	13.7	13.65
95% alcohol	13.6	13.7	13.65

method. The denatured alcohol found on the retail market is usually prepared by one of these formulas and should prove quite satisfactory for use in this test.

Procedure for Nonacid Method

The method as finally adopted requires two reagents. Reagent A, the alcohol-ammonia mixture, is made up as follows:

75 cc. of C.P. ammonium hydroxid
35 cc. of n-butyl alcohol
15 cc. of 95-percent ethyl alcohol or denatured grain alcohol

This reagent should be kept in a glass-stoppered bottle.

Reagent B is made up as follows:

200 grams of trisodium phosphate
150 grams of sodium acetate
1 liter of water

Trisodium phosphate and sodium acetate of commercial grade are satisfactory. Clean tap water may be used instead of distilled water. The reagent remains in solution at ordinary temperatures but will partially crystallize on becoming cold. If crystallization takes place the crystals should be dissolved by warming before the reagent is used. The reagent does not seem to deteriorate on long standing.

In addition to the regular Babcock equipment a shallow water bath provided with some means for heating and a burette graduated to .5 cc. are necessary. A 2.5-cc. pipette may be used instead of the burette. The heat may be supplied by a gas burner or a jet of steam.

As a result of this investigation, the following procedure is presented for the determination of fat in ice cream:

1. Weigh 9 grams of the well-mixed sample of ice cream into a Babcock milk-test bottle (8- or 10-percent) or a 20-percent ice-cream test bottle.
2. Add exactly 2.5 cc. of Reagent A from a burette or pipette. Mix thoroly.
3. Add 9 to 10 cc. of Reagent B and again mix thoroly. The reagent may be measured in an ordinary 9-cc. acid measure or in a 10-cc. pipette.
4. Place the test bottle in a shallow water bath and heat the bath to boiling,

TABLE 7.—PERCENTAGE OF FAT IN 54 SAMPLES OF ICE CREAM AS DETERMINED BY OFFICIAL METHOD AND BY NONACID BABCOCK METHOD

Sample	Kind of ice cream	Official method	Nonacid method				
			Original	Duplicate	Average	Difference	
	Samples from University of Illinois Creamery						
		perct.	*perct.*	*perct.*	*perct.*	(+)	(−)
64	Plain mix	8.940	9.0	9.0	9.00	.060	....
65	Plain mix	9.956	10.0	10.0	10.00	.044	....
66	Plain mix	10.840	10.9	10.9	10.90	.060	....
67	Plain mix	11.920	12.1	12.0	12.05	.130	....
68	Plain mix	13.245	13.4	13.2	13.30	.055	....
69	Plain mix	15.125	15.2	15.2	15.20	.075	....
70	Plain mix	16.270	16.4	16.4	16.40	.130	....
71	Plain mix	18.010	18.1	18.1	18.10	.090	....
72	Plain mix	19.020	19.1	19.1	19.10	.080	....
73	Plain mix	23.440	23.8	23.6	23.70	.260	....
74	Chocolate	11.205	11.0	11.0	11.00	....	.205
75	Vanilla	11.830	11.8	11.8	11.80	....	.030
76	Caramel	12.335	12.5	12.6	12.55	.215	....
77	Pistachio nut	12.405	12.6	12.6	12.60	.195	....
78	Fruit	10.205	10.4	10.5	10.45	.245	....
79	Fruit	10.665	10.7	10.7	10.70	.035	....
80	Fruit	11.100	11.2	11.2	11.20	.100	....
81	Plain mix	14.305	14.4	14.4	14.40	.095	....
82	Fruit sherbet	2.320	2.3	2.3	2.30	....	.020
83	Vanilla	13.830	13.8	13.8	13.80	....	.030
84	Chocolate	11.080	11.1	11.1	11.10	.020	....
85	Chocolate	10.290	10.4	10.3	10.35	.060	....
86	Chocolate	9.090	9.1	9.2	9.15	.060	....
87	Fruit	10.700	10.8	10.8	10.80	.100	....
88	Vanilla	11.840	11.8	11.9	11.85	.010	....
89	Fruit	10.280	10.4	10.4	10.40	.120	....
90	Vanilla	14.075	14.2	14.2	14.20	.125	....
91	Vanilla	13.300	13.2	13.3	13.25	....	.050
92	Vanilla	13.525	13.6	13.5	13.55	.025	....
93	Vanilla	13.935	14.1	14.1	14.10	.165	....
94	Vanilla	13.815	13.8	13.9	13.85	.035	....
1	Fruit	11.095	11.2	11.2	11.20	.105	....
2	Vanilla	13.580	13.8	13.9	13.85	.270	....
3	Mint	13.860	14.1	14.0	14.05	.190	....
4	Chocolate	12.415	12.3	12.3	12.30	....	.115
5	Fruit	10.935	11.0	11.0	11.00	.065	....
6	Vanilla	11.645	11.8	11.7	11.75	.105	....
7	Fruit	12.065	12.0	12.0	12.00	....	.065
8	Chocolate	11.780	11.6	11.6	11.60	....	.180
9	Fruit	12.190	12.2	12.2	12.20	.010	....
13	Fruit	12.910	13.0	13.0	13.00	.090	....
14	Chocolate	11.440	11.4	11.4	11.40	....	.040
15	Chocolate	11.270	11.2	11.3	11.25	....	.020
16	Fruit	8.285	8.4	8.5	8.45	.165	....
23	Vanilla	13.580	13.6	13.7	13.65	.070	....
	Samples from commercial ice-cream plants[1]						
10	Vanilla	11.435	11.4	11.4	11.40	....	.035
11	Chocolate	11.085	11.3	11.3	11.30	.215	....
12	Fruit	10.060	10.2	10.3	10.25	.190	....
17	Vanilla	10.770	10.8	10.8	10.80	.030	....
18	Fruit	9.705	9.8	9.8	9.80	.095	....
19	Chocolate	9.150	9.2	9.2	9.20	.050	....
20	Vanilla	14.935	15.0	15.0	15.00	.065	....
21	Fruit	14.225	14.3	14.3	14.30	.075	....
22	Chocolate	12.695	12.7	12.8	12.75	.055	....
	Summary of results from all samples[2]						
Average of 54 samples		12.2223	....	....	12.2898	.0675	....
Average of 25 plain mix and vanilla samples		13.7267	....	....	13.8000	.0733	....
Average of 15 fruit samples		10.4493	....	....	10.5367	.0873	....
Average of 11 chocolate samples		11.0455	....	....	11.0364	.....	.0091

[1]The authors wish to express their appreciation to the following named companies for the courtesy shown by them in furnishing samples: Snow and Palmer, Bloomington; John T. Cunningham Ice Cream Co., Chicago; and J. D. Rossell Co., Peoria. Many samples used in the development of the method were obtained from the Champaign Ice Cream Co., Champaign, Illinois. [2]Samples 76, 77, and 3 are not classified.

continuing the heating for several minutes. Shake the contents of the bottle two or three times while heating.

5. Usually at the end of 15 to 30 minutes the fat will separate and form a clear yellow layer on top of the liquid. The heating must not cease until the fat layer has definitely separated from the dark portion of the liquid and has become clear.
6. After all the fat has separated, place the test bottle in the centrifuge and whirl 5-2-1 minutes, adding hot water as in the regular Babcock milk test except that the water *must not* be softened with acid.
7. Place the bottle in a hot-water bath at 130°-140° F. for 5 minutes.
8. Read the test, measuring from the bottom of the fat column to the top of the upper meniscus.
9. If an 8- or 10-percent milk-test bottle is used, multiply the reading by two.

Nonacid Method Compared With Official Method

Determinations of fat by the method just described were made on fifty-four samples of ice cream and the results compared with those obtained by using the Official Roese-Gottlieb method (Table 7). Determined by the Official method, the fat in these samples varied from 2.32 percent in the case of a sherbet to 23.44 percent in the case of a high-fat ice cream. The solids-not-fat varied from approximately 8 to 14 percent. The samples included plain, unfrozen mixes and ice creams containing whole fruit, fruit sirups, various flavorings and extracts, caramel, nuts, mint candy, and chocolate. The whole fruits, nuts, and other solid particles were removed from the samples before testing by passing the liquid portion thru cheesecloth.

Of the results on fifty-four samples tested by the nonacid method, 64.82 percent came within ± .1 percent of the Roese-Gottlieb values, 24.07 percent fell between ± .1 and ± .2 percent, and 11.11 percent between ± .2 and ± .3 percent; that is, 88.89 percent of the tests showed a difference of less than ± .2 percent from the Roese-Gottlieb values while only 11.11 percent showed differences greater than ± .2 percent. The range of differences was from — .205 to + .270 percent. The average difference was + .0675 percent for all samples.

Conclusions and Observations

1. Butterfat can be separated quantitatively from ice cream without the use of sulfuric acid.

2. Babcock test bottles and other Babcock apparatus can be used in a volumetric method for determining fat by nonacid reagents.

3. The method presented is fairly rapid, sufficiently accurate for commercial use, and is inexpensive.

4. The operator must become completely familiar with the method in order to achieve success with it.

5. A large number of determinations may be made concurrently.

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