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THE DETERMINATION OF FAT IN CERTAIN MILK PRODUCTS

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DEPARTMENT OF CHEMISTRY

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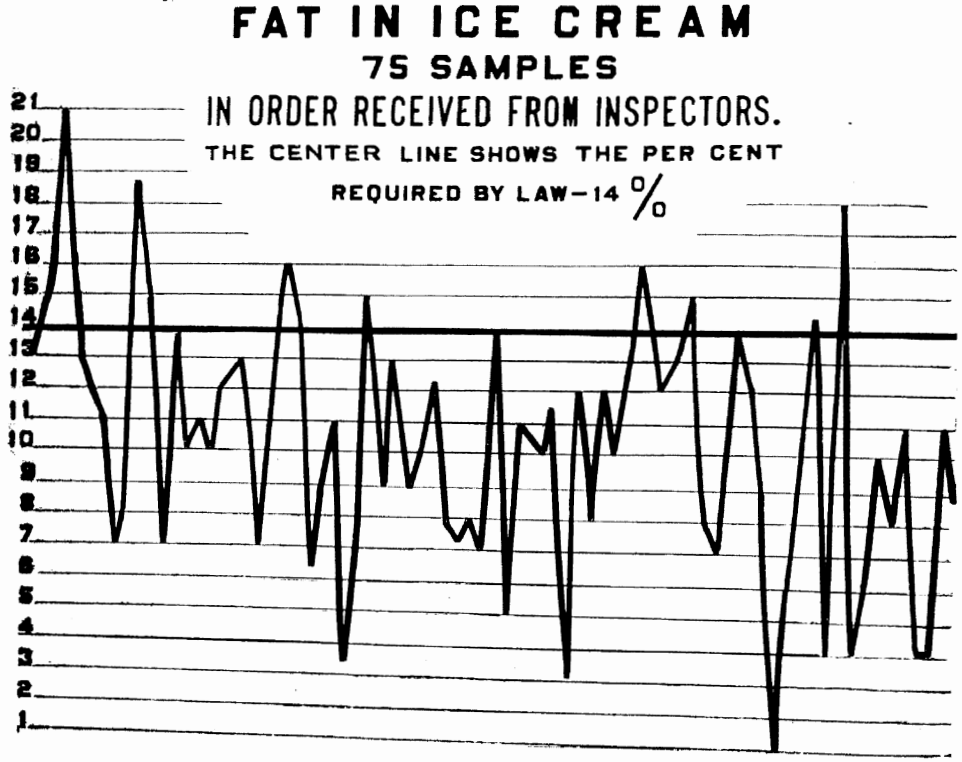
The Determination of Fat in Certain Milk Products

BY C. K. FRANCIS and D. G. MORGAN

The general public believes that ice cream is a uniform product, but chemical examination shows that much of the ice cream collected by the inspectors of the State Board of Agriculture is by no means of a uniform composition, and especially that the fat content varies considerably.



The chart was prepared by listing seventy-five samples of ice cream in the order in which they were submitted to the laboratory by the inspector, and as they were examined in the same sequence, the results were in no way selected. This chart demonstrates the need of an accurate and rapid method for the determination of fat in ice cream.

Many foods are valued for the amount of fat they contain. The chief dairy products may be considered in the class of fatty foods, and it is but natural that the estimation of fat is considered of great importance by the creamery and delated interests. The different products obtained from milk are quite unlike, both in form and composition, and for such products as ice cream, evaporated milk, malted milk and powdered milk, some other method of estimating the percentage of fat must be used than that employed with normal milk and cream.

Many methods based on the Babcock test have been proposed for use on ice cream, milk powders, etc., but have not received the endorsement of the Association of Official Agricultural Chemists, which is in charge of the methods recognized by the various courts. The official method of the Association of Official Agricultural Chemists for the determination of fat in the products mentioned, is known as that of Roese-Gottlieb, and is based on the use of ether or some other substance having the power to dissolve fats. Objections are made to the method because a special and somewhat expensive apparatus is necessary, and it appears that lower results are obtained when working on powders and semi-liquid preparations.

A number of the more recent methods for the determination of fat were tested by applying them to malted milk and powdered milk, but the most encouraging were those in which sulfuric acid was used. It appears that large quantities of acid were necessary to remove the carbohydrates. Considerable sugar was found in some samples, and when this and other organic matter was carbonized the separation of the fat from the black mass was poor, and in many experiments no fat was obtained. Owing to the extreme affinity of the sulfuric acid for water and carbohydrates, the action of this acid on powders and semi-liquid preparations was severe, and often excessive carbonization resulted. After this had occurred the addition of sulfuric acid appeared to have no effect. In an effort to separate the fat, other substances were added to the milk-acid mixture. The presence of small quantities of nitric acid appeared to influence the reaction, and as the preliminary tests seemed to meet with success, the method as herein described was developed.

Preparation of Samples

Ice Cream.—Some special precautions are necessary in order to obtain a representative sample of ice cream. Usually a good sample may be collected in the factory directly from the freezer by withdrawing small portions on a large spoon from different parts of the container and from the blades of the stirrer. When the ice cream is in a packer or can, a butter trier or thief may be used, if the cream is well frozen. The trier should be thrust into the cream from top to bottom at least three times, the separate portions being combined into one sample. If the cream has become soft in the container it will be necessary to stir it well with a spoon or paddle before sampling.

If the ice cream is not to be analyzed at once, or is to be forwarded to a laboratory at a distance, it should be allowed to melt at room temperature and a small quantity of bichloride of mercury added as a preservative. If a tablet is used it should be finely pulverized and thoroughly mixed with the sample, using one-fourth of a tablet for every quart of ice cream.

The representative sample, if in good condition, should be allowed to melt at room temperature, but if the fat has separated, it is necessary to heat to about 50° C. or 120° F. If the sample is sour or contains much curd a few cc. of ammonia may be added. The melted sample should be thoroughly mixed by pouring from one vessel to another and approximately 10 cc. of the uniform sample removed at once with a large bore pipette and 9 grams quickly weighed into a 30% cream test bottle.

Evaporated Milk.—Mix as under ice cream and weigh 4.5 grams of the sample into a 30% cream test bottle. If the sample is in a diluted form, use a 9-gram sample. In the case of sweetened condensed milk, slightly warm by immersing in warm water before mixing.

Malted Milk and Dried Milk.—Mix the entire sample thoroughly and quickly weigh 4,5 grams into a small beaker. Transfer to a 30% cream test bottle with the aid of the least possible amount of hot water. If the sample has a low fat content, as in the case of dried skimmed milk, a 10% milk test bottle, or a skimmed milk test bottle, should be used.

Determination of Fat

Ice Cream and Evaporated Milk.—Prepare a mixture of equal parts of glacial acetic and sulfuric acids, and add to the sample 4 to 5 cc. of the mixture at a time, mixing thoroughly after each addition, until a dark brown color develops. Add concentrated nitric acid, 1 to 2 drops at a time, and shake thoroughly after each addition until the action of the acid has subsided, or excessive foaming may result. After two or three such additions, the number of drops may be increased to 5 or 6 if the precautions to prevent excessive foaming are observed. When a light yellow color results, immerse the bottle in boiling water

for 3 to 4 minutes, or until the dark brown color returns. Then centrifuge for 5 minutes at 1200 r. p. m., fill nearly to the neck with hot water, and centrifuge an additional 2 minutes. Bring the fat column into the scale portion of the neck with more hot water and centrifuge for another minute, reduce the meniscus with glymol and multiply the reading by 2 for 9-gram samples and by 4 for 4.5-gram samples.

Malted Milk.—Prepare a mixture of sulfuric and nitric acids (20 cc. of H_2SO_4 to 1 cc. HNO_3) and add to the sample 1 to 1.5 cc. at a time, shaking thoroughly after each addition until a light yellow color results. This ordinarily requires about 15 to 20 cc. of the mixture. Immerse in boiling water until a dark brown, almost black, color develops. Now add a mixture of sulfuric and nitric acids (10 cc. of H_2SO_4 to 2.5 cc. of HNO_3), 1 cc. at a time, shaking thoroughly after each addition, until a light red color has developed and started to darken. Immerse again in boiling water until the dark brown color returns. Centrifuge for 5 minutes, fill nearly to the base of the neck with hot water and centrifuge 2 minutes. Raise the fat column into the scale portion of the neck with more hot water and centrifuge 1 minutes. Reduce the meniscus with glymol and multiply the reading by 4.

Dried Milk.—Add concentrated sulfuric acid, 5 to 6 cc. at a time, until a dark brown, almost black, color develops. Prepare a mixture of sulfuric and nitric acids (10 cc. of H_2SO_4 to 2.5 cc of HNO_3), and add half a cc. at a time, shaking thoroughly after each addition, until a clear, light red color results. This requires ordinarily 5 to 6 cc. of the mixture. Immerse in boiling water until the dark brown color returns, add more of the mixture until the light red color is produced, and again immerse in the boiling water until the dark brown color appears. Centrifuge 5 minutes and proceed as for malted milk.

Discussion of Results

The results obtained by this method on different products have been compared with those obtained by the Roese-Gottlieb method, as indicated in the following table:

Comparative Tests

Roese-Gottlieb Method Percent		Sample	This Method Percent	
7.58	7.68	Ice Cream	7.7	7.7
7.93	7.90	Unsweetened Evaporated Milk	8.0	8.0
9.35	9.35	Sweetened Condensed Milk	10.0	10.0
7.26	7.14	Malted Milk	7.4	7.4
1.36	1.29	Dried Skimmilk	1.2	1.2

It appears that our method gives uniformly higher results than the Roese-Gottlieb method. In every case a good separation of fat was obtained free from particles of carbon and bubbles. It is important that the nitric acid be added with care and an excess avoided; too much nitric acid may cause the formation of a large quantity of gas, the gas bubbles continuing to rise for some time and producing a froth which interferes with the reading of the fat column. The action of the acid is more rapid when added to the warm mixture, and it is suggested that the bottle be immersed in warm water while the acid is being added.

Summary

A method for determining fat, including treatment of the samples, in such dairy products as ice cream, evaporated milk, malted milk, dried skimmed milk and similar milk products, is described. The procedure is similar to that followed when using the well known Babcock test, but in place of sulfuric acid, mixtures of glacial acetic, sulfuric and nitric acids are prescribed. The fat is separated and read in a Babcock bottle.