# A RAPID AND ACCURATE GRAVIMETRIC METHOD FOR THE DETERMINATION OF FAT IN ICE CREAMS

A THESIS SUBMITTED TO THE FACULTY OF THE GRADUATE SCHOOL OF THE UNIVERSITY OF MINNESOTA BY GUY GEORGE PARKIN IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE, JUNE, 13, 1913.

## PURPOSE.

The purpose of this thesis was to work out a rapid and accurate method for determining butter fat in ice creams and to show by comparison that a larger percent of fat can be obtained by this method than by the Modified Babcock Methods, and that this method gives higher results, when compared with the Roese-Gottlieb Method, which is the provisional method for use in ice cream.

The output of the ice cream industry of the United States is worth considerably more than $100,000,000 annually. The trade in this product has increased enormously during the last few years. The profits derived from the use of cream in the making of ice creams as compared with that used in the making of butter is fully double. Many creameries are so located that they could enter upon this business to great advantage. The entire product of several creameries are being thus used today. The vastness of the ice cream industry has brought it at the present time to the attention of the

Federal and State inspection officials which has necessitated the use of more rapid and accurate control methods in determining the quantity of fat in the ice cream products.

## CLASSIFICATION OF ICE CREAMS.

Two general classes of ice cream are recognized, the Plain (raw) and the French (cooked custard).

## ESSENTIAL CHARACTERISTICS OF ICE CREAM.

The flavor is influenced by the fat contents of the cream, by its freedom from contamination, by a low cream acidity, by the addition of minute quantities of common salt, and by the ripening and aging of the cream.

A good body depends upon the fat contents, upon the aging and the thorough cooling of the cream and sometimes upon the use of fillers.

A fine texture depends upon the richness of the cream, the proper manipulation in the freezing process and the aging of the cream. If the product is not to be used at once the texture is influenced by the use of gelatinoid binders.

Swell or overrun in ice cream is caused by the incorporation of air into the cream and is affected by viscosity of the cream, by the rate of freezing, and particularly by the length of time it takes for the cream to drop from 34° F. to 29° F. and by the speed of the stirring mechanism.

The richness or leanness of the cream within working limits as well as the use of gelatin, gum tragacanth or other binders, has but little effect on the quality of the final product.

The time element in the freezing of ice cream depends upon the initial temperature of the cream, upon the rate of flow and the temperature of the brine, upon the proportion of ice and salt used, upon the fineness and coarseness of the fragments of ice and the particles of salt, and upon the amount of sugar used in the cream.

## THE CREAM.

The fat content, temperature and acidity of the cream, the method of handling, of separating, pastuerizing and homogenizing, all have effect on the final pro-

duct. A clean cream is essential. Neither a very rich nor a too thin cream should be used but a cream containing about 22 percent of fat. A days keeping at a low temperature improves the cream as the fat globules harden and a better body is obtained. A low acidity is desirable. Aged pasteurized cream may be used to improve the quality. The homogeneity of the cream greatly increases its viscosity and improves the body, texture and general creaminess of the final product. There is no essential difference between the ice cream product made from centrifugal and gravity creams. Condensed milk may be used to improve the body and smoothness of the product.

FILLERS AND BINDERS.

A filler is used to give body, a binder to prevent coarse crystallization, when ice cream is kept for a day or longer. Starch, flour, eggs and rennet are used as fillers. Gelatin, gum tragacanth and ice cream powders are used as binders with good satisfaction. The legality of fillers and binders vary in different states.

There appears to be many arguments regarding the

advisability of the use of binders in commercial ice cream. The adverse arguments are:- that inferiority and age are thus concealed, the swell unduly augmented, the use of low grade materials encouraged, unsanitary holding conditions favored and adequate food control rendered difficult. Those advanced in favor of their use are:- that they prevent granulation and consequent deterioration, discourage the return and reusage of unsold goods and assist trade regulation.

## TYPES OF FREEZERS.

Several types and variants therefrom are in use. The principal ones being vertical-batch-ice, vertical-batch-brine, horizontal-batch-brine and horizontal-continuour-brine.

## THE FREEZING PROCESS.

The successful prosecution of the freezing process depends largely upon the salt and ice, their degree of fineness and the proportion of each used, or the temperature and rate of flow of brine, upon the speed of the dasher, upon the stopping of the freezer at the proper

time or the drawing off of the brine at the proper temperature and upon the careful attention of the transference and the holding and the shipping of the product. Certain abnormalities occasionally occur as for instance churning in the freezer, which may be avoided by cooling the cream in advance and the use of a slow initial speed.

A softened ice cream may be rehardened but the product is generally unsatisfactory, or it may be refrozen but the process has its disadvantages. It is better to make such products into butter. The fat globules of a partly melted product rise in the holding can rendering the top portion richer than the bottom. Fat and fillers have no effect on the freezing point but sugar depresses it.

## HISTORICAL RESUME OF ICE CREAM AND THE METHODS USED IN ITS FAT EXTRACTION.

Jour. of Soc. of Chem. Ind., Vol. 28, P. 284, contains the following statements:- It is a fact well known to ice cream manufacturers and amply proven by experience that ice cream made without eggs, gelatin or

some similar colloidal ingredient is gritty, grainy or sandy or becomes so on standing, whereas ice cream made with small amounts of colloids possesses that rich, mellow, velvety texture so much in demand. Here the colloid acts as an inhibitor of crystallization or practically speaking, as a preserver of texture. A very misleading impression is given by some official food chemists referring to gelatin in ice cream as a filler, therefore conveying the idea that it is an inferior ingredient and added to cheapen the product but as gelatin is expensive and only half a percent is used, such a view is erroneous.

Arch. Farmacol, Sper., Sci., App. 7, 1-4, contains the examination of a new egg preparation "Ice Cream Powder". The powder on examination was found to contain oat and wheat starch and Martins Yellow.

Z. Chem. Ind. Kolloide, 5, 101-3; See C.A. 3, 1315, contains the following statements regarding gelatin: Gelatin acts as an irreversible colloid in preventing the coagulation of casein and, since physicians recommend its use in milk for feeding infants, it must be without bad

effects.

Lab. of Inland Rev., Dept., Ottawa, Can., Bull. 190, By A.M. McGill. This bulletin consists of the results of the examination of 129 samples of ice cream purchased in various parts of Canada. Of these samples 64 were below the U.S. standard of 14 percent fat.

Bull. 218, Lab. Inland Rev. Dept., Ottawa, Can. This bulletin contains definitions and standards for ice cream now made legal in Canada, which are for plain ice creams 14 percent butter fat and for nut ice creams 12 percent butter fat.

Bull. 162, Lab. Inland Rev., Ottawa, Can., consists of the results of the examination of 80 samples of ice cream. Of these 40 contained above 14 percent of fat and 40 less than that amount. Of the latter, 12 samples contained less than 10 percent of fat and only one sample contained nuts. Most of the 80 samples were uncolored and of those colored only two were dyed with coal tar products. The others were colored with genuine fruit juices.

Notices of Judgment U.S. Dept. of Agriculture:

No. 1450, Adulteration of Ice Cream because it was low in milk fat.

No. 1446, Adulteration of Chocolate Ice Cream and Vanilla Ice Cream because gelatin had been substituted in part for milk fat.

Food Inspection Decision, 132, U.S. Dept. of Agric.:- Investigations have shown that there has lately come into use an apparatus known as an "homogenizer", which so disrupts the globules of fat that a whole milk when so homogenized does not permit the separation of the cream by the ordinary gravity methods. Butter or other fats, when mixed with skimmed milk and homogenized form a product from which the butter fat does not separate on standing and which resembles whole milk in its physical characteristics. These investigations have further shown that butter and skimmed milk are passed through the homogenizer to form a so called "cream", which is used in place of genuine cream in the manufacturing of ice cream.

These investigators are of the opinion that

skimmed milk and butter fat in appropriate proportions passed through the homogenizer is not entitled to the name of "milk" or "cream" and that the product made from a homogenized butter or skimmed milk can not properly be called "ice cream".

Fifth Biennial Report of Idaho State Dairy, Food and Sanitary Inspector and State Chemist, 1911-12. This report contains Idaho Standards for ice creams, which are for plain ice creams not less than 14 percent butter fat and nut ice cream not less than 12 percent. Fillers are decried as unfit for food. The use of condensed milk and skimmed milk is not tolerated in manufacturing ice cream. Objections to the use of homogenizers are given. Seventeen samples were analyzed, of which four contained "fillers" or "binders" and one contained less than 14 percent butter fat.

Bacteriological Control of Ice Creams (Production and Handling of Clean Milk by Winslow):- Tyrotoxicon is a ptomaine or chemical product due to the splitting up or putrefaction of milk products by bacteria of the

colon group. In ice cream the number of germs is often many times that found in milk or cream. This follows because the cream is often pasteurized, which kills the harmless lactic acid bacteria and then often kept at a low temperature, which allows other germs to grow, which may be harmful. On the examination of 263 samples of ice cream, 26 million germs were found on an average in one fourth teaspoonful. Streptococci are found twice as frequently as in milk. Storing ice cream does not decrease the number of germs but renders the cream much more dangerous by allowing time for the development of ptomaines. The danger in commercial ice cream lies in uncleanly apparatus, unfit raw material and the storing of the finished product. When made from fresh, unpasteurized, clean cream and consumed within a short time, ice cream is harmless. About twenty-five percent of market cream contains Tubercle Bacilli, which are not killed during the making of ice cream. Therefore ice cream should be made from tuberculin tested cows or from freshly pasteurized cream. Ice cream can also be the

carrier of typhoid germs, as freezing does not kill them.

Hoards Dairyman, Dec., 4, 1908:- The following rules regulate the sale of ice cream, exposed for sale or kept with the intention of selling within the City of Chicago after Jan., 1, 1909:

1. Ice cream shall not contain more than 200,000 bacteria per c.c. from May 1st to Sept. 30, and not over 100,000 per c.c. between Oct. 1st and Apr. 30th.

2. An original package of ice cream exposed at room temperature for 48 hours and stoppered with a Sterile cotton plug shall not show evidence of putrefaction.

3. To 5 c.c. of pasteurized ice cream add 2 drops of a 20% solution of paraphenylenediamine and one drop of a 2% solution of hydrogen peroxide and agitate. Not more than a tinge of blue should develop after standing 30 seconds.

Journal Amer. Chem. Soc., Vol. 29-1907, P. 1632, by Chas. D. Howard:- With the recent anactment of standards for ice creams by several states, the analysis of ice creams is just beginning to receive the attention of food inspection chemists. As literature seems to contain no specific methods, the following (in part simple modification of those generally used) are offered as having been successfully used at the New Hampshire Lab. of Hygiene.

In analyzing ice creams it is necessary to determine the fat as soon as possible after the sample has melted, as on short standing even before appreciable souring takes place, the butter fat tends to separate and when mixed returns to the surface so rapidly, as to render the removal of a representative portion exceedingly difficult and if care is not taken in emulsifying butter clots are apt to form.

Unless approximate results only are required or unless the sample is very largely diluted, the ordinary Babcock Method can not be used. Leach's copper sulphate method for determining fat in condensed milk is not directly applicable, because the fat is not completely carried down by the precipitated proteids and no precipitant has het been found which will completely carry down the fat.

The method finally adopted in this laboratory was a modification of Leach's method in which the fat was dissolved in chloroform and the chloroform removed in a current of steam. In this method eighteen grams of cream

is transferred to a Babcock cream bottle by means of a pipette and accurately weighed. Three c.c. of chloroform are then added and the bottle filled three-fourths full of water and agitated. Ten c.c. of Fehling's copper sulphate solution is then added and mixed and the whole centrifuged for three minutes. (The fat is held in the chloroform layer. In the presence of appreciable quantities of gum tragacanth or gelatin the supernatant liquid becomes turbid. This turbidity may be avoided by the addition of 2 or 3 c.c. of n/10 alkalie.) The resulting copper hydroxide precipitates gum quickly. A glass tube of small bore is inserted in the neck of the bottle and connected with an aspirator and most of the supernatant liquid removed by suction. The bottle is again filled three-fourths full of water, agitated and centrifuged for three minutes, and the supernatant liquid removed as above. A small bored tube provided with a stopcock is then introduced into the bottle and connected with a source of live steam, which is blown through for two to three minutes until all the chloroform has been expelled.

The use of a water bath for the removal of the chloroform is not advisable, as the precipitated proteids retain the chloroform with great tenacity and its removal is not completed after long heating. The contents of the bottle is then cooled and 17.5 c.c. of water added and the fat determined as in the ordinary Babcock method. Care whould be taken to secure a complete solution of the hardened proteid substance.

There is evidence that an "ice cream" is put on the market, the fat of which consists wholly or in part of Oleo or Cottonseed Oil. In separating the oil for a qualitative examination, 30 to 40 c.c. of the cream layer is transferred to a cream bottle, one c.c. of a strong mercuric nitrate solution and 20 c.c. of petroleum ether are added and the whole mixed and whirled in a centrifuge. the ether layer is then removed, washed with water and evaporated, leaving the oil.

Bull. No. 155, Sept., 1910, P. 81, Ver. Agric. exp. Station:- The following method is a modification of an existing method and, although requiring care and close

attention to details in order to give satisfactory results, it gives a clear and clean fat column and is not difficult to carry out.

Eighteen grams of a well melted and mixed sample of ice cream are transferred to a 30% cream bottle and accurately weighed. Four or five c.c. of luke warm water is added to this and 9 to 12 c.c. of ordinary sulphuric acid showly added with agitation, until the solution retains the color of strong coffee when shaken. Four or five c.c. of cool water is then added to check further action of the acid. The test is then completed as in the ordinary Babcock test. If the above directions are carefully followed, particularly those regarding the addition of acid, the fat should rise in the neck of the bottle in a clear, light brown color and with a clean clear separation from the liquid below.

A method for the determination of fat in ice creams as recommended by Paul, taken from the collaborators sheet sent out by Paul in 1912:- In a Büchner funnel wet a 11 cm. filter of loose texture. Cover this with a

thin layer of a fibrous asbestos mixture, being careful to cover the sides as far up as possible. In a 250 c.c. beaker boil 25 c.c. of Soxhlet's copper sulphate solution and add 50 grams of the material, stirring vigorously. Immediately remove the source of heat and filter, washing once or twice with cold water. Suck as dry as possible and dry the cake in an air oven over night. Then grind the cake in a mortar with 25 grams of anhydrous copper sulphate and let stand a few minutes. The mixture should be perfectly dry. This mixture is then placed in a large extractor and extracted with sulphuric ether for sixteen hours, allowing the ether to percolate through the material before commencing extraction. After a sixteen hour extraction the ether is evaporated and the residue dried at 100° C. to constant weight and weighed.

Rep. Ill. State Food Commissioner, 1906, P. 80, contains a method for determining fat in ice cream, which is as follows:- Nine grams of a sample are weighed into a test bottle and 30 c.c. of a mixture of equal parts by volumn of Con. Hydrochloric Acid and 80% acetic acid are added. This mixture is next heated on a water bath, until

the mixture darkens to a rich chocolate brown. Warm water is then added to run the fat up into the neck of the bottle and the whole centrifuged for five minutes. The percent of fat found in the fat column times two gives the direct percent of fat. If charring has interferred with the fat reading, the fat may be dissolved in ether, the ether layer drawn off into a weighed flask and the residue weighed and the percent of fat calculated.

Report of Chemist, Minn. Dairy and Food Dept., 1909, contains a report of West's Method for determining fat in ice creams, which is as follows: Nine grams of the sample are weighed into a Babcock cream bottle and the bottle filled to its neck with a solution of sulphuric acid (1 part acid to 3 parts water) and the temperature of the solution adjusted so as not to be above 50° C. The test bottle is fitted with a rubber stopper carrying two small tubes, one reaching to the bottom of the bottle and the other extending only through the stopper. The bottle is then placed in a centrifuge and whirled from three to five minutes. Any particles of casein or fat that may be lodged in the longer tube are blown out, the

tube connected with an empty flask provided with a stopper and a piece of glass tubing and the sulphuric acid liquor drawn off by suction into the flask. The tube is removed and carefully rinsed off into the bottle with a little hot water, and cold water added until the bottle is about one-half full and the operation completed as in the determination of fat in cream by the usual Babcock method. The reading multiplied by two gives directly the percent of fat in the sample.

Leach's Second Edition, 1911, gives the Roese-Gottlieb method for determining fat in ice creams as follows:- Ten c.c. of a 40% solution of ice cream diluted with 0.5 c.c. of water is measured into a Rohrig tube and shaken with 1.25 c.c. ammonia (2 c.c. if sample is sour). Five c.c. of 55% alcohol is next added and the tube shaken. Twenty-five c.c. of sulphuric ether is then added and the whole thoroughly shaken. The ether layer is allowed to separate and then blown off into a clean, dried and weighed flask. The above extraction is repeated once and the filter paper finally well washed with small

portions of ether, blowing all the ether into the same flask. The ether is finally evaporated on a water bath, the residue dried at 100° C. to constant weight and weighed and the percent of fat calculated.

U.S. Dept. of Agric., Bur. of Chem. Ar. No. 66, contains a description of the Roese-Gottlieb method modified in such a way that the sample of ice cream is weighed directly from the well mixed product instead of making up a 40% solution as recommended in Leach. The remaining part of the method is carried out as in Leach. This method was considered by the writer to give the best results of those methods reviewed above and was used as the comparison method in compiling the following data.

The Harding-Parkin method* as used in determining fat in condensed and desiccated milks was extended and applied in determining fat in the various ice creams and found to give the best results.

The principle of this method is the breaking

*Jour. of Ind. and Eng. Chem., Vol. 5, Feb. 1913.

down of the emulsion by dissolving the proteins in acetic acid, the solution of the fat in a mixture of alcohol and carbon tetrachloride and its extraction with petroleum ether.

The reagents used are:- Acetic acid, 25% by volumn, $CCl_4$, 95% ethyl alcohol and petroleum ether B.P. 50°-70° C.

The apparatus used is:- A 100 c.c. Nesseler Jar or any convenient 100 c.c. extraction tube with two rubber stoppers, one unperforated and used while shaking, the other doubly perforated and bearing blow-off tubes similar to Werner-Schmidt blow-off tubes with a double bulbed rubber air pressure pump for forcing the ether layer out of the extraction jar. A 11 cm. filter paper, funnel and stand, wide mouthed flasks of 50 to 75 c.c. capacity for weighing the fat and a small evaporating dish for holding wash petroleum ether, a petroleum ether wash bottle, a condenser and a drying oven.

The process in detail is as follows:- The ice cream is melted by warming to 50° C. and well mixed without churning to give a uniform sample. Five grams of this

uniform mixture are weighed into a 100 c.c. extraction jar and 5 c.c. of 35% acetic acid added and the contents warmed to about 50° C. by cautiously rotating the jar over a low flame. When the protein has dissolved, 12 c.c. carbon tetrachloride are added, and the jar vigorously shaken for two minutes. Ten c.c. of alcohol are then added and the jar again vigorously shaken. Then 35 c.c. ether is added and the shaking continued one minute longer. The jar is then allowed to stand from one to two minutes, when the separation will be complete, (any emulsion can be broken up with a few drops of alcohol). The blow-off tube is then inserted and the ether layer cautiously blown onto the filter with the double bulbed air pump, filtering the ether into the tared weighing flask. (In blowing off the ether layer, care must be taken that none of the carbon tetrachloride layer is blown off, which may happen if a too close separation of the two layers is attempted. This may be avoided with reasonable care in manipulation.) Five c.c. of carbon tetrachloride are then added to the contents of the jar, which is thoroughly shaken, then 30

c.c. of petroleum ether and the jar again thoroughly shaken and one minute allowed for separation, when the ether layer is blown off with the necessary precautions as above stated. Another addition of 5 c.c. of carbon tetrachloride and 30 c.c. of petroleum ether is made with thorough shaking after each addition and the separation of the ether layer after one minute standing is repeated as in the first blow-off. Five c.c. of petroleum ether are then placed in a small evaporating dish and gently drawn into the jar by sucking on the blow-off tube. After a few seconds this ether, which has mixed with the thin ether layer in the jar is blown off and filtered as before. The filter paper is then well washed with small portions of petroleum ether, about 15 c.c. in all, (three, five c.c. portions), the flask connected with a condenser, the ether distilled off and the flask heated for one hour to constant weight in an oven at 100° C. and then weighed.

During the process, while the jar is being prepared for the second and third blow-offs, the weighing flask is connected with the condenser and most of the

ether distilled off. This shortens the process to about one and one-half hours and permits the use of a 50 to 75 c.c. flask for weighing the fat.

COMPILED DATA.

I.

Data on Chocolate Ice Creams obtained with the Harding-Parkin Method, the Modified Babcock as given in Leach and West's Modified Babcock being used as comparison methods.

| Sample No. | Wgt. of Sample | Harding-Parkin Wgt. of Fat | Harding-Parkin Percent Fat | West Percent Fat | Mod. Babcock Percent Fat |
|---|---|---|---|---|---|
| A | 5 grams | 0.6603 | 13.20 | 11.75 | 12.00 |
| B | 5 grams | 0.6050 | 12.10 | 10.00 | 11.00 |
| C | 5 grams | 0.8152 | 16.30 | 15.00 | 15.50 |
| D | 5 grams | 0.6579 | 13.15 | 12.00 | 12.50 |
| E | 5 grams | 0.6470 | 12.92 | | |
| F | 5 grams | 0.6515 | 13.03* | | |
| G | 5 grams | 0.9157 | 18.31 | | |

*A fourth blow-off gave 0.0003 grams or .00%.

II.

Data obtained with the Harding-Parkin Method. These determinations were made to find out if a fourth blow-off were necessary.

| Sample No. | 1, 2, & 3 Blow-offs Grams Weight | Percent Fat | 4 Blow-off Grams Weight | Percent Fat |
|---|---|---|---|---|
| I | 0.3909 | 13.03 | .0003 | 0.01 |
| II | 0.3303 | 11.02 | .0012 | 0.04 |
| III | 0.3378 | 11.29 | .0009 | 0.03 |
| IV | 0.3601 | 12.00 | .0003 | 0.01 |
| V | 0.2741 | 9.14 | .0006 | 0.02 |

III.

Data obtained on Vanilla Ice Creams with the Harding-Parkin Method. The Roese-Gottlieb Method as given in Cir. 66 and the Babcock Method as given in Leach were used as comparison methods.

| Sample No. | Harding-Parkin Sample Wgt. Grams | Grams Fat | Percent Fat | Roese-Gottlieb Sample Wgt. Grams | Grams Fat | Percent Fat | Mod. Babcock Percent Fat |
|---|---|---|---|---|---|---|---|
| 389 | 3.00 | 0.3375 | 11.25 | 3.00 | 0.3387 | 11.29 | 10.00 |
| 389 | 3.00 | 0.3363 | 11.21 | 3.00 | 0.3312 | 11.04 | 10.00 |
| 389 | 3.00 | 0.3378 | 11.29 | | | | |
| 395 | 3.00 | 0.3307 | 11.02 | 3.00 | 0.3303 | 11.01 | 10.00 |
| 218 | 3.00 | 0.3601 | 12.00 | 3.00 | 0.3540 | 11.80 | 11.00 |
| 218 | 3.00 | 0.3612 | 12.04 | 3.00 | 0.3580 | 11.93 | 11.00 |
| 219 | 3.00 | 0.2741 | 9.14 | 3.00 | 0.2755 | 9.18 | 8.00 |
| 233 | 3.00 | 0.2804 | 9.34 | 3.00 | 0.2787 | 9.29 | |
| *231 | 3.00 | 0.2897 | 9.65 | 3.00 | 0.2850 | 9.50 | 7.00 |
| X | 3.00 | 0.2251 | 7.50 | 3.00 | 0.2250 | 7.50 | 6.00 |
| Y | 3.00 | 0.2338 | 7.79 | 3.00 | 0.2261 | 7.53 | |
| Z | 3.00 | 0.2404 | 8.01 | 3.00 | 0.2350 | 7.83 | |
| I | 3.00 | 0.2438 | 8.12 | 3.00 | 0.2415 | 8.05 | |
| II | 3.00 | 0.2493 | 8.31 | 3.00 | 0.2419 | 8.06 | |
| III | 3.00 | 0.2379 | 7.99 | 3.00 | 0.2394 | 7.98 | |

*Determination was made on a soured sample.

IV.

Data obtained with the Harding-Parkin Method, to illustrate the change in the butter-fat contents of the various parts of an ice cream packer after standing.

The samples were two gallon packers which were well filled and in a fresh frozen condition when first sampled. The second sampling with resulting data were obtained after the packer had stood twenty-four hours

at room temperature without disturbing.

| Sample No. | Fresh Frozen Condition Top % Fat | Middle % Fat | Bottom % Fat | After Standing 24 hrs. Top % Fat | Middle % Fat | Bottom % Fat |
|---|---|---|---|---|---|---|
| I | 12.17 | 12.19 | 12.05 | 12.38 | 12.21 | 12.12 |
| II | 12.66 | 12.34 | 12.63 | 12.61 | 12.57 | 12.41 |

## CONCLUSION.

The method described in this thesis for the determination of fat in ice creams is recommended by the writer for the following reasons:

(1) It is a short practical method readily adapted to commercial work, requiring one to one and a half hours for a complete determination.

(2) It gives a pure fat.

(3) It gives a larger percent of fat when compared with other methods.

(4) It will produce accurate results without a close adherance to the amounts of reagents used.

(5) Its use would be recommended in order that justice may be done the ice cream manufacturer.

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## HARDING-PARKIN METHOD.

## AS APPLIED TO PRODUCTS OTHER THAN ICE CREAMS.

The Harding-Parkin Method was applied to various fibrous products, such as cereals, compounded cereals, fertilizers and chocolates, and found to give a complete and quantitative extraction of the oil.

The method used in the determination of oil in cereals, compounded cereals and fertilizers with one exception, was the same as that applied to ice creams. It was found by the writer that acetic acid would not cut the fibre and liberate the oil quantitatively. Therefore the twenty-five percent acetic acid, as used in the deter-

mination of fat in ice creams, was replaced by hydrochloric acid, specific gravity 1.12, with quantitative results.

The method as applied to chocolate was the same as that applied to cereals. It was found that it was necessary to centrifuge two minutes to obtain the first separation. The following data will show that the Bigelow and Albrech Method gave higher results than the Harding-Parkin Method but in every case the extracted residue in the Bigelow and Albrech Method contained fibrous material which was impossible to filter off and was therefore weighed as oil.

## COMPILED DATA.

### I.

Data obtained with Harding-Parkin Method on Graham Flour. The sixteen hour ether extraction method as given in Leach for cereals, was used as a comparison method.

| Sample No. | Harding-Parkin Flour Grams | Oil Grams | Oil Percent | 16 Hr. Ether Ext. Flour Grams | Oil Grams | Oil Percent |
|---|---|---|---|---|---|---|
| I | 2. | 0.0518 | 2.59 | | | |
| I | 2. | 0.0521 | 2.60 | | | |
| I | 2. | 0.0529 | 2.64 | | | |
| I | 2. | 0.0535 | 2.67 | | | |
| II | 2. | 0.0422 | 2.11 | 2. | 0.0410 | 2.05 |
| III | 2. | 0.0311 | 1.56 | 2. | 0.0300 | 1.50 |
| III | 2. | 0.0310 | 1.55 | | | |
| IV | 2. | 0.0492 | 2.46 | 2. | 0.0482 | 2.41 |
| IV | 2. | 0.0476 | 2.38 | 2. | 0.0475 | 2.37 |
| IV | 2. | 0.0505 | 2.52 | | | |
| IV | 2. | 0.0493 | 2.46 | | | |
| IV | 2. | 0.0494 | 2.47 | | | |
| IV | 2. | 0.0490 | 2.45 | | | |
| IV | 2. | 0.0477 | 2.38 | | | |
| IV | 2. | 0.0498 | 2.49 | | | |
| IV | 2. | 0.0510 | 2.55 | | | |
| IV | 2. | 0.0497 | 2.48 | | | |
| V | 2. | 0.0490 | 2.45 | 2. | 0.0478 | 2.39 |
| V | 2. | 0.0492 | 2.46 | | | |
| VI | 2. | 0.0372 | 1.86 | 2. | 0.0385 | 1.92 |
| VI | 2. | 0.0361 | 1.80 | | | |
| VII* | 2. | 0.0630 | 3.15 | 2. | 0.0590 | 2.95 |
| VIII* | 2. | 0.0504 | 2.52 | 2. | 0.0500 | 2.50 |
| VIII* | 2. | 0.0516 | 2.58 | | | |
| IX* | 2. | 0.0540 | 2.70 | 2. | 0.0533 | 2.66 |
| X 1 | 2. | 0.0760 | 3.80 | 2. | 0.0774 | 3.87 |
| X 1 | 2. | 0.0744 | 3.72 | | | |
| X 1 | 2. | 0.0740 | 3.70 | | | |
| XI 2 | 2. | 0.2128 | 10.64 | 2. | 0.2127 | 10.63 |
| XI 2 | 2. | 0.2371 | 11.85 | 2. | 0.3360 | 11.80 |

*Determination on buckwheat flour.
1 " " compounded cereals.
2 " " fertilizers.

II.

Data obtained with Harding-Parkin Method. These determinations were made to find out if a fourth blow-off were necessary.

| Sample No. | 1, 2, 3 Blow-offs Grams Oil | Percent Oil | 4 Blow-off Grams Oil | Percent Oil |
|---|---|---|---|---|
| 207 | 0.0422 | 2.11 | 0.0013 | 0.06 |
| 203 | 0.0630 | 3.15 | 0.0008 | 0.04 |
| 311 | 0.0516 | 2.58 | 0.0006 | 0.03 |

III.

Data obtained with the Harding-Parkin Method on Chocolate. Bigelow and Albrech Method as given in the Proceedings of A.O.A.C., Bull. No. 137, P. 102-3, as a comparison method.

| Sample No. | Harding-Parkin Method Choco-late Grams | Cocoa Butter Grams | Cocoa Gutter Percent | Bigelow & Albrech Method Choco-late Grams | Cocoa Butter Grams | Cocoa Butter Percent |
|---|---|---|---|---|---|---|
| 173 | 2.00 | 1.0393 | 51.96 | 2.00 | 1.0720 | 53.60# |
| 173 | 2.00 | 1.0341 | 51.70* | 2.00 | 1.0680 | 53.40# |

*The refractive index of this fat was taken to deduce its purity and found to be 1.4575 at 40° C.

#This cocoa butter had a dirty appearance and contained chocolate fibre.

## CONCLUSION.

(1) The Harding-Parkin Method would be recommended for cereals and compounded cereals, because it extracts all the oil in a pure state, requiring only two hours for a complete determination. Other reliable methods require a sixteen hour extraction.

(2) The Harding-Parkin Method would be recommended for chocolates because it extracts all the oil in a pure, fibrous free condition.