DETECTING FOREIGN FATS IN ICE CREAM

by

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INTRODUCTION

In the past few years the practice of substituting various foreign fats for butterfat in ice cream has been increasing. Foreign fats used as substitutes include animal fats other than butterfat and vegetable oils, either in their natural state or hydrogenated. The shortage of butterfat due to increased fluid milk consumption in some areas, the inexpensiveness of butterfat substitutes, and consumer acceptance of many new types of food and dairy products have made it economically feasible for processors of vegetable fats to supply ice cream manufacturers with substitute fats. It is a known fact that some ice cream manufacturers are using vegetable fats as the only fat in products some of which are labeled ice cream. In some instances these products resemble ice cream closely enough that experts cannot detect them from ice cream by organoleptic methods.

It is illegal in most states to use any fat in ice cream other than butterfat, or to market an iced product containing mixtures of fats unless properly labeled. Methods of detecting mixtures of fats in ice cream have assumed great importance. At the present such methods or procedures are not well established.

The purpose of this study was to obtain a satisfactory method for detecting foreign fats in ice cream. As a first step it was necessary to determine a satisfactory method for extracting fat from ice cream.

REVIEW OF LITERATURE

Butterfat Extraction Procedures

Detecting foreign fats in ice cream presents two problems. First, it is necessary to employ some method of recovering all of the fat from ice cream, or a representative portion of the fat. Second, it is necessary to detect mixtures of various fats with butterfat.

In the past studies connected with butterfat, which required the removal of homogenized fat from a dairy product in relatively large quantities, have been limited. The removal and study of butterfat in ice cream has received little attention. Epple and Horral (7), in 1939, described a modified Mojonnier method of extracting butterfat from ice cream for the purpose of analysis. To obtain more complete recovery they proposed the use of glacial acetic acid instead of ammonium hydroxide. The extraction was made from 200 grams of ice cream mix or melted ice cream in a 500 ml separatory funnel. The completeness of extraction was only about 75 percent and they did not compare the saponification and Reichert-Meissl values of the extracted butterfat with butter churned from the cream used in ice cream. Kingsley (14) reported a method of butterfat determination in ice cream which compared very closely in accuracy with the Roese-Gottlieb procedure. The method described consisted of the removal of butterfat from four grams of sample after additions

of 40 ml of chloroform and ten grams of anhydrous sodium carbonate. The contents were shaken in a 50 ml Erlenmeyer flask, filtered into a previously weighed dish, and then heated to a uniform weight in a hot water bath.

According to Kingsley (14) one of the main disadvantages of the Roese-Gottlieb and Mojonnier methods is the formation of a gelatinous mass which often inhibits separation of the ethereal fraction.

Golding (8) proposed a churning method for determining the percentage of butterfat in milk, cream or ice cream. This method has not been reported as a method to extract butterfat from several hundred grams of ice cream which may be necessary to obtain enough butterfat for thorough analysis. Golding (8) did not state that the butterfat was suitable for chemical analysis after extraction. However, the percentage of butterfat recovery compared very favorably with Roese-Gottlieb butterfat determinations.

Only in the last two years has consideration been given to the method of butterfat extraction in lipase studies. Johnson and Gould (13) recently described a method of butterfat extraction from cream in relatively large quantities. Butterfat from 125 grams of cream was extracted by adding ethanol with subsequent additions of ethyl ether and Skellysolve F. Reichert-Meissl values of the extracted butterfat were in agreement with those obtained from churned cream which was used as a control. This method has not been applied to ice cream.

Detection of Foreign Fats in Butterfat

Many physical and chemical determinations used to differentiate various fats and oils are based upon the differences of the component glycerides which yield various fatty acids upon hydrolysis. Butterfat is unique among fats and oils because it contains larger quantities of volatile, water soluble fatty acids. Reichert-Meissl, Polenske and Kirschner procedures are based upon this fact. The Kirschner procedure approximates the butyric acid content; the Polenske process determines the volatile, insoluble fatty acids; and the Reichert-Meissl determination measures the volatile, soluble fatty acids of fats and oils. These three procedures have been widely used for about 50 years to differentiate butterfat from other fats and oils primarily in butter and butter substitutes. The use of them in studying mixtures of vegetable fats and oils in ice cream was not found in the literature.

In England, from 1900 to 1925, numerous investigators used this method for determining the percentages of mixtures of fats and oils. Elsdon and Smith (6), Cocks and Nightengale (4), Manley (15) and Arup (1), compiled and studied tables, graphs and formulae by which Reichert-Meissl, Polenske and Kirschner values were used in studying mixtures of butterfat, particularly with coconut oil and margarine. Later, many investigators recognized the limitations of this method since the composition of butterfat varies with the type of feed, season and to a

smaller degree, individuality of cows and breed of cows.

Cranfield and Taylor (5) observed as early as 1915 that butterfat from cows on poor pastures had Reichert-Meissl numbers lower than 24 which was the accepted minimum Reichert-Meissl number for unadulterated butterfat. In general, vegetable fats and oils, in cows' diets tend to lower the Reichert-Meissl, Polenske and Kirschner numbers (12, 3, 24). Hill and Palmer (12), after extensive feeding experiment, concluded that feeding 0.8 to 1.25 pounds of linseed oil per cow per day, caused one group of cows to produce butterfat having a Reichert-Meissl number of 22.9, while cottonseed oil did not appear to have that effect. Brown and Sutton (3) and Sutton, Brown and Johnston (24) reported corn oil and fish oil in cows' rations greatly reduced the Reichert-Meissl number of the resultant butterfat. Some samples of butterfat were liquid at room temperatures and had Reichert-Meissl numbers as low as 14.4.

Results reported by Hilditch and Slightholme (11) and Spitzer and Epple (21) indicate that dry, summer and autumn pasture reduced the Reichert-Meissl number of butterfat.

Palmer and Crockett (18) indicated that silage increased Reichert-Meissl numbers of butterfat, while cottonseed meal depressed them.

For many years, according to Arup (1), it had been a known fact that Irish butter made in the Irish Free State, had abnormally low Reichert-Meissl, Polenske and Kirschner numbers. Of 310 samples taken from 30 creameries and two agricultural

school creameries, 50 samples of butter had Reichert-Meissl values of less than 24, and three samples had values less than 21. Arup (1) stated that Polenske and Kirschner values were correspondingly low. A year later the condition had been partially corrected due to improved feeding practices.

Hill and Palmer (12) concluded that when oats or corn are substituted for 35 to 50 percent of the digestible nutrients of a low fat ration containing alfalfa hay, fed to dairy cows, the chemical characteristics of the fat are more or less specific for the type of ration fed. In their experiments, alfalfa hay tended to lower Reichert-Meissl numbers. This was substantiated by Stout and Wilster (23) who studied chemical values of butterfat obtained during winter months from three sections of Oregon in which there were different feeding practices.

Since the Reichert-Meissl, Polenske and Kirschner combination has limitations due to the variable composition of butterfat, several investigators have proposed other methods of detecting foreign fats in butterfat.

Manley (15) reported a procedure somewhat similar to the Reichert-Meissl process but without distillation. The titration values which he obtained corresponded to Kirschner numbers and were termed M values. However, the accuracy of this method was later questioned by Shrewsbury (19).

A colorimetric process was proposed by Henville and Paulley (10). From 97 to 98 percent of margarines examined by these workers contained a dye which could be extracted by ammonia.

About 94 to 95 percent of butter samples examined gave no color upon an ammonium treatment which they described.

Venkatachalam (25) claimed that highly hardened oils, mutton and beef fats could be detected in butter by a test based upon crystallization of stearate glycerides. Foreign fats in butter tended to form crystals when one ml of the suspected sample was placed in 15 ml of a mixture of acetone and alcohol and allowed to stand for several hours.

Most of the proposed methods listed above were no more specific than the Reichert-Meissl, Polenske and Kirschner combination and some procedures were criticized for being inaccurate.

Vegetable oils contain phytosterol: butterfat contains cholesterol, but no phytosterol. In 1922, Buttelet (16) experimented with a method called the phytosterol acetate test. By extracting the sterols from the oil or fat by precipitation with the glucoside, digitonin, and converting to the corresponding acetates by heating with acetic anhydride, the melting points may be determined after purification and recrystallization. The cholesterol acetate separated from 15 samples of French butter melted at 113.6° C. to 114.2° C. while phytosterol acetate crystals melted at a temperature of 125.0° C., according to Buttelet (16). The mixed acetates from butter containing five percent coconut oil melted at 115.5° C.

In India, Hawley (9) reported half-starved cows frequently produced butterfat with Reichert-Meissl values as low as 14. It was then often adulterated with hardened vegetable oils. No

reported the use of the phytosterol acetate procedure as a routine test for butterfat adulteration analysis and stated that five percent vegetable fat could be detected with certainty. He modified the procedure because it was long and tedious.

Woodman (26) reiterates the complexity of the procedure and states that one must obtain experience before definite conclusions can be drawn.

The phytosterol acetate test is listed as a tentative method in the "Methods of Analysis" of the Association of Official Agricultural Chemists (2).

From reviewing the literature it appears, therefore, that the phytosterol acetate test is the most precise and conclusive test to detect adulteration of butterfat with vegetable oils, either in a liquid or hydrogenated state. However, it is complex, time consuming and difficult to interpret results. In addition, a problem still exists of detecting adulteration of butterfat with other animal fats in which no phytosterol is present.

Numerous investigators (17, 21, 23) have also shown that butterfat, from cows fed normal rations, varies in composition within certain limits. Overman and Garrett (17) concluded, after studying variation in chemical properties of butterfat from cows which were fed normal rations, that differences between butterfats did not appear to be large enough to be of other than scientific interest.

Stout and Wilster (23), who studied butterfat which cows

produced during the winter months in three different sections of Oregon where different types of feeding were practiced, obtained no Reichert-Meissl numbers lower than 26.03 and the highest was 31.5.

Probably the most conclusive work with Reichert-Meissl numbers of butterfat was done by Spitzer and Epple (21). During a period of four to five years, 500 creamery samples of butter were analyzed. The lowest Reichert-Meissl number obtained was 26.45, and the highest 32.10. The average of 500 samples was 29.91.

Thus it appears that the Reichert-Meissl process assumes importance in the detection of foreign fats in ice cream. It has been shown that it is not infallible, but can be used with proper interpretations. At the present no other test for adulteration found in the literature appears to be more satisfactory when specificity, time, cost and other factors are considered.

EXPERIMENTAL PROCEDURE

The problem of detecting foreign fats in ice cream was based upon the long known fact that different fats have different chemical values. Butterfat is the only fat which contains glycerides that yield appreciable amounts of volatile, soluble fatty acids. This is the basis of the Reichert-Meissl number. Butterfat from cows fed normal rations has Reichert-Meissl values ranging from 24 to 33, while all other fats and oils have

numbers of approximately one or less. Coconut oil is an exception, because it more closely resembles butterfat than any other vegetable oil. It has a Reichert-Meissl number of about 17. Therefore, if butterfat in ice cream is mixed with a foreign fat, the Reichert-Meissl number of the resulting mixture should be lowered in proportion to the amount of foreign fat which has been added.

The Kirschner number which approximates the butyric acid content and the Polenske number which measures the insoluble, volatile fatty acids of fats are also higher for butterfat than for other fats with the exception of coconut oil. Butterfat has a Kirschner number ranging from 19 to 26 according to Woodman (26), coconut oil gives an average of 1.9, while the majority of other fats and oils give values from 0.1 to 0.2. Polenske numbers of butterfat vary between 1.5 and 3.0 and coconut oil has Polenske numbers between 16.3 and 17.8. However, other fats and oils generally have values less than one. These three chemical values of butterfat were the criteria used in studying ice cream containing foreign fats mixed with butterfat.

The experimental procedure was divided into two phases. The preliminary work and first phase included a study of methods of butterfat extraction from ice cream. The second phase consisted of applying a satisfactory fat extraction procedure to ice cream containing mixed fats and studying Reichert-Meissl, Polenske and Kirschner numbers of these fats.

Yield of Extraction Procedure

Experimentation indicated that butterfat could be extracted from ice cream by shaking or churning the melted ice cream with Minnesota Babcock reagent. When the regular Minnesota Babcock test (22) is employed to determine the percentage of butterfat in ice cream, the prescribed amount of reagent used is a volume in ml 1.67 times the weight in grams of the ice cream. To determine if this was the correct ratio to use for a churning extraction procedure, ten 35 gram portions of ice cream were weighed, with each portion being placed into a 300 ml Erlenmeyer flask. These portions were designated numerically one to ten. Fifty ml of Minnesota Babcock reagent (22) were added to each of portion one and two, 40 ml to portions three and four, 30 ml to portions five and six, 20 ml to portions seven and eight, and ten ml to portions nine and ten. The ratio of the volume of reagent to the weight of ice cream expressed in grams for each of the five duplicate portions was 3, 1.6, 1.2, 0.8 and 0.4. Portions one, three, five, seven and nine were churned by shaking vigorously for three minutes while portions two, four, six, eight and ten were churned for six minutes.

Upon completion of the churning which was evidenced by globules of fat floating in the reagent, enough cold water at 20° C. was added to fill each of the ten flasks. The butterfat from the top of each flask was then removed with a butter trier and placed in a Mojonnier butterfat extraction flask. Twenty-

five ml of ethyl ether were added to each extraction flask and shaken for one minute and 25 ml of petroleum ether were added to each extraction flask and the contents shaken for one minute. The Mojonnier flasks were then centrifuged, the ethereal portion decanted into previously weighed aluminum dishes, and a second extraction was performed similar to the first except 15 ml of each of the ethers were used. The aluminum dishes were heated, tempered in a dessicator and weighed according to the regular Mojonnier procedure for determining butterfat content in ice cream.

After this entire procedure was performed two times, the correct amount of Minnesota Babcock reagent to use for churning extractions appeared obvious. No further trials were conducted.

Application of Extraction Procedures to Ice Cream

Butterfat from four ice cream mixes, which were made in the College Creamery, were studied. Each mix contained 12 percent butterfat, 11 percent serum solids, 15 percent sugar and 0.3 percent gelatin. More than 80 percent of the butterfat was supplied by cream obtained from milk produced by the college herd. The remaining 20 percent or less was obtained from milk also from the college herd or another herd under similar feeding conditions. These mixes were made during February, March and April. The cream used for the butterfat in each of the four mixes was from cows fed normal rations. A representative quart

sample of cream was removed from each lot of cream prior to making the mixes and processing. The cream was churned in a Daisy hand churn. The butter thus obtained was used as a control.

The mixes were pasteurized at 160° F. for 30 minutes, homogenized at 1000 and 3000 pounds per square inch pressures in a two-stage Gaulin homogenizer, and immediately cooled to 40° F. After each mix was frozen in a 40 quart direct expansion batch freezer, pint samples of the ice cream were selected at random for subsequent examination which included extraction of butterfat by three methods later described and the determination of Reichert-Meissl, Polenske and Kirschner numbers.

Reichert-Meissl Determinations. All Reichert-Meissl numbers were determined according to the procedure adopted by the Association of Official Agricultural Chemists (2). In the preliminary work, two condensers were used for the distillation portions of the process. However, to facilitate faster analysis, four condensers were later used simultaneously. One operator can perform the distillations satisfactorily using this number. Saponification was accomplished by heating five grams of sample, together with 20 ml of a mixture of one part sodium hydroxide to nine parts glycerol, over a Bunsen burner. The flame was adjusted to insure complete saponification in a period of time ranging from seven to ten minutes. Then 135 ml of distilled water and six ml of one part sulfuric acid in four parts of distilled water were added, and distillation was begun. Two

small pea-size pieces of pumice were used in each 300 ml Erlenmeyer flask to prevent violent boiling during distillation. The one-tenth normal sodium hydroxide solution was checked periodically against potassium acid phthalate to insure absence of contamination and error. The indicator used was a one percent solution of phenolphthalein in ethyl alcohol.

Polenske Determinations. Polenske numbers were determined according to the method of the Association of Official Agricultural Chemists (2). Rinsing the condensers with water was started immediately after titrating the Reichert-Meissl distillate. After rinsing each condenser three times with 15 ml portions of distilled water, similar rinsings were made using 15 ml portions of neutral 95 percent ethyl alcohol. The number of ml of one-tenth normal sodium hydroxide required to neutralize the combined alcoholic rinsings was recorded as the Polenske number. The same base and indicator were used in all of the titrations determining Reichert-Meissl, Polenske and Kirschner numbers.

Kirschner Determinations. All Kirschner numbers were determined according to the method described by the Association of Official Agricultural Chemists (2) except that instead of using one-tenth normal barium hydroxide, one-tenth normal sodium hydroxide was used. The Kirschner number was calculated according to the following formula after the result was corrected for titration obtained in a blank determination.

$$K = \frac{A \times 121\ (100 + B)}{10{,}000}$$

A = corrected Kirschner titration and B = number of ml of standard alkali solution required to neutralize the 100 ml Reichert-Meissl distillate.

First Phase of Experimental Procedure

The first phase was designed to determine if the method used to extract butterfat from ice cream had any influence upon the Reichert-Meissl, Polenske and Kirschner numbers of butterfat. This was important since detection of foreign fats in ice cream, in phase two of the project, was to be based upon these procedures.

The first phase consisted of using methods of butterfat extraction from ice cream described by Golding (8), Epple and Horral (7) and a method developed during the preliminary work, which shall be referred to as the Minnesota reagent churning method.

Four lots of ice cream mixes were made according to the method previously described. A control, consisting of butter churned from cream used in the mixes, was used in each lot. After the mixes were frozen, butterfat from a random pint of ice cream from each lot was extracted by the Golding method (8) and the Minnesota reagent churning method. It was also planned to use the Epple and Horral (7) method. However, a gelatinous mass, which frequently formed in the separatory funnel, prevent-

ed the normal separation of the ethereal layer. Consequently, it was used only to extract butterfat from the first lot of ice cream.

The Reichert-Meissl, Polenske and Kirschner numbers were determined in replicate of four from the butterfat extracted by each procedure. Values were then compared to determine if either the reagent or the procedure changed the values of the butterfat during extraction.

Golding Method of Extraction. Golding (8) proposed a gravimetric method for determining butterfat in dairy products which could be used instead of the Babcock butterfat test. The fat was separated from the serum by churning, following the addition of a variable amount of reagent, dependent upon the amount and type of product being tested. The reagent consisted of a mixture of 60 percent ammonium hydroxide, 28 percent n-butyl alcohol and 12 percent ethyl alcohol of 90 percent strength.

One pint of ice cream from each of the four lots of ice cream was melted, weighed and placed into a four liter Erlenmeyer flask. An amount of reagent equal in volume to the weight of the melted ice cream was added. After shaking vigorously for ten seconds, another portion of the reagent, half as large as the first, was added in addition to an equal amount of distilled water. The contents were shaken for three minutes and enough water, at 20° C. was added to completely fill the flask. The butterfat, which had been released by churning and had

floated to the top of the flask following the addition of the cold water, was then removed with a butter trier and placed into small glass jars which could be placed into a centrifuge. The butterfat was melted in an oven at 50 to 60° C. to separate the butter oil from small quantities of reagent. After centrifuging, removing the butter oil from the upper layer by siphonation, and filtering it through medium retentive, 11 centimeter size paper, the butter oil was heated in a Mojonnier oven for one hour at 135° C. in a vacuum of 22 inches of mercury to remove traces of volatile material. Four determinations of Reichert-Meissl, Polenske and Kirschner numbers were conducted simultaneously. This was repeated for each of the four lots of ice cream used in phase one of the experiment.

Epple Method of Extraction. A method of extracting butterfat from ice cream for the purpose of analysis was reported by Epple and Horral (7). The procedure consisted of extracting butterfat from approximately 200 grams of melted ice cream which was placed in a 500 ml separatory funnel. The same reagents as are used in the Mojonnier and Roese-Gottlieb butterfat determination procedures were added to the separatory funnel with the following exception. Epple and Horral (7) stated that more efficient extraction was obtained by the use of glacial acetic acid than ammonium hydroxide. As stated previously this method was employed only on the first lot of ice cream due to difficulties encountered.

Minnesota Reagent Churning Method. Preliminary work indi-

cated that Minnesota reagent (22) added to melted ice cream would release the butterfat upon shaking or churning. The method developed was similar to the Golding (8) procedure except that the Minnesota Babcock (22) reagent was substituted for the Golding reagent. A pint of ice cream from each of the four mixes was melted, weighed and placed in a four liter Erlenmeyer flask. A volume of Minnesota Babcock reagent, approximately 1.67 times the weight of the melted ice cream, was added. After shaking the flask vigorously for three minutes, enough cold water at 20° C. was added to fill the flask. The remaining procedure was similar to that previously described under the Golding method of extraction. After making many determinations it was discovered that heating in the Mojonnier oven for one hour was not justified. Consequently, the chemical values were determined for the butterfat immediately after filtering. This procedure was repeated for each of the four mixes in phase one.

Second Phase of Experimental Procedure

The second phase of experimental procedure consisted of preparing five ice cream mixes, each of which had one-third of the total fat supplied by some foreign fat or oil. The fat was removed from the ice cream by the Minnesota reagent churning method and Reichert-Meissl, Polenske and Kirschner numbers were determined in duplicate on three extractions from each mix.

Preparation and Analysis of Foreign Fat Mixes. Five 80 pound mixes were made. Each mix consisted of 15 percent sugar, 11 percent serum solids, 0.3 percent gelatin and 12 percent fat. Two-thirds of the total fat in each mix was supplied by milk and cream obtained from milk produced by the college herd. Foreign fat was used for the remaining one-third of the fat. Mix one to five contained the following foreign fats respectively; mix one, Hoonstar; mix two, cottonseed oil; mix three, Primex; mix four, Sweetex and mix five, Crisco. Hoonstar, a coconut oil and cottonseed oil are oils, while Primex, Sweetex and Crisco are hydrogenated vegetable oils. The Proctor and Gamble Company of Cincinnati, Ohio, supplied the Hoonstar, Primex and Sweetex fats. Cottonseed oil and Crisco were obtained locally.

The constituents of each mix were placed in a small jacketed copper kettle, which was designed for heating or pasteurizing, and heated to 160° F. Agitation was applied during heating. After each mix had reached the pasteurization temperature it was removed from the pasteurizer and homogenized at 1000 and 3000 pounds pressure per square inch in a two-stage Gaulin homogenizer. The mixes were then cooled to 40° F., stored for 24 hours and frozen in a 40 quart direct expansion batch freezer. Random pint samples were taken from each frozen lot for analysis of fat, and body and flavor scoring.

Three fat extractions were made on each of the five frozen mixes by the Minnesota reagent churning method and the fat was analyzed in duplicate for Reichert-Meissl, Polenske and Kirschner

numbers as previously described. About 250 grams of ice cream was used for each extraction. Similar values were also obtained for butter which was churned from a representative quart sample of the cream used in the five experimental mixes.

Flavor, Body and Texture Scoring. Two days after the five mixes were frozen, they were scored in an impartial manner by four experienced ice cream judges to determine if the ice cream containing foreign fats could be identified organoleptically. The judges were not familiar with the history of the adulterated ice cream and were not allowed to converse or discuss them while scoring was in progress. Only flavor, and body and texture scores were recorded. The control was regular college ice cream which contained no foreign fat.

EXPERIMENTAL RESULTS

Experimental results are listed under the following headings: yield of extraction studies, butterfat extraction studies, mixed fat extraction studies and flavor, body and texture scores.

Yield of Extraction Studies

Experimentation during the preliminary phase indicated that butterfat could be extracted from ice cream by shaking or churning melted ice cream with Minnesota Babcock reagent. This prompted yield of extraction studies to determine what ratio of

reagent to ice cream was most satisfactory. Two trials appeared sufficient to draw conclusions. Examination of data in Table 1 indicate that the most satisfactory amounts of Minnesota reagent to use for extracting fat from ice cream are approximately 1.6 parts of Minnesota reagent to 1.0 part of ice cream. However, efficiency of extraction also depended upon the length of time the sample was shaken. When the ratio of Minnesota reagent to ice cream was 1.6 to 1, three minutes churning time was enough to insure satisfactory extraction. Ratios of 1.0 to 1.0 removed approximately one-half of the butterfat regardless of length of churning time. Data indicate a ratio of 2.0 to 1.0 was not necessary.

Table 1. Yield of extraction procedures in which two churning periods and varying amounts of Minnesota reagent were used.

Ml of Minn. reagent used	Ratio of reagent to ice cream[a]	Churning time in minutes	Recovered fat in grams		
			Trial 1	Trial 2	Av.
50	2-1	3	1.33	1.16	1.27
50	2-1	6	2.16	1.76	1.96
40	1.6-1	3	1.99	1.64	1.81
40	1.6-1	6	2.22	1.74	1.98
30	1.2-1	3	2.49	1.11	1.75
30	1.2-1	6	1.73	1.29	1.50
20	0.8-1	3	0.58	0.78	0.68
20	0.8-1	6	1.08	0.97	1.02
10	0.4-1	3	0.06	0.27	0.17
10	0.4-1	6	1.08	0.75	0.91

[a]25.00 grams of ice cream were used.

Butterfat Extraction Studies

This section presents the results of the first phase of the experiment which was a comparison of chemical values of butterfat extracted from ice cream by three different methods. Reichert-Meissl, Polenske and Kirschner numbers were determined for butterfat extracted from four lots of ice cream by the Golding method (9) and the Minnesota reagent churning method. It was also planned to extract butterfat from the four lots of ice cream by the method described by Epple and Horral (7). However, a gelatinous mass which frequently formed in the separatory funnel prevented normal separation of the ethereal layer. Consequently, only two successful extractions were obtained which was enough butterfat for five Reichert-Meissl determinations.

A comparison of Reichert Meissl numbers of butterfat extracted from ice cream by three different methods are shown in Table 2.

There was no statistical significance of differences between Reichert-Meissl numbers of the butter control and butterfat extracted by the Minnesota reagent churning method. The differences of Reichert-Meissl numbers of butterfat extracted by the Golding method and the Epple method compared with the butterfat control were significant at less than the one-tenth percent level. Values of t for the three methods were as follows: Golding method $t_{30} = 5.5$, Epple method $t_{19} = 5.1$,

Table 2. Reichert-Meissl numbers of butter controls and butterfat extracted from four lots of ice cream by three different methods.

Mix no.	Method of extraction	Determination of Reichert-Meissl numbers 1	2	3	4	Av.	Standard deviation	Standard error of mean
1	Butter control	29.19	29.06	29.10	29.74	29.28	.32	.16
	Minn. reagent	29.29	29.04	29.33	29.99	29.16	.12	.12
	Golding method	28.12	28.37	28.18	28.41	28.29	.14	.07
	Epple method[a]	29.40	29.86	31.62	31.39	30.70	.37	.39
2	Butter control	28.95	29.77	29.10	29.21	28.98	.18	.09
	Minn. reagent	29.23	29.18	29.10	29.85	29.09	.16	.08
	Golding method	28.77	29.10	28.71	28.61	28.79	.21	.11
3	Butter control	28.07	28.75	28.05	28.09	28.03	.16	.08
	Minn. reagent	28.61	28.07	28.79	28.60	28.52	.31	.16
	Golding method	27.94	27.82	27.86	27.72	27.89	.15	.08
4	Butter control	30.07	29.78	29.93	29.80	29.95	.20	.10
	Minn. reagent	29.97	29.75	29.81	29.94	29.85	.09	.05
	Golding method	28.51	28.01	28.32	29.15	28.62	.49	.24

[a] Results are based upon five trials: fifth was 31.31.

Minnesota reagent churning method $t_{30} = 0.53$. Statistical analysis methods were those of Snedecor (20). The great variance in the results of the Epple procedure was probably due to difficulties encountered with beginning procedures.

The arithmetic mean of Reichert-Meissl numbers of butterfat obtained from cream which was used in the four mixes was 29.24. This was almost identical to 29.15 which was the arithmetic mean Reichert-Meissl number of the butterfat extracted by the Minnesota reagent churning method. Butterfat from ice cream extracted with Golding consistently had lower Reichert-Meissl numbers than the control, and for five determinations, Reichert-Meissl numbers obtained by the Epple method were higher than the control.

Table 3. Polenske numbers of butter and butterfat extracted by two different methods from four lots of ice cream.

Ice cream lot number	Method of extraction	Determination of Polenske numbers 1	2	3	4	Mean
1	Butter control	2.15	2.31	2.10	2.02	2.15
	Minn. reagent	2.32	2.23	*	*	2.30
	Golding method	2.11	1.78	2.00	1.92	1.95
2	Butter control	2.27	2.56	2.95	1.35	2.43
	Minn. reagent	2.47	3.07	2.45	2.05	2.51
	Golding method	2.60	1.95	2.42	2.23	2.30
3	Butter control	2.30	2.07	2.30	2.10	2.19
	Minn. reagent	2.27	2.39	2.30	2.10	2.26
	Golding method	2.21	2.63	2.07	1.75	2.17
4	Butter control	1.90	2.19	1.91	1.63	1.91
	Minn. reagent	1.56	1.56	1.57	2.10	1.70
	Golding method	1.65	1.61	1.45	1.46	1.59

*Accident prevented completion of data.

The arithmetic mean of Polenske numbers of the butter control from each of the four lots was 2.17, while the arithmetic mean of Polenske numbers of butterfat extracted by the Minnesota reagent churning method was 2.10. However, the Golding method of extraction was not as satisfactory as Polenske numbers were lower in every mix compared to the butter control. They averaged 2.00 which was 0.17 lower than the butter control and 0.19 lower than butterfat extracted by the Minnesota reagent churning method.

Table 4. Kirschner numbers of butter and butterfat extracted by two different methods from four lots of ice cream.

Lot no.	Method of extraction	Determination of Kirschner numbers 1	2	3	4	Mean
1	Butter control	23.14	24.36	23.22	23.71	23.61
	Minn. reagent	*	*	*	*	*
	Golding method	22.91	22.50	23.62	23.69	23.16
2	Butter control	24.32	24.34	25.01	25.01	24.67
	Minn. reagent	25.58	25.71	24.77	25.07	25.28
	Golding method	25.12	24.23	24.57	24.24	24.50
3	Butter control	25.10	24.39	24.73	24.34	24.77
	Minn. reagent	23.96	*	24.78	24.95	24.53
	Golding method	23.44	*	24.51	24.51	23.97
4	Butter control	24.60	25.16	24.52	24.43	24.69
	Minn. reagent	23.96	24.07	23.60	24.76	24.06
	Golding method	*	*	*	*	*

*Accident prevented completion of data.

Since the Kirschner number approximates the butyric acid content in addition to small amounts of caproic acid, one would expect the Kirschner numbers from butterfat extracted by the

Minnesota reagent churning method and the Golding reagent to vary in similar proportions as the Reichert-Meissl numbers varied. The results listed in Table 4 show this relationship existed. The butterfat, obtained from four lots of ice cream by the Minnesota reagent churning method, had an average Kirschner number of 24.63, compared to an average of 24.44 for the butter control. The butterfat extracted by the Golding reagent had an average Kirschner number of 23.77.

The results shown in the first phase indicate that the reagent or procedure used in the Golding method of extraction lowers the Reichert-Meissl number about one point lower than the butterfat control. This would be important in border-line cases where the butter had a Reichert-Meissl number of about 24. The method of extraction would then determine whether the sample could be called adulterated or be considered as butterfat. The Minnesota reagent churning method had insignificant effects upon the butterfat during extraction. Difficulties encountered in performing the Epple method eliminated it as a method of extraction during these studies. In the second phase of the experiment, only the Minnesota reagent churning method was used to remove fat in studying mixtures of fats in ice cream.

Mixed Fat Extraction Studies

Reichert-Meissl, Polenske and Kirschner numbers were determined on fat extracted by the Minnesota reagent churning method

from five lots of ice cream containing foreign fats. In each lot a different foreign fat was substituted for one-third of the butterfat. Three extractions were made from each lot. The same three chemical numbers were determined for each of the five foreign fats and for the butterfat which was used in all the mixes.

An examination of chemical numbers of the foreign fats listed in Table 5 indicates Reichert-Meissl, Polenske and Kirschner values of approximately one or less, except the fat Moonstar. The abnormally high Polenske number of Moonstar compared to butterfat, indicated that the Polenske process would be satisfactory for detecting coconut oil in butterfat providing no other foreign fat or oil are present. However, a small percentage of coconut oil could be blended with a fat having a Polenske number lower than butterfat. The resultant mixture would then have a Polenske number which would be normal for butterfat. The Kirschner and Reichert-Meissl numbers of Moonstar were somewhat higher than the other fats used. However, they did not approach the corresponding values for normal butterfat.

Since the Reichert-Meissl and Kirschner numbers of the foreign fats studied are low in relation to butterfat, a mixture of a foreign fat with butterfat should lower the Reichert-Meissl and Kirschner numbers in proportion to the amount of foreign fat added. Results listed in Table 6 show this relationship existed. The Reichert-Meissl numbers were approximately one-

Table 5. Reichert-Meissl, Kirschner and Polenske numbers of butterfat, Moonstar, cottonseed oil, Primex, Sweetex and Crisco selected for use in five lots of ice cream.

Product	Determination of Reichert-Meissl numbers				
	1	2	3	4	Av.
Butterfat	29.82	29.71	29.71	29.55	29.70
Moonstar	8.08	7.96	7.97	8.06	7.99
Cottonseed oil	1.10	1.10	1.21	0.98	1.15
Primex	0.85	0.71	0.71	0.71	0.75
Sweetex	1.43	*	1.52	1.00	1.32
Crisco	1.05	*	0.95	0.77	0.92
			Polenske numbers		
Butterfat	2.45	2.50	2.00	2.20	2.29
Moonstar	13.35	13.22	13.19	13.50	13.46
Cottonseed oil	1.55	0.95	0.90	0.95	1.06
Primex	0.85	0.68	0.65	0.90	0.78
Sweetex	0.45	*	0.65	0.45	0.52
Crisco	0.54	*	1.29	0.55	0.79
			Kirschner numbers		
Butterfat	24.73	24.81	24.84	24.82	24.80
Moonstar	3.54	3.09	3.40	3.44	3.32
Cottonseed oil	0.76	0.68	0.65	0.79	0.72
Primex	0.67	0.61	0.67	0.68	0.66
Sweetex	0.21	*	0.58	0.73	0.71
Crisco	0.73	*	0.67	0.61	0.67

*Omitted because of incorrect procedure.

Table 6. Reichert-Meissl, Kirschner and Polenske numbers of butterfat from five lots of ice cream: each lot contained two-thirds butterfat and one-third foreign fat.

Ice cream containing	Extraction 1		Extraction 2		Extraction 3		Av.
	1	2	1	2	1	2	
Reichert-Meissl numbers							
Hoonstar	23.27	23.27	24.00	23.96	23.43	23.60	23.59
Cottonseed oil	21.60	20.90	20.91	21.10	21.10	21.34	21.14
Primex	19.96	19.67	*	*	20.13	20.13	19.95
Sweetex	19.34	19.36	19.20	19.36	19.26	19.36	19.30
Crisco	29.23	19.14	19.11	19.10	19.50	19.33	19.40
Kirschner numbers							
Hoonstar	19.96	19.14	20.73	20.12	20.36	19.74	19.93
Cottonseed oil	18.16	18.19	18.01	17.97	17.90	18.13	18.06
Primex	16.08	16.30	14.53	15.08	18.25	19.61	16.47
Sweetex	16.55	16.22	16.36	*	16.49	16.29	16.48
Crisco	16.69	16.65	16.90	15.94	16.31	16.65	16.64
Polenske numbers							
Hoonstar	4.25	4.30	4.95	4.50	4.10	4.20	4.38
Cottonseed oil	2.60	*	2.62	2.50	2.70	2.75	2.69
Primex	1.60	1.60	1.90	1.70	1.78	1.56	1.69
Sweetex	2.40	2.45	1.90	1.57	1.47	1.33	1.60
Crisco	2.30	1.70	1.36	1.32	1.65	1.35	1.61

*Data not available.

third lower than the Reichert-Meissl number of the butterfat which was used in the mixes. The extracted fat from the mix in which Crisco was substituted for one-third of the butterfat, had an average Reichert-Meissl number of 19.40, while the corresponding numbers of fat from Sweetex and Primex mixes also ranged between 19 and 20. The fat extracted from the mix in which Moonstar furnished one-third of the total fat had an average Reichert-Meissl number of 23.50. These differences can be accounted for since the coconut oil, Moonstar, had a Reichert-Meissl number of 7.99, while Crisco, Primex and Sweetex had Reichert-Meissl numbers of approximately one or less.

Kirschner numbers of the extracted fat from the adulterated ice cream were also about one-third lower than the Kirschner number of the butterfat. The original butterfat had a Kirschner number of 24.30 and the Kirschner numbers of the extracted fats from mixes containing Primex, Sweetex and Crisco were 19.95, 19.30, and 19.40, respectively.

The Polenske number of the oil, Moonstar, was 13.46 compared to 2.29 for the butterfat. When Moonstar was substituted for one-third of the total fat, it raised the Polenske number of the resultant mixed fats to 4.30. Primex, Sweetex and Crisco had lower Polenske numbers than butterfat. When any of these fats were substituted for one-third of the butterfat in a mix, the Polenske number of the fat, subsequently extracted, was lowered about one-third.

Thus, the addition of foreign fats in ice cream influences

the Reichert-Meissl and Kirschner numbers of the butterfat in proportion to the amount of foreign fat added. The Reichert-Meissl and Kirschner numbers of the foreign fat must also be taken into consideration. However, mixtures of foreign fats may be added to ice cream in such proportions that the Polenske number will correspond to normal butterfat.

Flavor, Body and Texture Scores

Results of data which are shown in Table 7 indicate that it was impossible to identify by organoleptic methods all of the mixes which contained foreign fats. Each lot of adulterated ice cream had a different foreign fat substituted for one-third of the butterfat. Flavor scores of lot number four, which contained Sweetex, a hydrogenated vegetable oil, presented interesting results. None of the four judges were able to differentiate it from the ice cream control. The mix containing Crisco as a source of part of the fat was also difficult to differentiate from ice cream. The mix containing cottonseed oil scored the lowest in flavor.

Body and texture scores of all of the lots ranged from 26.0 to 29.0. All of the lots containing foreign fats, except four and five, were criticized for having a peculiar, characteristic flavor which was described as oily or shortening flavoring. Other judges detected caramel flavors.

It is doubtful if the average consumer could have detected

Table 7. Flavor, body and texture scores of ice cream and five lots of ice cream adulterated with foreign fats.

Lot	Fat	Type of scoring	Judge 1	Judge 2	Judge 3	Judge 4	Av.
1	Moonstar	flavor	38.0	37.0	37.0	37.0	37.2
		body & text.	29.0	26.0	28.0	27.0	27.5
2	Cottonseed	flavor	37.0	36.0	37.0	35.5	36.4
		body & text.	29.0	28.0	29.5	29.0	28.4
3	Primex	flavor	38.5	37.0	37.0	36.0	37.1
		body & text.	29.0	28.0	28.0	28.0	28.2
4	Sweetex	flavor	40.0	40.0	39.0	39.0	39.5
		body & text.	29.0	28.5	29.0	29.0	29.0
5	Crisco	flavor	38.5	39.0	40.0	38.0	38.9
		body & text.	29.0	29.0	29.0	28.0	28.8
6	Butterfat	flavor	39.0	40.0	37.0	39.0	38.8
		body & text.	29.5	28.5	29.5	29.0	28.6

any abnormality in any of the adulterated ice cream mixes except the one containing cottonseed oil.

DISCUSSION

Investigators (7, 9, 13, 14) have reported various methods of extracting butterfat from dairy products. However, no references were found in which butterfat was extracted from a quantity of ice cream as large as one pint.

A part of this experiment was confined to developing a rapid, inexpensive method of extracting butterfat from ice cream which removed the butterfat in such a manner that Reichert-Meissl, Polenske and Kirschner numbers of the extracted butterfat compared favorably with the same numbers of the original butterfat before processing. A churning method was desired because it is possible to adapt it to variable amounts of ice cream. After it was found that Minnesota Babcock reagent would extract butterfat from ice cream without applying heat, merely by shaking or churning, a series of trials indicated that the amount of reagent most satisfactory to use was a volume approximately 1.6 times as large as the sample of ice cream from which the butterfat was to be extracted. Butterfat was extracted from pint samples of ice cream in a four liter Erlenmeyer flask. Reichert-Meissl, Polenske and Kirschner numbers of butterfat extracted from ice cream by the use of Minnesota Babcock reagent were then found to agree very favorably with Reichert-Meissl,

Polenske and Kirschner numbers of the original butterfat obtained by churning a sample of cream which was used in the ice cream mix. Statistically, the differences of Reichert-Meissl numbers were insignificant.

No studies concerning adulteration of butterfat in ice cream with other animal and vegetable fats were found in the literature. During this study Reichert-Meissl, Polenske and Kirschner procedures were used as a basis for studying adulteration.

In making mixes containing foreign fats, cottonseed oil was used to represent many similar oils which have low Reichert-Meissl, Polenske and Kirschner numbers. The coconut oil, Moonstar, was used because of its relatively high numbers. Crisco, Sweetex and Primex represented hydrogenated vegetable oils. These five foreign fats were believed to be representative of fats and oils used for adulterating ice cream.

After extracting and analyzing fat from ice cream in which one-third of the total fat was supplied by Moonstar, it was evident that had any smaller amounts of Moonstar been used, the Reichert-Meissl number would have been over 24. Approximately 25 percent of the butterfat in ice cream could then have been replaced by Moonstar, and the ice cream still be considered unadulterated in view of present accepted standards of a minimum Reichert-Meissl number of 24 for unadulterated butterfat. However, from data observations, about five percent adulteration with foreign fats, including Moonstar, could be detected by the

Reichert-Meissl process, providing a sample of the butterfat in the ice cream or a similar sample was available for analysis.

Since coconut oil has a higher Polenske number than butterfat, and other fats and oils have lower numbers, the Polenske process is of little value in detecting adulteration of butterfat with mixtures of coconut oil and other fats or oils.

The specificity of the Kirschner method is similar to that of the Reichert-Meissl determination. However, the Reichert-Meissl determination is probably more practical because it requires less than one-half as much time as the Kirschner process.

Data indicate that it was impossible to identify adulteration in all of the frozen products by organoleptic methods because certain foreign fats appeared to have little effect upon flavor, body and texture of ice cream. It appears, therefore, that some chemical determination is necessary for detecting foreign fats in ice cream. The Reichert-Meissl determination can be used by laboratories, providing a chemical balance, condensers and a technician with some knowledge of chemistry are available. Using four condensers and the Minnesota reagent churning method for extracting the butterfat, from 10 to 20 ice cream samples can be analyzed daily by one technician.

At the present time it appears that the Reichert-Meissl procedure is the best method for detecting foreign fats in ice cream, particularly if the origin of the butterfat in the ice cream is known, or if a sample of the butterfat or a similar sample is available for analysis.

SUMMARY AND CONCLUSIONS

1. A convenient, rapid method of extracting butterfat from ice cream for the purpose of determining Reichert-Meissl, Polenske and Kirschner numbers was developed.

2. Results of 15 extractions from five lots of ice cream adulterated with different fats and oils indicated that the method of extraction was also satisfactory for removing mixtures of fats from ice cream.

3. Organoleptic methods of detecting adulteration are unreliable since some fats appear to have little influence upon flavor, body and texture when substituted for one-third of the butterfat.

4. The Polenske procedure is of little value in detecting ice cream adulterated with mixtures of coconut oil and other fats or oils because with certain proportions, the Polenske number may be similar to the Polenske number of butterfat.

5. The Kirschner and Reichert-Meissl procedures were similar in specificity as tests for adulteration. However, because more time is required for the Kirschner process, it probably is not warranted.

6. The Reichert-Meissl procedure for detecting adulteration of ice cream with foreign fats may be used effectively when accompanied by information concerning the origin of the butterfat used in the adulterated ice cream. When a sample of the original butterfat from adulterated ice cream, or a similar

sample, can be obtained for analysis, it is possible to detect approximately five percent adulteration.

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