Minor Thesis in Dairy Chemistry.

Submitted by

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In Application for

Master of Science Degree.

Subject.

The Detection, Chemically, of Condensed Milk in Ice Cream.

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THE DETECTION, CHEMICALLY, OF CONDENSED MILK IN ICE CREAM.

Introduction:

This study was taken up at the suggestion of the Pure Food Department of Virginia, and a preliminary report containing a suggestion of the method most likely to give results was submitted by one of the chemists of that Department.

The proposed method was based on the theory that the process of condensing milk causes a portion of the fat to resist all methods of extraction in general use; that not only do the usual methods of ether extraction fail to recover all the fat from condensed milk, but that results are the same with the Babcock Test and all its modifications. Using this theory as a basis, a method of procedure was submitted for the complete recovery of the fat, and it was concluded that the difference in results obtained by it and by one of the old methods would be evidence of the presence of condensed milk.

Principle of Procedure:

In dealing with this problem it was necessary first, to establish as a basis on which to work, some difference between the composition or properties of condensed milk and the substances with which it is mixed to make ice cream, - secondly, to show that the differences when found was a definite one under all conditions, - thirdly, the difference must be of such a character and magnitude that it can be used as conclusive evidence. With these two essentials in mind a review of the work of chemists in comparing the different methods of fat extraction will be made, then other phases of the problem will be taken up.

Review of Work on Fat Determination in Condensed Milk:

In 1905 G. E. Patrick reported as follows:[1] "The difficulty of extracting fat from sweetened condensed milk by the usual method was quite well known, Geisler having investigated the subject and found that very good results could be obtained by using only 1 gram of condensed milk with 3 or 4 grams of water, drying, and extracting for a very long time." G.E.Patrick further stated that in his opinion one of the methods in which the fat is set free by an acid is preferable to the ether extraction method for analysis of condensed milk.

Previous to this,[2] Leach, of Massachusetts, used a modification of the Babcock method for sweetened condensed milk, and[3] Farrington published another method later.

Leech's modification is as follows:

Preparation of Sample: Mix thoroughly by transferring the contents of the can to a large evaporating dish and working it with a pestle until homogenous. Weigh 40 grams of the mixed sample in a 100 c. c. sugar flask and transfer thereto by washing, and make up to the mark with water.

Fat.[4]- Measure 15 c.c. of the above 40% solution, corresponding to 6 grams of the condensed milk, into a Babcock text bottle. Fill nearly to the neck with water, add 4 c.c. of Fehling's Copper solu-

1. Rpt. A.O.A.C., 1905.
2. Food Inspection
3. Wisconsin Seventeeth Annual Report 1900, P.86.
4. Leach, Jour. Am. Chem. Soc. 1900.

tion and shake thoroughly and rapidly, separating the precipitated proteids and fat by means of a centrifuge,[1] or the precipitate may be allowed to settle of itself, which it does more quickly in the cold. Withdraw the supernatant sugar containing liquid by means of a small stemmed pipette with a wisp of wet absorbent cotton twisted over the bottom to serve as a filter. Wipe off the cotton into the bottle on withdrawing the pipette. Give the precipitated proteids and fat two additional washings as above by shaking with water, separating the precipitate and removing the washings with the pipette. If the precipitate is caked hard after centrifuging, use a stiff platinum wire as a stirrer. Finally, add water to an approximate volume of 17.5 c. c. and 17.5 c. c. of H_2SO_4, and continue test as in Babcock method, multiplying the reading by 3 for the percentage.

The Farrington Test is as Follows: From 40 to 60 grams of condensed milk are weighed into a 200 c.c. graduated flask; about 100 c. c. of water are added and the solution of the condensed milk effected. The flask is then filled to the mark with water and after mixing thoroughly 17.5 c. c. is measured into a Babcock test bottle. About 3 c.c. of the sulphuric acid commonly used are then added and the milk and acid mixed by shaking vigorously. The milk is curdled by the acid, and the curd and whey separated somewhat. In order to make this separation complete and to compact the curd into a firm lump, the test bottle is whirled for about six minutes at a rather high speed (1000 rev.) in a steam turbine centrifuge.

The chamber in the tester should be heated to about 200° F. This can be done either by the exhaust steam or a valve and pipe. After

1. A steam driven centrifuge may be used but it is better to centrifuge in the cold to prevent caking of precipitate.

whirling, the bottles are taken out, and by being careful not to break the lump of curd, nearly all the whey or sugar solution can be poured out of the neck. Ten c. c. of water are then poured into the test bottle and the curd is shaken up with it so as to wash out more of the sugar. Three c. c. of acid are now added as before, and the test bottle whirled a second time in the centrifuge. The whey is decanted again and the second washing removes so much of the sugar that what remains will not interfere with the test. The curd remaining in the test bottle after the second washing is shaken up with ten c. c. of water, and to this water emulsion of the curd the usual amount, 17.5 c. c. sulphuric acid is added and the test completed in the usual way. The amount of fat finally obtained is calculated to the weight of condensed milk taken.

When 60 grams of condensed milk are used in a 200 c. c. flask, each 17.6 c. c. of the 200 c.c. is equal to 5.28 gr. of the sample tested.

The Gottlieb Method[1] is now the official method in Sweden and Denmark for the determination of fat in skim milk and buttermilk, recent work by European chemists having shown that the ordinary extraction methods give too low results for milks low in fat. The Gottlieb Method is as follows:

Ten C. c. of milk are measured into a glass cylinder three fourths inch in diameter and about 14 inches long (a 100 c.c. burette or a eudiometer tube will do); one c. c. concentrated ammonia is added and mixed thoroughly with the milk; the following chemicals are next

[1]Landw. Versuchs.-Stat.,1892, 40.

added in the order given; 10 c. c. of 92 per cent alcohol, 25 c. c. of washed ether, and 25 c. c. of petroleum ether (boiling point below 80° C), the cylinder being closed with a moistened cork stopper and the contents shaken several times after the addition of each. The cylinder is then left standing for six hours or more. The clear fat solution is next pipetted off into a small weighed flask by meansoof a siphon drawn to a fine point, which is lowered into the fat solution to within 0.5 c. m. of the turbid bottom layer. After evaporating the ether solution in a hood, the flasks are dried in a steam oven for two or three hours and weighed.

In the case of products high in fat a second extraction with 10 c. c. of ether and petroleum is advisable in order to recover the last traces of fat.

F. H. Woll[1] submits reports of work done by seven different chemists on condensed milk. The Babcock centrifugal method as modified by Leach, that of Farrington, the Babcock asbestos extraction method and the Gottleib method were used.

The following table gives the results for fat by those chemists.

[1] Rpt. A.O.A.C. 1906.

Table 1.

Percentage of Fat in Condensed Milk Samples.[1]

Analyst.	Asbestos Method			Babcock Test.		Gottleib
	Extraction 1	Ex. 2	Total	Leach	Farrington	
Sample A. Sweetened Condensed Milk.						
Smith	2.25	6.09	8.34			7.97
Whittier	2.43	5.82	8.25			
Fulton				9.0	8.58	
Norton				8.4		[a]4.70
Bartlett			8.76		8.7	8.81
Olson	3.18	5.08	8.26	9.08	8.8	8.13
Jaffa & Stewart			9.92	9.75		
Sample B. Unsweetened						
Smith	6.84	.45	7.29			7.12
Whittier	6.86	.42	7.28		[b]6.48	
Norton					[b]6.9	[a] 4.9
Bartlett			7.34		[b]5.93	7.03
Olson	7.20	.02	7.22	6.90	(7.7 ([b] 6.8	7.09
Patrick & Boyle			7.55			[a] 7.24
Other Samples.						
Olson	(7.24 (8.08	0.08 0.60	7.32 8.68			7.53 8.93
Jaffa & Stewart			7.20	6.70		

[1] A.O.A.C., 1906.

Table 2.

Averages of comparative determinations.

Method.	Sweetened.	Unsweetened.
By extraction method	d 8.61	d 7.55
Gottleib method	d 8.59	d 7.54
By extraction method.	e 9.29	f 7.20
Babcock test Leach modification	e 9.38	f 6.80
By extraction method	f 8.51	e 7.28
Babcock Test (original)	fg 8.75	e 6.40

a. Extracted once.

b. Babcock original method.

c. Adams method.

d. Five determinations.

e. Three determinations.

f. Two determinations.

g. Farrington modification.

In the discussion of the results given in Table 1 the following points are to be noted:

The necessity of the double extraction in the ether extraction method with both sweetened and unsweetened condensed milk.

The Leach method is provisional only, and with it should be used the double extraction method with intervening removal of sugar to insure complete recovery of the fat.

The average results obtained by the Gottleib and extraction methods are almost identical.

The original Babcock method is not applicable for either sweetened or unsweetened condensed milk.

The Gottleib method as compared with the extraction method for fat in dried milk and milk powders was investigated and reported in 1906.[1] The results showed plainly that the ordinary ether extraction of fat in dried milks give lower results than the Gottleib method, the differences ranging from about 0.5 per cent to 2.5 per cent in dried milks high in fats, with the average difference of 1.30 per cent in favor of the Gottleib method.

This latter method is particularly applicable to milks, skim milks, and buttermilk low in fat.

Later a comparison was made between the Roese-Gottleib method, (one extraction) which is essentially the same as the Gottleib method, previously described, except in the amounts of the reagents used, and the Adams method, (extraction on strips of paper) with two extractions. With unsweetened condensed milk the majority of the Roese-Gottleib de-

[1] Rpt. A.O.A.C. 1906, P.104.

terminations (one extraction) were higher than the Adams determinations with two extractions. The reverse was the case with sweetened condensed milk, but the query arose whether or not two extractions by the Roese-Gottleib method would have raised the results to those of the Adams methdd

With buttermilk the Roese-Gottleib method gave higher results than the Adams method.

Further Work by Official Chemists: In 1909[1] the instructions as previously given for the Roese-Gottleib method were unmodified. For the extraction method instructions were as follows:

Prepare strips of soft white filter paper about 4 by 24 inches of the quality of the S & S No. 597, by soaking two or three hours in alcohol and then, after thoroughly drying in the oven, extract several hours with ether, or until no residue is left from the ether as it comes through. Distribute 10 c. c. of a 20 per cent solution of the condensed milk, (previously prepared as directed in Circular No. 43, Bureau of Chemistry P. 8) carefully over the whole surface of the dried paper. (This is best done by attaching one end of the paper to some object and holding the other out straight so that the pipette can be emptied by passing the point back and forth over the whole surface. To dry the paper suspend it over a copper wire in the drying oven, where it will thoroughly dry out in two hours, or much more rapidly than if coiled up, or put in a tube. After drying roll up in a coil, wind with thread or small copper wire, place in the extractor, and extract for not less than eight hours. Remove the coil form the extractor, loosen

1 Rpt. A.O.A.C. 1909.

the wire or thread, dry,and suspend in 500 c c of water for two hours, then return the coils to the oven and dry as before, and extract again for not less than five hours. Five c c of milk and a coil 4 by 12 inches can be used if preferred.

The following table gives the results, each determination being made by a different chemist.

Table 3.

Sweetened Condensed Milk.

Double Extraction.	Gottleib.
9.44	
9.09	9.16
9.14	9.20
9.19	9.29
10.07	9.44
9.48	9.39
9.39	9.28
9.35	9.40
9.82	9.88

In June 1909[1] Hunziker worked on the extraction of fat in unsweetened evaporated milk by modified methods.

In August 1909[2] G.E.Patrick commented as follows on fat analysis in condensed milks. He said "As to the methods for fat in

[1] Bul. 134, Purdue Exp.Sta.
[2] A O A C Proceedings 1909.

condensed milk, sweetened and unsweetened, we have in this laboratory used exclusively the Roese-Gottleib method for three years past, and believe it to be correct. In view of Professor Hunziker's recent claim that the Roesse-Gottleib method gives low results, in comparison with his modified extraction method, I shall, during the next few months, make some critical studies of both these methods.

In the following year, 1910, the A.O.A.C, after using Hunziker's method, adopted a recommendation that the Roese-Gottleib method be made provisional for the determination of fat in condensed milk both sweetened and unsweetened.

In the next year, 1911, Associate Referee, A.E.Paul, described another method as follows:

Into a 1,000 c.c. beaker, weigh 100 grams (cream 50 grams) of the material. Add 300 c c water, mix thoroughly, and bring to a boil. Then add, while boiling, very gradually, 25 c c of Soxhlet's copper sulphate solution, diluted with 100 c c water.

In a Büchner funnel wet a filter of suitable size and of loose texture. Filter with suction and wash three times with a little boiling water. Filter as dry as possible. Remove the cake, which should be dry enough to be broken up easily between the fingers. Break into small particles and dry in the open air overnight. Grind in a mortar with sufficient amount of anhydrous copper sulphate (usually 25 grams is enough) and let stand for a few minutes, or until the product seems quite dry and not at all lumpy.

Put a layer of anhydrous copper sulphate in the inner

tube of a large extractor and then add the powdered mixture. Place a loose plug of cotton on top of the mixture and and extract with ordinary ether. The ether should be poured into the extractor and allowed to percolate through before the heating is begun. Approximately 50 c c of the solvent will be required. Dry and weigh the fat.

Results by A. E. Paul.

Vanilla Ice Cream.

Fat	Roese--Gottleib	Per cent	7.58
Fat	recovered from 100 grams	Grams	7.61

Evaporated Milk.

Fat	Roese--Gottleib	Per cent	7.83
Fat	from 100 grams	Grams	7.68

Condensed Milk:

Fat	Roese--Gottleib	Per cent	9.03
Fat	from 100 grams	Grams	8.95

It would seem from the above results that the Paul method is not as applicable to condensed and evaporated milks as the Gottleib method.

Reviewing now the results of this series of methods we are led to conclude that the Babcock method as modified by Leach and the Roese -Gottleib method, which are both provisional, also the double extraction method with ether are recommended at present for the determination of fat in condensed milk.

Results of Analyses by Different Investigators:

For the purpose of noting the differences in results between the various methods, and in order to ascertain if these differences are constant enough and of sufficient magnitude to form a basis for detecting the presence of condensed milk in ice cream, the following summary of results are given:

1 Hunziker and Spitzer record a shortage in the results given by the Babcock test of 0.48 per cent, the shortage varying in individual determination from 0.05 per cent to 0.75 per cent.

The ether extraction, (paper coil 8 hours, one extraction) showed shortage of 0.69 per cent, varying between 0.44 per cent and 1.22 per cent.

Ether extraction, 24 hours, shortage 0.63 per cent.

Gottleib method (two extractions) shortage 0.31 per cent.

Modified ether extraction, by Hunziker, no shortage.

2. Official Chemists results as recorded here show conclusively that there is a wide range of variation between the results of individual chemists for the same sample. Table 4 has been prepared to show these differences.

1 Bul. 134 Purdue University.
2 A O A C 1912 P. 119

Table 4.

Table showing magnitude of differences of results in analysis of condensed milk for Fat.

	Adams	R.Gottleib	From Official Prodeedings A O.A C.				
						Babcock	
Unsweetened.	Adams	R.Gottleib	Asbestos	Leach	Farrington	Old Method	Mod. Method
	a 7.55	7.26					
	a 7.61	7.17					
	a 7.50	7.24					
	a 7.86	7.90					
	a 9.64	9.67					
	a 9.54	9.68					
	a 9.55	9.68					
	a 8.29	8.47					
	a 8.36	8.40					
	a 7.00	7.12					
	a 7.06	7.17					
Sweetened	a 8.83	8.72					
	a 9.05	8.74					
	a 10.03	9.55					
	a 10.06	9.49					
Sweetened		7.87	a 7.66	8.05	7.75		
		8.61	a 8.52				
		8.15	a 7.90	8.1			
Unsweetened			a 8.02	8.4			
		8.41	a 8.47	8.1		8.20	
		8.49	a 8.12				
		8.32	a 7.91	8.1			
Sweetened	a 8.10	8.64					8.32
	a 8.24	8.04					8.10
	a 8.00						8.25
	a 7.80						8.40
		8.50					8.70
		8.70					8.50
	a 8.66						7.80
	a 8.63						7.95
	a 8.16						8.10
	a 8.56						8.55
	a 8.19						7.2
	a 8.04	7.98					7.5
		7.96					7.20
	a 7.01	7.80					7.20
	a 8.52	8.30					8.40

a - double extraction.

Table 4. (cont)

A. O. A. C.

Water Leached Double Extraction.	Gottleib.	Modified. Babcock	Paul Method.
" 8.54	7.89	8.70	
" 8.45	7.78	8.70	
" 8.04	8.25	7.20	
" 8.05	8.11	7.20	
" 9.36	9.36		
" 9.09	9.16		
" 9.14	9.20		
" 9.19	9.29		
" 10.07	9.44		
" 9.48	9.39		
" 9.39	9.28		
" 9.35	9.40		
" 9.82	9.88		
	7.68)		
	)		7.82
	7.83)		
	8.34		7 82
	8.47		8.02
	7.94		7.98
	8.08		8.08
	8.26)		
	)		8.15
	8.23)		

A Study of the Results Given in the Preceding Table
May Be Summarized in the Following Table.

Summary Averages. (Compiled from above table).

Times Compared.	Adams.	Gottleib.	Asbestos.	Leach.	Farrington.	Old Bab.	Mod Bab.	Paul
2	8.66	8.59						
1			8.61	8.52				
1		7.87	7.66	8.05	7.75			
1		8.49	8.12					
4		8.19	7.98	8.09				
1		8.41	8.47	8.10		8.20		
9	8.11	8.09					7.92	
7	8.29						8.04	
3		8.39					8.13	
8		8.13						7.98

In no case was the average difference greater than 0.39%.

From this summary of analyses it will be seen also that the Gottleib method gave highest results, when compared with the Double Extraction method, 17 out of 32 times. However, the Double Ether Extraction method gave a slightly higher average per cent fat.

The Gottleib method was higher than the							Asbestos	5	out of	6 times.
"	"	"	"	"	"	"	Leach	3	" "	4 "
"	"	"	"	"	"	"	Farrington	in a single trial		
"	"	"	"	"	"	"	Babcock	in a single trial		
"	"	"	"	"	"	"	Mod.Bab.	7	out of	12 times.
"	"	"	"	"	"	"	Paul	3	" "	5 "

It is safe to assume that the Gottleib and Double Extraction methods are the most reliable for fat determination in condensed milk.

EXPERIMENTAL WORK.

Fat Analysis Made: The plan outlined involved comparative fat determiniations with condensed milk alone and later with this substance in ice cream. Few of the former were made because of the conclusiveness of the more authoritative data given by official chemists and outlined here. The methods of analysis were essentially those as given in the A.O.A.C. Reports.

Table 5.

Percentage of Fat in Condensed Milk by Different Methods.

	Extraction Purdue.	Gottleib Method	Purdue Modified Method	Original Babcock
	Per cent	Per cent		
Sample 1 Sweetened	7.911	7.907		
" 2 Unsweetened	8.750	8.590		
" 3 Sweetened	7.170	7.230	7.59#	7.43*
" 4 "	7.89	7.60	7.50#	7.50*
Average	7.93	7.832		
Average		7.415	7.545	7.465

* Charred badly.
\# Charred slightly.

Table No 5 reviews a limited number of results for fat in condensed milks. There is a slight difference in favor of the Purdue or Adams method with double extraction and leaching when compared with the Gottleib method. The difference between the Purdue Babcock method and original Babcock method is negligable for it is within the limits of error for the Babcock test bottle, but these latter two methods are not applicable to fat in sweetened condensed milk.

Ice Cream Batches.

The ingredients used in the batches of ice cream were all weighed carefully on a balance accurate to one deci-gram. Samples of the cream and condensed milk were taken for separate analysis. Later, control batches were made without condensed milk and comparison made between these and the batches containing condensed milk.

The following tables give the results of the fat determinations.

Constituents of Ice Cream

	31	Ozs. cream,
	31	ozs. condensed milk,
Batch 1.	9	ozs. sugar,
	1	oz. Gelatin made up to 4 ozs. with water,
	1/2	oz. Vanilla extract,

Table VI.

Per cent Fat in Batch 1.

	[a] Gottleib	[a] Purdue Extraction	[a] Thimble extraction
Cream	16.52	16.75	
Condensed milk	7.907	7.911	
Ice cream	9.78	9.74	9.77
Theoretical [b] Per cent	9.98	9.98	9.98

[a] Extracted three times.

[b] calculated from extraction method

Batch II.

40 ozs. cream,
10 ozs. condensed milk,
9 ozs. sugar
1 oz. gelatin made up to 8 ozs. with water,
1/2 oz. vanilla extract.

Table 7.

Per cent Fat in Batch II.

	[a] Gottleib	[a] Purdue Extraction
Cream	19.30	19.46
Condensed Milk	8.59	8.75
Ice Cream	12.41	11.91
Theoretical	12.83	

[a] Extracted three times.

40 ozs. cream,

10 ozs. condensed milk,

Batch III. 9 ozs. sugar,

1/2 oz. Vanilla.

Table 8.

Per cent Fat in Batch III.

	[b] Extraction	Babcock Purdue	Babcock	[b] Gottleib.
Cream	19.28	19.90	19.7	
Condensed Milk	7.17	[d] 7.59	[d] 7.43	7.23
Ice Cream		[d] 14.05	[c] 14.21	
[e] Theoretical	14.65			

[b] Two extractions.

[c] Clear

[d] Charred.

[e] Calculated from highest per cent.

	1201	grams	cream
	240	"	condensed milk
Batch IV.	270	"	sugar
	15	"	Vanilla extract.

	410	grams	cream
Batch V.	90	"	sugar
	5	"	Vanilla

The cream was the same in each batch, being mixed thoroughly and taken from the same can.

Table 9.

Per cent Fat in Constituents of Batches IV & V.

	Babcock Purdue	Babcock	Gottleib.	Extraction.
Cream	12.6	12.7		12.67
Condensed milk	7.5	a 7.5	7.6	7.89
Ice Cream IV.	8.3	charred	9.70	9.77
Ice Cream V	9.3	9.5	10.21	10.24
Theoretical [b] IV	9.8			
" V	10.2			

a. Slightly flocculent.
b. Calculated from extraction.

Number of Extractions necessary:

Both the Ether extraction method, with soaking, and the Gottleib method, need several extractions. Two are usually enough, but for very accurate results, three are necessary. For the purpose of illustrating this point Table X is given.

Table 10.

Sample	Method	1st.Extraction	2nd.Extraction	3rd.Extraction.
1	(Extraction	9.27 per cent	9.77 per cent	... per cent
	(Gottleib	9.48	9.70	9.78
2	(Extraction	7.74	8.75	
	(Gottleib	8.39	8.53	8.59
3	(Extraction	6.90	7.17	
	(Gottleib	7.22	7.23	
4	(Extraction	7.89	8.00	

Conclusions on Fat Analyses.

The extraction method on paper strips, with soaking in dilute acetic acid between extractions, and the Gottleib method with two extractions, are the most reliable for work with ice cream. No differences that are shown could be used as evidence of the presence of condensed milk. However, if but five or ten per cent of condensed milk was used in the ice cream batch, these differences would be so small that they would be well within the limits of error of variations arising from lack of uniformity in conditions under which different analysts work.

Summarizing the averages of fat extractions for ice cream the extraction method was 0.287 per cent and the Gottleib method 0.177 per cent lower than the calculated fat.

Review of All Work on Fat Extraction.

Reviewing all the results of the work on fat determination the points to be noted are as follows:

1. There is a large variation in results by different methods.
2. There is a variation in results with the same method when used by different manipulators.
3. The variations are not constant but may be great or small.
4. The variations in results by the same method may be great or small
5. Higher results on the average are secured by same methods.

The above conclusions show that the differences seem to be too variable to be of any determinative value when they are to be used as evidence of the presence of condensed milk. The resistance of different brands of even batches of condensed milks to extraction agents must vary considerably, so much so that no definite conclusions could be drawn. Moreover, any method that is based on the partial extraction of the fat must always be more or less variable in the hands of different manipulators working under different conditions. Therefore the solution of this problem cannot be obtained by differences in analytical methods.

Albumin in Condensed Milk.

One important difference between all condensed milks and raw, unheated milk is that they have been subjected to heat for a considerable length of time. This fact formed the basis for a series of analyses with the object of determining the effect of heat on the solubility of the albumin. A number of samples of unheated milk, cream, and condensed milk were analyzed, Sebelien's Method[1] being used.

The casein was precipitated with a saturated solution of magnesium sulphate. An aliquot portion of the clear filtrate was taken and the albumin precipitated with tannin. The nitrogen was determined in the precipitate.

1. Blythe, "Foods, their composition and analysis." PP 227 -228.

Table 11.

Sample No.		Per cent Soluble Albumin.
1	Pasteurized Cream	0.24
2	Raw Cream	0.59
3	Condensed M.	0.084
4	Condensed M.	0.008
5	Condensed M.	0.030

Table XII gives the percentage of soluble albumin in batches of ice cream made, also the percentages of soluble albumin in the cream and condensed milk used in making the ice cream.

Table 12.

Batch 1.

Composition. 40 ozs. cream
10 " condensed milk.
9 " sugar
1 " gelatin made up to 8 ozs.
½ " Vanilla extract.

Soluble Albumin in cream 0.37 per cent.
condensed milk 0.08 per cent.
ice cream 0.21 per cent

Batch 2.

Composition. Same as Batch 1.

Soluble albumin in ice cream 0.25 per cent.

Batch 3.

Composition. Same as Batch 2, but without condensed milk.

Soluble albumin in ice cream 0.46 per cent.

Batch 4.

Made up without gelatin, as it was thought that this substance might influence the results of the nitrogen determination for soluble albumin.

Batch 5 was a check without condensed milk.

Soluble albumin in cream		0.42 per cent.
" " "	condensed milk	0.03 per cent.
" " "	ice cream	0.35 " "

Batch 5.

Soluble albumin in ice cream 0.46 per cent.

It is quite evident from these results that there is but little, if any, albumin in the soluble form in condensed milk, and that low results for this constituent might indicate the presence of condensed milk.

The objections are as follows: Pasteurized cream may give low results also. Albumin containing ingredients might be added to the ice cream. The limits of variation are wide for this constituent. The amount of condensed milk used in ice cream is rarely above ten per cent, hence the difference in soluble albumin would be too saml1 to be of any value. The results show that when the expected reswlt for soluble albumin is calculated from the formulas, the margin is very narrow between it and what would be expected normally,

Ash in Condensed Milk.

The following table gives the per cent ash in sweetened and unsweetened milks examined.

Table 13.
Per Cent Ash.

Sweetened, Bordens	1.930
	2.180
	1.860
Unsweetened, Van Camps	1.610
" "	1.490
" "	1.680
" "	1.660
" "	1.590
" "	1.702
" "	1.806
"	1.730
"	1.380

Several batches of ice cream were made up and the ash of the cream, condensed milk and ice cream analyzed.

Batch 4.	Cream	0.549 per cent ash
	Condensed Milk	1.610 " " "
	Ice Cream	0.561 " " "

Batches 5, 6, 7 & 8 were made up with and without condensed milk as indicated in the table.

Table 14,

Ash in Ice Cream Ingredients.

Batch No:	Cream	Condensed milk	Ice Cream
4	o.549	1.610	0.561
5	0.560	1.490	0.61
6	0.560	none	0.39
7	0.695	1.60	0.71
8	0.695	none	0.57

Inspection of these results shows that there is a noticable difference in results for ash, and that this difference is the most striking so far encountered. Although the ash content is high, the amount of condensed milk used in ice cream is small. When gelatin is added to ice cream the ash content would be increased, for gelatin contains about 2 per cent ash.

Lactose.

The presence of an excess of lactose could not be determined when less than fifty per cent of unsweetened condensed milk was used in the ice cream, since the excess would be within the range of variation for that constituent. If condensed milk containing 8 per cent lactose were added to cream containing 4 per cent in the proportion of 1 to 1, the per cent lactose in the ice cream when sugar and

gelatin is added might be as low as 5.5, which is not abnormal. When but ten per cent condensed milk is used no difference would be detected since the lactose would be increased only 0.8 to 1.0 per cent.

Qualitative tests were made for lactids which may be formed under special conditions in lactose solutions and which are insoluble, but the results were negative.

Ratios of Constituents of Condensed Milk in Ice Cream.

Condensing milk reduces the water content and increases the percentage of all other milk constituents. When the bulk is reduced to one half by evaporation of water, the per centage of each constituent is doubled. In milk containing 13 per cent solids the ratio of solids to water is as 1 : 6.7. In the same milk, reduced to half its bulk by condensing, the ratio is as 1 : 2.8. If then a constituent could be found, the per centage of which is not affected by non-milk substances added in ice cream making, and its ratio to the water content of the ice cream found, the narrowing of the ratio below normal would indicate the use of condensed milk.

Fat would not be a suitable constituent for comparison since it can be added by the homogenizer.

Nitrogen determination would be affected by addition of gelatin. Casein could be precipitated with acid if soluble salts were added

Lactose is unaffected and may be used.

The percentage of ash is affected slightly by the addition of binders and filters.

Casein, lactose and ash are the constituents that could be used for such a comparison, but the limits of normal variation of these constituents in cream would have to be determined in order to find the ratio limits.

Casein determination would have to be conducted without involving the albumin content, for if that were involved, the albumin of the gelatin would interfere with the calculations and conclusions. The difficulties involved in working with a highly heated product like condensed milk are many, and it is probable that accuracy could not be secured in this determination.

Lactose could be determined with a fair degree of accuracy and could be used. Its variation in milk is wide, however, and its ratio to water is narrow.

The minimum total solids that are allowed by law in condensed milk is 28 per cent. Therefore, the maximum water content is 72 per cent. The minimum ash content for condensed milk containing the required amount of solids is not less than 1.5 per cent, and the maximum ratio between ash and water that could be found in legal condensed milk is 1 to 48.0. This would not apply to condensed skim milk which has no legal standard. Generally, however, the skim milk product is condensed to a greater extent than condensed whole milk, and often contains 28 per cent solids without the fat. Therefore, this product is comparable to condensed whole milk.

Analyses of Condensed Milk
And Ratio of Constituents to Water.

Table

Total Solids.	Water.	Lactose.	Ratio	Proteids	Ratio	Fat	Ratio	Ash	Ratio	
28.45	71.55	10.84	1 : 6.6	8.19	1 : 8.7	7.85		1.57	45.6)	
29.10	70.90	11.00	1 : 6.45	8.40	8.4	8.09		1.61	44.0)	
30.06	69.94	11.03	1 : 6.3	8.53	8.2	8.85		1.65	42.4)	
27.86	72.14	10.87	1 : 6.6	7.59	9.5	7.81		1.55	46.5)	
28.88	71.12	11.01	1 : 6.5	8.09	8.8	8.10		1.68	42.3)	
25.31	74.69	9.48	1 : 7.8	7.07	10.5	7.31		1.45	51.5)	
28.24	71.76					8.23				)N.Dakota
27.02	72.98	10.22	1 : 7.1	7.20	10.1	7.94		1.52	48.0)	
26.92	72.08	11.10	1 : 6.5	7.09	10.1	7.20		1.53	47.1)	
29.73	70.27	11.62	1 : 6.0	8.60	8.2	7.65		1.86	37.7)	
23.99	76.01	9.75	1 : 7.8	6.35	11.9	6.58		1.34	56.6)	
27.10	72.89	10.85	1 : 6.7	7.46	9.7	7.26		1.54	)	
35.17	64.82	13.90	1 : 4.7	15.37	1 : 4.2	4.20		1.70	1 : 47.3)	
71.35	28.65	6.96		11.33		4.30		2.15	)	
			1 : 8		3.7					
	70.81	12.24	1 : 5.8	6.97	1 : 10.2	8.20		1.36	1 : 52.0)	Borden Average
29.55				7.32				1.34	)	
29.81				8.44				1.48		)A O A C
30.04				7.04				1.79	)	
28.33		11.0		7.50 curd				2.17	)	
29.25	70.75	9.75	1 : 7.2	8.44	1 : 8.4	9.42		1.54	1 : 45.8)	
24.63	75.37	8.56	1 : 8.8	7.41	1 : 10.1	7.45		1.36	1 : 55.4)	
26.2	73.80	9.10	1 : 8.1	7.54	1 : 9.8	8.07		1.47	1 : 50.2)	
27.18	72.82	9.23	1 : 7.9	7.39	1 : 9.8	9.07		1.49	1 : 48.8)	
29.04	70.90	10.37	1 : 6.8	7.86	1 : 9.0	8.35		1.62	1 : 43.8)	
31.08	68.92	10.47	1 : 6.6	8.26	1 : 8.3	10.49		1.67	1 : 41.3)	
23.81	76.19	7.55	1 : 10.1	6.49	1 ; 11.7	8.05		1.24	1 : 61.4)	
28.38	71.62	9.94	1 : 7.2	8.39	1 : 8.5	8.47		1.56	1 : 45.9)	
27.89	72.11	9.66		7.52		8.69		1.54		)Purdue
25.23	74.77	8.68		6.53		8.70		1.37	)	
26.70	73.30	10.35		6.77		8.09		1.44	)	
24.96	75.04	7.92		7.06		8.16		1.33	)	
26.66	73.34	10.21		6.88		8.08		1.45	)	
25.62	74.38	9.20		6.89		8.25		1.43	)	
27.04	72.96	9.36		7.21		8.73		1.48	)	
28.02	71.98	9.86		7.68		8.93		1.61	)	

Table 16.

Batch 1.	Grs.	Water	Fat Adams	Fat Gottleib	Fat Babcock	Fat Purdue	Sol Albumin	Ash	Total Solids	S N F
Ice Cream.	2265									
Pasteurized Cream	930		9.77 16.75	7.98 16.52			0.0 0.0			
CondensedM	930		7.91	7.91			0.0			
sugar	270									
Gelatin	120									
flavoring.	15									
Batch II.										
Ice Cream	2115	61.89	11.91	12.41			0.21	0.72	38.11	8
Raw Cream	1200	72.51	19.46	19.3			0.37	0.55	27.39	7.93
Condensedm	300	69.5	8.75	8.59			0.0	1.61	30.5	21.75
sugar	270									
Gelatin	240									
flavoring	15									
Batch III.(a)										
Ice Cream	1785	63.23			*14.21	*14.05	0.35	0.61	36.77	
Raw Cream	1200	73.34	19.2		#19 7	#19.9	0.42	0.56	26.66	7.44
Condensed M	300	72.83	7.17	7.23	# 7.4	# 7.5		1.49	27.17	20.00
sugar	270									
flavoring	15									
III.(b)										
Ice Cream	1485	60.57			Charred	*15.93	0.46	0.39	39.43	
Raw Cream	1200	73.34	19.2		#19.7	#19.9	0.42	0.56	26.66	7.44
sugar	270									
flavoring	15									
Batch IV.(a)										
Ice Cream	1725	65.86	9.77		* 8.4	* 8.4		0.71	34.14	
Raw Cream	1201	79.59	12.67			12.6		0.72	20.41	7.74
Condensed M	240	72.55	8.00	7.6	7.5	7.5		1.59	27.45	19.45
sugar	270									
flavoring	15									
IV.(b)										
Ice Cream	505	64.85	10.24		* 9.5	* 9.25		0.57	35.15	
Raw Cream	410	79.59	12.67		12.7	12.6		0.71	020.41	7.74
sugar	90									
flavoring	5									

Charred
took about 5 grams only.
calculated without sugar

The ratio of ash to water content could be used then to determine if condensed milk had been added as such in ice cream, for if this had been done, the ratio should be narrower than normal. Condensed milk is generally added for its effect on the body, texture and stabilizing qualities of ice cream. When it is diluted to be used as normal milk, no ratio comparison could be used for its detection.

Table 15 is given to show the ratio of the constituents in condensed milk to each other, and is made from a large number of analyses.

Table 16 gives a summary of the analyses made of batches of ice cream with and without condensed milk. The ratio of the ash to water in ice cream without condensed milk, should be above 1 to 110. When any considerable quantity of undiluted condensed milk has been added to the ice cream mix, this ratio will fall below, and will indicate the presence of condensed milk. In ice cream batches without condensed milk the ash content should not be over 0.6 per cent and the ratio to water not below 1 to 110. The same cream with 10 per cent condensed milk containing the minimum solids would have an ash content of 0.7 per cent and the ratio would be 1 to 105. T

The foregoing examples represent the extremes. A few samples might correspond to these conditions but in most cases the differences would be greater.

For the purpose of showing that normal cream, of the percent fat used in ice cream, never falls to as low a water and ash ratio as 1 to 110, the following table of ratios is given. Note that the lowest per cent fat is 25.67 or 13 per cent higher than the State

standard, and that the ratio would be increased by lowering this fat to the State standard.

Table 17.

Ratio of Ash in Cream to Water.

Fat	S N F	Ash	Ratio
25.67	6.83	0.57	1 to 118
31.40	5.14	0.52	1 to 112.
45.90	5.02	0.42	1 to 108
50.40	4.65	0.38	1 to 107
50.40	4.77	0.39	1 to 104
51.00	4.47	0.38	1 to 109
52.00	4.40	0.38	1 to 109
53.60	4.17	0.41	1 to 113
64.80	3.30	0.28	1 to 114

Summary

Differences in results of analyses for fat in Condensed milk are not constant. They vary in the hands of different manipulators. Chemists.

The differences between any two methods are not wide enough to be of any determinative value when small quantities of Condensed milk are used in ice cream.

When ice cream is made from Condensed milk entirely, or in any proportion over 50 per cent, the soluble albumin content would be very low, and would indicate the use of condensed milk.

The low albumin content conbined with a high ash content would be strong evidence that condensed milk had been used.

If the ash to water ratio fell below 1 to 110, further support would be given to the conclusions.

The boiled milk test with hydrogen peroxid and paraphenylenediamin might be used as confirmatory evidence.

When ice cream is made entirely, or in large part, from condensed milk diluted to normal milk proportions with water, as is sometimes done, the low per cent of soluble albumin would be conclusive evidence of the fact.