

SEPTEMBER, 1940

RESEARCH BULLETIN 320

UNIVERSITY OF MISSOURI

COLLEGE OF AGRICULTURE

AGRICULTURAL EXPERIMENT STATION

M. F. MILLER, *Director*

A Microscopic and Statistical Analysis
of Texture and Structure of Ice
Cream as Affected by Compo-
sition, Physical Properties,
and Processing Methods

W. S. ARBUCKLE.

(Publication Authorized September 18, 1940)



COLUMBIA, MISSOURI

TABLE OF CONTENTS

	Page
Introduction	3
Review of Literature	3
Crystallization	3
Microscopic Study of the Texture of Ice Cream	4
Factors Affecting Texture of Ice Cream	5
Lactose Crystals Found in Ice Cream	11
Object	12
Procedure	13
Chemical Analyses	13
Preparation of Ice Cream for Microscopical Examination	13
Method of Determining Texture	14
Statistical Treatment of Texture and Structure Measurements	15
Identification of the Ice Crystal	15
Optical Identification of the Sandy Crystals Found in Ice Cream ...	16
Experimental Data	16
Structure Examination	18
Identification of the Ice Crystal	21
Accuracy of Texture and Structure Examination	23
The Effect of Fat Content Upon Texture and Structure	23
The Effect of Serum Solids Content Upon Texture and Structure ..	25
The Combined Effect of Fat and Serum Solids Upon Texture and	
Structure	27
The Effect of Sugar Content Upon Texture and Structure	27
The Effect of Sugar and Gelatin Content Upon Texture and Struc-	
ture	30
The Effect of Acidity Upon Texture and Structure	30
The Effect of Variable Sources of Serum Solids Upon Texture and	
Structure	31
The Effect of Overrun Upon Texture and Structure	33
The Effect of Combined Disaccharide and Monosaccharide Sugars	
Upon Texture and Structure	33
The Effect of Added Increments of Dextrose Accompanied by In-	
creased Serum Solids Upon Texture and Structure	35
Statistical Relationship of Various Mix Components to Texture and	
Structure	39
Lactose Crystals in Ice Cream	40
Discussion	43
Conclusions	45
Bibliography	46

ACKNOWLEDGMENT

The writer desires to express his obligation to Professor W. H. E. Reid, Professor of Dairy Husbandry, University of Missouri, and Professor A. C. Ragsdale, chairman, Department of Dairy Husbandry, University of Missouri for their counsel during the course of these investigations and for their helpful criticisms and suggestions in the preparation of this manuscript.

A Microscopic and Statistical Analysis of Texture and Structure of Ice Cream as Affected by Compo- sition, Physical Properties, and Processing Methods

W. S. ARBUCKLE.

The texture of ice cream is known to be affected by many factors but depends principally upon the presence of unfrozen materials, ice crystals and air cells of varying sizes, arrangements, and distribution. Early investigators used the general effect of coarseness or fineness as a measurement of texture, judged by the combined senses of taste, touch, and sight to distinguish between large ice crystals in comparison to the more dispersed small crystals. As texture investigational technic developed, these organoleptic examinations were supplemented with chemical and microscopical tests for the more technical studies.

During the past few years the Department of Dairy Husbandry, with the cooperation of the Department of Geology, of the University of Missouri, has developed a microscopic technic which gives more exact information on the crystalline structure of ice cream than any method previously described in the literature. The same technic was used in this investigation to study several of the factors which affect smoothness and closeness of ice cream with special reference to the influence of the ingredients of the mix upon the size of the air cells, the unfrozen materials, the size of ice crystals, and the distribution of the air cells and ice crystals.

REVIEW OF LITERATURE

Crystallization

This investigation is primarily concerned with the physical conditions present in the ice cream mix which affect crystallization and crystal growth. Turnbow (1928a) states that in the past, three theories of crystallization have been advanced: namely, the physical and chemical, the biological, and the mechanical.

Supporters of the physical and chemical theory claim that crystals originate spontaneously, providing the solution from which crystallization takes place is sufficiently concentrated with respect to the substance being crystallized. They maintain that nuclei aid the formation of crystals but that nuclei are not necessary providing

a certain degree of super-saturation is reached. Ostwald (1897) postulating on this theory, divides the so-called metastable area into two parts. One part, the metastable field, is based on the fact that a substance cannot spontaneously crystallize, even though agitation occurs, but that crystallization can occur only as a result of seeding. The liable field or truly unstable area which is just below the metastable field encounters spontaneous crystallization sooner or later, but is aided by agitation or the presence of nuclei which reduce the time required to establish equilibrium.

Kucharenko (1925) is an advocate of the biological theory. He postulates the presence of crystallons and colloidons in the air. His work suggests that they act as nuclei for crystallization.

Young (1913) and his co-workers, as well as a few others, supporting the mechanical theory, demonstrated that supercooling can be largely prevented in the case of water, providing the proper amount of mechanical impacts are employed during the process of cooling. He claims that crystallization can be explained on the basis of mechanical energy, that amorphous solids can be produced from liquids by rapid cooling, and that the method is merely one of cooling very quickly, its effectiveness depending upon the avoidance of mechanical stimuli for crystallization by not allowing time for crystals to occur.

Microscopic Study of the Texture of Ice Cream

Brainerd (1915) was one of the first to make a successful microscopic examination of the texture of ice cream. He worked in a room at a controlled temperature of 40 degrees Fahrenheit using indirect light and a device that consisted of a piece of iron pipe, cooled by the expansion of ammonia, for freezing the specimen for examination. In this manner he was able to show a difference in crystal structure between coarse and smooth ice creams. Hall (1921) using a modification of Brainerd's method took photographs of thin flakes of ice cream broken from the surface with dissecting needles showing clusters of ice crystals. These photographs were prepared at hardening room temperatures and the magnification used was 22 to 60 times.

Dahlberg (1925) working at hardening room temperatures made free hand thin sections of ice cream, covered them with a cover glass, and by using light reflected from a mirror examined them microscopically to determine the relation of air cell size and composition to the texture of the ice cream. Cole (1928) also working at hardening room temperatures used a rotating microtone and made sections of 10 to 15 microns thickness for examination. The sec-

tion was covered with a cover glass on an ordinary slide, and measurements were made by an ocular scale standardized against a stage micrometer. Later he decided that photomicrographs would be more satisfactory for making texture measurements; however, he made few measurements by this method.

Cole (1932) using the microscopic technic, developed by himself, studied the effect of freezing and hardening on the physical make-up of the finished product. Light filters were used to acquire contrasts and distinction in the microscopic prepared sections of ice cream. Dahle and Bradley (1932) used the same technique in determining the effect of type of freezer, hardening time, whipping time, and butterfat content upon ice crystal size.

Reid and Hales (1934) developed a method for using oil for imbedding the thin sections of ice cream examined by which means the texture of ice cream could be studied in more detail. Photographs were presented showing the structure of ice cream in more detail than reported by other investigators. Reid, Drew, and Arbuckle (1939) used the same technic in making microphotographs of the structure of ice creams varying in percentage of fat, serum solids, sugar, and gelatin.

Cole and Boulware (1940) used the microscopic examination of the ice crystal size in evaluating the effect of percentage butterfat and serum solids upon texture.

Reid, Decker, and Arbuckle (1940) used the technic developed by Reid and Hales (1934) in presenting microphotographs of the effect of acidity, serum solids, overrun, and different sources of serum solids upon the texture of ice cream. Reid, Cooley, and Arbuckle (1940) used the same technic in showing the effect of various sugars upon the structure of ice cream.

Factors Affecting Texture of Ice Cream

Alexander (1909) explained the action of gelatin in making ice cream smooth as due to the prevention of ice crystallization by the protective colloidal nature of gelatin.

Washburn (1910) in his classical bulletin on ice cream defines texture as referring to its internal structure and states that smooth, coarse, grainy, and sandy ice creams are all matters of texture, and that processing methods have considerable effect on smoothness.

Brown (1913) concluded that butterfat and overrun were the outstanding factors affecting texture and stated that rehardening ice cream injures texture.

Brainerd (1915) supplemented organoleptic examinations of ice cream texture with microscopic examinations and concluded that

smoothness depends upon the amount and fineness of division of solids present, other than those in true solution. More specifically smoothness depends on the size and distribution of ice crystals, which are influenced by the arrangement of solid particles that interfere with crystallization and thus reduce the size of the crystals. He states that up to the point at which the solids merge into a true solution the finer the particles the better the keeping qualities. The size of the ice crystals was found to be reduced by a high percentage of milk solids, gelatin, and homogenization.

Mortensen (1915, 1918) found that viscosity was of major importance in determining texture since the size of the air cells was governed by it. The smaller air cells were stronger than the larger ones and thus resulted in a smoother ice cream. Photographs showed that the air cells were smaller in homogenized than in un-homogenized ice cream. Stabilizers were found to affect smoothness due to their effect on viscosity.

Baer (1916) found that aging, homogenization, and fillers improved the texture of ice cream and that a raw mix produced a better body than a pasteurized mix. Increased overrun produced an open texture; too rapid freezing produced a soggy ice cream and too slow freezing produced a coarse ice cream.

Davis (1916) showed that sugar is essential to smoothness in ice cream.

Hall (1921) stated that water freezes out of the ice cream mix in the form of pure ice but did not offer proof of the statement. He pointed out that the size of the ice crystals was responsible for texture in ice cream and that rapidly formed crystals were smaller than slowly formed crystals.

Zoller (1921) found that gelatin produced supercooling due to its heat convection effect resulting in a reduced quantity of large ice crystals. Many workers since have been unable to notice such a supercooling effect.

Ambrose and Tracy (1924) using organoleptic tests found that the percentage of serum solids in ice cream had more to do with texture and resistance than did the percentage fat.

Dahlberg (1924) in examining ice cream found that normally the ice crystals size ranged between one and 50 microns; however, one crystal was found that measured 600 microns.

Zoller (1924) stated that proteins and fats in the ice cream mix readily give up their water in the form of ice; the sugars tend to hold a portion of their water with great tenacity because of their molecular structure.

Mortensen (1924, 1925) stated that fillers used in ice cream do not form a definite structure, that the benefits derived are from the increased viscosity which causes smaller air cells to be formed thereby strengthening the walls of air cells. He believed that the principal factor governing texture is the proper incorporation of air.

Dahlberg (1925a) made a microscopic examination of ice creams to determine the relation of air cell size and composition to the texture of ice cream, and concluded that the size of the air cell could not be definitely related to texture. The average air cell size of the ice cream examined was 60 microns. He also concluded that sugar improved the texture by reducing the amount of ice frozen and that milk fat and solids-not-fat had a greater combined effect on texture than either of these components alone. Dahlberg (1925) concluded that fat prevented the growth of large ice crystals by mechanically obstructing their growth. Serum solids did not show this characteristic. Gelatin retarded crystal growth due to the formation of a gel, and casein was as effective as gelatin in preventing crystal formation.

Lindewirth (1925) stated that structure of ice cream was influenced by air cell size, and that incorporation of air cells in ice cream is a fundamental factor in controlling structure.

Alexander (1926) explained the action of gelatin in making ice cream smooth by the prevention of ice crystallization due to the protective colloidal nature.

Dahle and Caulfield (1926) found that four hours aging at 40 degrees Fahrenheit was sufficient to produce ice cream with a satisfactory body and texture but a 24-hour aging period was more satisfactory.

Hunziker (1935) measured the size of sandy crystals in relation to texture of condensed milk, and several workers have used his measurements in judging the texture of ice cream. He found that crystals should not be more than 10 microns in length in order to present a smooth, velvety texture. When the majority of the crystals were 10 to 12 microns long the texture was slightly pasty, between 15 and 30 microns it was slightly sandy, and beyond 30 microns the texture was objectionable.

Martin (1926, 1928) found that the aging of low solids mixes materially improved the texture, while the aging of high solids mixes had practically no effect on body and texture.

Cole (1928) made a microscopic examination of ice cream and concluded that the greatest opportunity for securing the desired texture was offered by the freezing process. Sherfy and Small-

wood (1928) presented a bibliographic contribution concerning the effect of many factors upon body and texture.

Turnbow (1928) made several measurements of ice crystals and air cells in ice cream produced by different freezing times and composition, and found the size of the crystals to vary from a minimum of 3.5 x 3.5 microns to 182 x 274 microns. Texture improved with increased total solids and shorter freezing time.

Turnbow (1928) reviewed the different theories of crystallization and discussed the application of the physical and chemical, biological, and mechanical theories to crystallization in ice cream.

Cole (1929) compared the size of ice crystals in ice cream frozen rapidly with that frozen slowly, and reported an average size of 7 x 9 microns for the rapidly frozen ice cream as compared to an average size of 106 x 152 microns for slowly frozen ice cream. He also stated that overrun did not affect crystal size.

Muller and Button (1929) made microscopic examinations of air cells and showed that as the size of the air cell decreases the film surrounding the air cell increases.

Munkwitz and Meade (1929) studying the effect of sharp and dull freezer blades in relation to texture, prepared photographs of coarse-textured ice cream which they described as having crystals standing out giving it a loose background and a rough topography. Close textured ice cream was described as having a smooth, compact background with a smooth surface.

Dahle and Bradley (1932) using the same technic as Cole (1928) studied the effect of whipping time on texture and size of ice crystals, and found that as freezing and whipping time increased from 5 to 15 minutes, at the same temperature, the average size of ice crystals increased from .072 to .116 mm. at 24.5 degrees Fahrenheit. The superior texture in the shorter whipping and freezing time was due, they explained, to the fact that under normal operating conditions, the ice cream frozen and whipped in a shorter time contains more water frozen to ice and consequently less ice needs to be formed in the hardening room, the latter being a slower process. Similar drawing temperatures did not give similar textures. The ice crystals were smaller and the texture score was higher when the ice cream was hardened faster. Ice cream from the continuous freezer appeared smoother than ice cream from the batch freezer with the same size ice crystal. When studying mixes with 10 to 18 per cent fat they concluded that fat had little influence on texture of ice cream frozen in a continuous freezer.

Reid (1933) found that the crystalline structure of ice cream was altered with each two per cent variation in fat, serum solids, and sugar, and each one-tenth per cent of gelatin in the composition of the mix.

Keller (1934) examined thin sections of ice cream with the petrographic microscope and found three distinctly different materials present, namely: small anhedral grains having low indices of refraction to be ice crystals (they are uniaxial, positive, with low birefringence), an amorphous bounding material which surrounded the ice crystals, and larger irregular shaped air bubbles which were scattered abundantly through the ice cream.

Reid and Hales (1934) using an improved method of examination by embedding the thin sections of ice cream in an immersion oil reported that a fine texture is associated with the presence of a uniformly dispersed system of ice crystals and air cell boundaries. Improvement in texture was directly proportional to increased increments of fat, solids-not-fat, and sugar.

Dahle (1926) identified texture as a condition due to the size of the ice crystals and stated that on the basis of ice crystal measurements butterfat was the most important of the ingredients in the mix that improved texture. Serum solids had a like effect but because the milk sugar of the serum solids goes into true solution the effect was not as extensive as that caused by the fat. Sugar improved the texture in a different manner due to the fact that less ice was frozen. Homogenization also improved texture.

Sommer (1938) defines texture as the attribute of a substance relating to its finer structure—the size, shape, and arrangement of the small particles, and body as the attribute of a substance relating to the properties of the mass as a whole—its consistency or firmness and in the case of ice cream its melting resistance.

From a review of the literature he made the following statements: an analogous condition must be expected with regard to the size of ice crystals and other solid particles to the texture of ice cream, as Hunziker had previously found the size of lactose crystals had to the texture of condensed milk. Namely, the size of the crystals must be below 12 microns if the texture is to be smooth, and if the crystals are above 30 microns a coarse texture is encountered. He expressed the opinion that the most common texture defect is due to the presence of large ice crystals and that the lamellae of the air cells are thinnest when the cells are smallest; this is shown by calculations. He also states that when the air is spread out in fine films, it is logical to expect the size of the

ice crystal to be limited and the entire structure to be finer thereby producing a smooth texture.

On experimental indications as well as logic Sommer states that small air cells are conducive to a smooth texture. If the total solids content of the mix is varied by increasing the percentage content of any one of the mix constituents, a smoother texture may be expected because less water will be frozen; mechanical obstruction of crystal growth will result, and the mix will have a lower freezing point with proper resistance to whipping.

In addition to the effect of fat globules in limiting ice crystal growth by mechanical obstruction, the fat produces a characteristic sensation of smoothness in the mouth. It is safe to say that an increase would have to be one per cent or more in fat content before a recognizable effect on texture could be observed. Serum solids improved the texture through mechanical obstruction, replacing water, and lowering the freezing point of the mix.

We would expect a high acidity or a high calcium and magnesium content to be harmful to the texture, and a high citrate and phosphate content to be helpful.

Increasing the sugar content improves the texture especially through its effect on the freezing point, causing less complete freezing of the product. The smoothing effect of sugar is more apparent than real because if the ice creams were hardened to the same hardness, all apparent improvement in texture would vanish.

The use of larger amounts of glucose to secure the same sweetening effects as sucrose produces a smoother texture because of a lower water content of the mix.

Gelatin probably owes its smoothing effects to its mechanical obstruction to crystal growth, and to its ability to imbibe large quantities of water from the mix.

Egg yolk results in a smoother texture due to high whipping ability, and facilitates the incorporation of air in small cells.

Homogenization decidedly smooths the texture due to the finer dispersion of the fat globules which offer mechanical obstruction to ice crystal growth, and the whipping properties are improved, which results in a smaller air cell and smoother texture.

In heat shocking ice cream the rise in temperature causes the small ice crystals to melt; as the temperature is lowered, and when the water is again crystallized out, it deposits onto the larger crystals that were left.

Freezer design is an obvious and important factor in determining the texture, but there are no comparative studies available.

Freezing to a stiffer consistency produces a smoother ice cream because the freezing is much more rapid than that encountered in the hardening room, thus producing a smaller ice crystal; the longer whipping gives finer subdivision of the air.

Sommer concluded that the experimental evidence concerning some of the above deductions were not very positive, and that some of the discussion was theoretical and speculative, but there are indications that they are correct.

Reid, Drew, and Arbuckle (1939) presented photomicrographs from which they concluded that added increments of fat, serum solids, and gelatin produced a smoother textured ice cream with a smaller ice crystal, and that variable increments of sucrose had little effect upon ice crystal size.

West (1939) suggested the use of 19 to 35 per cent hydrofluoric acid as an immersion medium in determining the principal indices of the ice crystal in ice cream. The principal indices of pure ice are 1.309 and 1.313.

Cole and Boulware (1940) combined organoleptic observation with microscopic examination and dilatometer measurements in evaluating the factors which contribute to the texture of ice cream frozen on experimental hand freezers, and concluded that fat and serum solids content improved the texture and reduced the size of ice crystal. The solids-not-fat was more effective in reducing crystal size than fat, which was due to a retarding action, as shown by the dilatometer. Increased fat in ice cream was found to have a greater effect upon texture judged organoleptically than it did in reducing ice crystals size, the reduced crystal size being the result of the growth of the crystal. A good correlation may be expected between ice crystal size and smoothness of texture where variations in composition are not too great.

Lactose Crystals Found in Ice Cream

Since Bothell (1920) first identified sandiness as being due to the presence of lactose crystals, several different forms of these crystals have been isolated from ice cream.

Zoller and Williams (1921) isolated sandy material and found crystals, some of which they describe as being tomahawk shaped and others that were columnar. Photographs also showed that the crystals they examined contained some tomahawk crystals with ragged sawtooth ends. They identified the bulk of this material as alpha lactose.

Hunziker and Nissen (1927) described two different forms of alpha lactose. They stated that one form was fully developed,

having trapezoidal side faces and rhombic tops and bottoms. The presence of sugar in the solution apparently caused a modification in the appearance of this crystal. The modification resembled short stubbed truncated pyramids with flat rhombic base and apex that lacked full development; the beveled faces at the base and apex were entirely absent. They stated that the habit of the crystal was probably the only element of difference between the two.

Williams and Peters (1930) found a new diamond shape crystal in ice cream which they identified as alpha lactose.

Herrington (1934) found that the crystallization habit of alpha hydrate lactose varied greatly under different conditions of crystallization.

Arbuckle, Decker, and Reid (1939) used the petrographic microscope in studying sandy material and described the tomahawk shape alpha lactose and a columnar shape crystal as beta lactose.

Decker, Arbuckle, and Reid (1939) present optical data and photographs of alpha and beta lactose crystals.

OBJECT

The object of this investigation was to make a detailed analysis of the relative effect of several components of the ice cream mix upon the size and arrangement of ice crystals and the air cells in relation to the texture of the finished ice cream; thus presenting technical information concerning some of the less familiar aspects of texture.

PROCEDURE

The ice creams examined in this investigation were those used over a three year period in a number of projects carried on by the Dairy Husbandry Department in a research program concerned with the effect of various factors in the composition, physical, and chemical properties of the mix upon the finished product. All ice creams were frozen under carefully controlled conditions on modern commercial freezers, having accurate temperature, pressure, and speed control gauges.

The standard method of processing the mix was as follows: (1) pasteurization at 150 degrees Fahrenheit (65.5 degrees Centigrade) for 30 minutes; (2) homogenization at pasteurization temperature and 2500 pounds pressure; (3) cooling immediately to 40 degrees Fahrenheit (4.4 degrees Centigrade); (4) aging for one hour at 40 degrees Fahrenheit (4.4 degrees Centigrade); (5) freezing in a freezer with the adjustments—ammonia temperature -8 degrees Fahrenheit (-22.2 degrees Centigrade), mix pump vacuum 10 inches, back pressure 55, freezer speed 8, drawing temperature of ice cream 21 degrees Fahrenheit (-6.11 degrees Centigrade), overrun 90 per cent; and (6) hardening at -10 degrees Fahrenheit (-23.3 degrees Centigrade).

Chemical Analyses

Chemical analyses were made of the mixes immediately after the aging period. The surface tension was determined by the DeNouy surface tension apparatus, at a temperature of 20 degrees Centigrade. Viscosity values were determined at 20 degrees Centigrade by the Ostwald tube, and expressed in comparison with water.

The acidities were determined by titration with sodium hydroxide, and the pH values were determined by the use of a glass electrode potentiometer.

Preparation of Ice Cream for Microscopical Examination

All ice creams were hardened in the hardening room at -10 to -15 degrees Fahrenheit (-23.3 to -26.1 degrees Centigrade) for 48 hours after freezing, before texture studies were made. The microscopic technic used was similar to that used by Reid and Hales who prepared all sections in the hardening room at -15 degrees Fahrenheit (-26.1 degrees Centigrade). All apparatus, including the ordinary microscope and lamp, immersion oils (ranging in refractive indices from 1.38 to 1.60), slides, cover glasses, razor-knives, and microscope camera, were taken into the hardening room

for use. Free hand sections were made of the ice cream in the form of ribbons, approximately 10 microns thick. These sections were then embedded in an immersion oil, with a refractive index of 1.42 on a microscope slide. They were then covered with a cover glass, and finally examined with a microscope and photographed, using a magnification of approximately 100 times. Figure 1 shows the equipment used in the preparation, examination, and photographing of the sections.

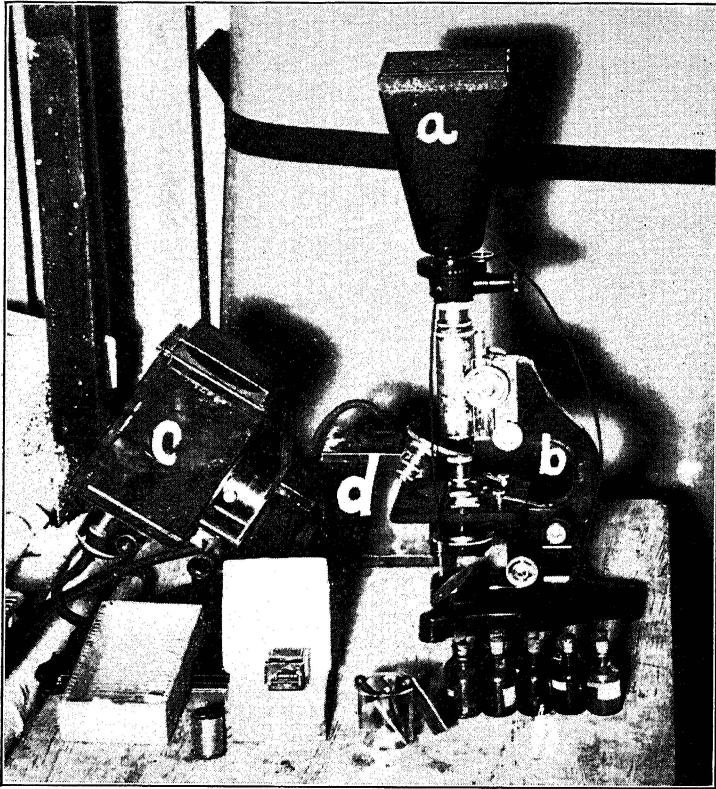


Fig. 1.—Apparatus used in securing, examining, and photographing sections of ice cream: a, microscope camera; b, microscope; c, illumination lamp; d, transformer; e, slide container; f, slides and cover glasses; g, razor-knife; h, immersion oils.

Method of Determining Texture

Due to the uncomfortable temperature and the presence of undesirable health conditions of the hardening room, photographs were taken of at least two fields from each section; texture measurements were made from these photographs as suggested by Cole

1928). The camera was standardized with a stage micrometer; thus the photographs were of known magnification. This procedure proved very satisfactory, and enabled the taking of more texture measurements than have heretofore been reported. The measurements included the size of the ice crystals, the size of the air cell (although in some cases the air cell was destroyed due to the thinness of the section and the sectioning methods), the distance between air cells, and the amount of unfrozen material between crystals. From these measurements the effect of any factor upon texture could be accurately determined. The average of at last twenty-five measurements was taken of air cell size and distance between air cells, while the average of at least one hundred measurements was taken for the other measurements used. Microscopic examinations were supplemented with organoleptic observations on all ice creams studied. It required about one hour's preparation at hardening room temperatures in order to section and photograph each sample of ice cream. Approximately one hundred and fifty photographs were taken for this investigation.

Statistical Treatment of Texture and Structure Measurements

The correlation of different components of the mix to ice crystal size, air cell size, distance between cells, and distance between crystals was determined by statistical treatment. From these correlations the linear regression equation for each of the factors studied was calculated. Pearson's product moment correlation coefficient method for calculation of simple correlation of ungrouped data was employed, and the formulas that were applied to secure the various constants in the derivation of the correlations and for the calculation of the regression equations were the same as those outlined by Holzinger (1928).

Identification of the Ice Crystal

The petrographic microscope was used to check the optical properties of the ice crystals as observed by Keller (1934), and the ordinary microscope was used in searching for an immersion medium with a low enough refractive index to determine the principal indices of the ice crystals.

Grosse (1937) stated that the refractive index of hydrofluoric acid is theoretically the lowest of any compound. West (1939) suggested that if the work was done rapidly and if care was taken that the lenses of the microscope were protected hydrofluoric acid could be used as an immersion medium. Thus hydrofluoric acid

was diluted to secure the desired refractive index and used as an immersion medium to approximate the principal indices of the ice crystals.

Optical Identification of the Sandy Crystals Found in Ice Cream

A series of lactose solutions were prepared to meet the conditions under which lactose crystallizes in ice cream, and the optical properties of the types of crystals that have been found in ice cream were determined by the use of the petrographic microscope thereby presenting in a summarized form the optical properties of the different forms of sandy crystals reported in the literature as found in ice cream.

EXPERIMENTAL DATA

A number of ice creams of different composition combinations were prepared to determine the effect of several ingredients, and also the effect of physical and chemical properties upon texture and structure. Tables 1 and 2 present the composition and physical and chemical properties of the ice creams examined in this investigation.

TABLE 1—(Section 1).—THE COMPOSITION OF ICE CREAM EXAMINED

Mix No.	Fat Per Cent	Serum Solids Per Cent	Sugar Per Cent	Gelatin Per Cent	Total Solids Per Cent	Overrun Per Cent
			<i>Variable Fat</i>			
1	10.00	11.00	14.00	0.30	35.30	90.00
2	12.00	11.00	14.00	0.30	37.50	90.00
3	14.00	11.00	14.00	0.30	39.30	90.00
4	16.00	11.00	14.00	0.30	41.30	90.00
			<i>Variable Serum Solids</i>			
5	12.00	9.00	14.00	0.30	35.30	90.00
6	12.00	11.00	14.00	0.30	37.50	90.00
7	12.00	13.00	14.00	0.30	39.30	90.00
8	12.00	15.00	14.00	0.30	41.30	90.00
			<i>Variable Sugar</i>			
9	12.00	11.00	12.00	0.30	35.30	90.00
10	12.00	11.00	14.00	0.30	37.50	90.00
11	12.00	11.00	16.00	0.30	39.30	90.00
12	12.00	11.00	18.00	0.30	41.30	90.00
			<i>Variable Sugar and Medium Serum Solids</i>			
13	12.00	13.50	12.00	0.30	37.80	90.00
14	12.00	13.50	14.00	0.30	39.80	90.00
15	12.00	13.50	16.00	0.30	41.80	90.00
16	12.00	13.50	18.00	0.30	43.80	90.00
			<i>Variable Sugar and Gelatin and High Serum Solids</i>			
17	12.00	15.50	12.00	0.20	39.70	90.00
18	12.00	15.50	14.00	0.30	41.80	90.00
19	12.00	15.50	16.00	0.40	43.90	90.00
20	12.00	15.50	18.00	0.80	46.00	90.00

TABLE 1—(Section 2).—THE COMPOSITION OF ICE CREAM EXAMINED

Mix No.	Fat Per Cent	Serum Solids Per Cent	Sugar Per Cent	Gelatin Per Cent	Acidity Per Cent	Total Solids Per Cent	Overrun Per Cent
<i>Variable Acidity and Medium High Serum Solids</i>							
21	12.00	13.50	14.00	0.30	0.24	39.80	90.00
22	12.00	13.50	14.00	0.30	0.18	39.80	90.00
23	12.00	13.50	14.00	0.30	0.12	39.80	90.00
24	12.00	13.50	14.00	0.30	0.08	39.80	90.00
<i>Variable Acidity and High Serum Solids</i>							
25	12.00	15.00	14.00	0.30	0.24	41.30	90.00
26	12.00	15.00	14.00	0.30	0.18	41.30	90.00
27	12.00	15.00	14.00	0.30	0.12	41.30	90.00
28	12.00	15.00	14.00	0.30	0.08	41.30	90.00
<i>Variable Overrun and High Serum Solids</i>							
29	12.00	15.00	14.00	0.20	0.11	41.20	85.00
30	12.00	15.00	14.00	0.20	0.11	41.20	100.00
31	12.00	15.00	14.00	0.20	0.11	41.20	115.00
32	12.00	15.00	14.00	0.20	0.11	41.20	130.00
<i>Variable Sources of Serum Solids*</i>							
33	12.00	15.00 ¹	14.00	0.30	...	41.20	90.00
34	12.00	15.00 ²	14.00	0.30	...	41.20	90.00
35	12.00	15.00 ³	14.00	0.30	...	41.20	90.00
36	12.00	15.00 ⁴	14.00	0.30	...	41.20	90.00
37	12.00	15.00 ⁵	14.00	0.30	...	41.20	90.00
*Sources of serum solids.							
		1	2	3	4	5	
Milk and cream		6.00	6.00	6.00	6.00	6.00	
Dry milk powder		3.00	6.00	9.00	7.00	4.50	
Mineralized milk powder .		6.00	3.00	0.00	0.00	4.50	
<i>Variable Increments of Dextrose</i>							
38	12.00	11.00	14.00 ¹	0.30	...	37.30	90.00
39	12.00	11.00	12.00 ²	0.30	...	35.30	90.00
40	12.00	11.00	14.00 ³	0.30	...	37.30	90.00
41	12.00	11.00	16.00 ⁴	0.30	...	39.30	90.00
42	12.00	11.00	18.00 ⁵	0.30	...	41.30	90.00

TABLE 1—(Section 3).—THE COMPOSITION OF ICE CREAM EXAMINED

Mix No.	Fat Per Cent	Serum Solids Per Cent	Sugar Per Cent	Gelatin Per Cent	Total Solids Per Cent	Overrun Per Cent
Sources of Sugar:						
			1. 14.00 per cent sucrose			
			2. 10.00 per cent sucrose; 2.00 per cent dextrose			
			3. 10.00 per cent sucrose; 4.00 per cent dextrose			
			4. 10.00 per cent sucrose; 6.00 per cent dextrose			
			5. 10.00 per cent sucrose; 8.00 per cent dextrose			
<i>Variable Increments of Cerelese</i>						
43	12.00	11.00	14.00 ¹	0.30	37.30	90.00
44	12.00	11.00	12.00 ²	0.30	35.30	90.00
45	12.00	11.00	14.00 ³	0.30	37.30	90.00
46	12.00	11.00	16.00 ⁴	0.30	39.30	90.00
47	12.00	11.00	18.00 ⁵	0.30	41.30	90.00
Sources of Sugar:						
			1. 14.00 per cent sucrose			
			2. 10.00 per cent sucrose; 2.00 per cent cerelese			
			3. 10.00 per cent sucrose; 4.00 per cent cerelese			
			4. 10.00 per cent sucrose; 6.00 per cent cerelese			
			5. 10.00 per cent sucrose; 8.00 per cent cerelese			
<i>Variable Increments of Dextrose and Medium Serum Solids and Low Fat</i>						
48	10.00	13.00	14.00 ¹	0.30	37.30	90.00
49	10.00	13.00	12.00 ²	0.30	35.30	90.00
50	10.00	13.00	14.00 ³	0.30	37.30	90.00
51	10.00	13.00	16.00 ⁴	0.30	39.30	90.00
52	10.00	13.00	18.00 ⁵	0.30	41.30	90.00
Sources of Sugar:						
			1. 14.00 per cent sucrose			
			2. 10.00 per cent sucrose; 2.00 per cent dextrose			
			3. 10.00 per cent sucrose; 4.00 per cent dextrose			
			4. 10.00 per cent sucrose; 6.00 per cent dextrose			
			5. 10.00 per cent sucrose; 8.00 per cent dextrose			

TABLE 2.—THE PHYSICAL AND CHEMICAL PROPERTIES OF ICE CREAMS EXAMINED

Mix No.	Acidity Per Cent	pH	Surface Tension	Coefficient of Viscosity in Poise
1	.22	6.64	48.00	.1260
2	.22	6.65	48.00	.1366
3	.22	6.65	48.00	.1536
4	.22	6.64	48.00	.1690
5	.23	6.35	47.20	.1571
6	.21	6.50	47.20	.1780
7	.21	6.60	46.80	.1980
8	.21	6.70	47.20	.2103
9	.21	6.20	47.90	.1690
10	.21	6.25	48.60	.1740
11	.21	6.25	47.90	.1960
12	.20	6.20	48.60	.2060
13	.21	6.50	45.40	.1220
14	.20	6.55	46.10	.1588
15	.19	6.50	46.10	.1966
16	.18	6.50	46.10	.2458
17	.20	6.70	47.50	.2460
18	.19	6.70	46.10	.2513
19	.18	6.70	46.10	.5998
20	.17	6.70	46.10	.9889
21	.24	6.80	48.06	.0962
22	.18	6.90	48.06	.1104
23	.12	7.50	47.69	.1318
24	.08	7.65	48.06	.1740
25	.24	6.60	48.06	.0688
26	.18	6.90	48.42	.0744
27	.12	7.65	48.42	.0936
28	.08	8.50	48.06	.1336
29	.11	7.70	48.79	.1122
30	.11	7.70	48.79	.1122
31	.11	7.70	48.79	.1122
32	.11	7.70	48.79	.1122
33	.19	6.75	49.89	.2551
34	.21	6.70	49.89	.1993
35	.31	6.45	49.89	.2835
36	.28	6.50	49.89	.0850
37	.23	6.83	49.89	.2022
38	.23	6.50	48.40
39	.23	6.50	47.50
40	.23	6.50	48.80
41	.22	6.50	47.90
42	.22	6.50	47.90
43	.23	6.50	53.60
44	.22	6.50	49.90
45	.23	6.50	50.60
46	.22	6.50	52.10
47	.22	6.50	54.30
48	.26	6.30	50.60
49	.25	6.40	51.30
50	.25	6.40	51.30
51	.24	6.40	51.30
52	.25	6.50	52.80

Structure Examination

Before any texture measurements were conducted in this investigation, a microscopic comparison was made of fine and coarse textured ice creams in order to determine the differences in their microscopic appearance.

The following photographs give the macroscopic and microscopic comparison of texture of ice cream.

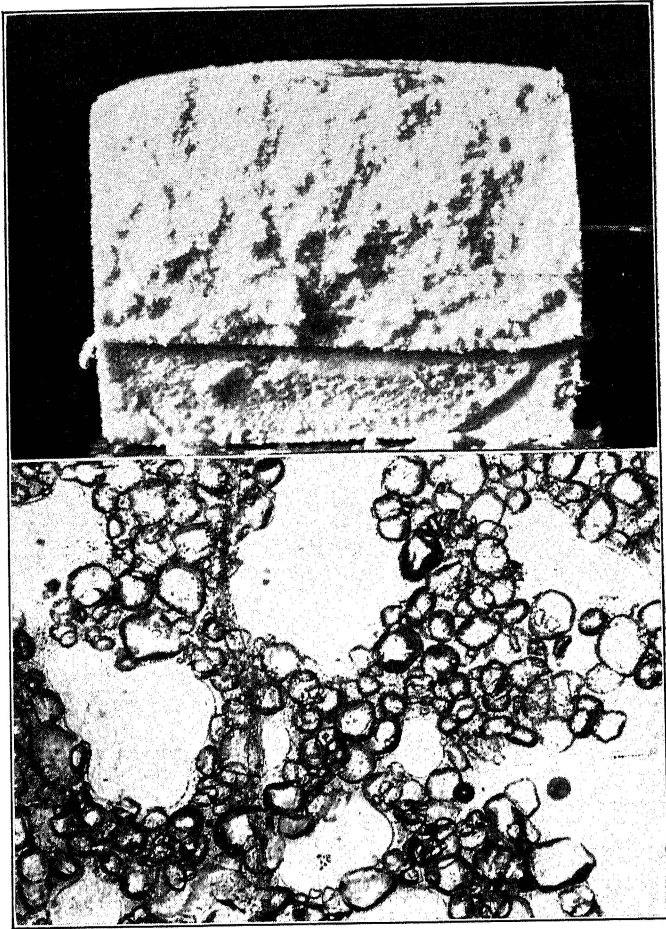


Fig. 2.—Photographs showing macroscopic and microscopic appearance of fine texture in ice cream.

It can be seen from Figures 2 and 3 that macroscopic texture differences can easily be detected by microscopic examination of ice crystal size. The ice creams shown were rated organoleptically as smooth and fine, and open and coarse in texture, while the average ice crystal size was 4.3×3.6 , and 11.4×9.2 microns, respectively. Figure 4 shows that in the process of heat shocking ice cream the small crystals melt and upon freezing much larger ice crystals are formed which results in a coarse textured ice cream.

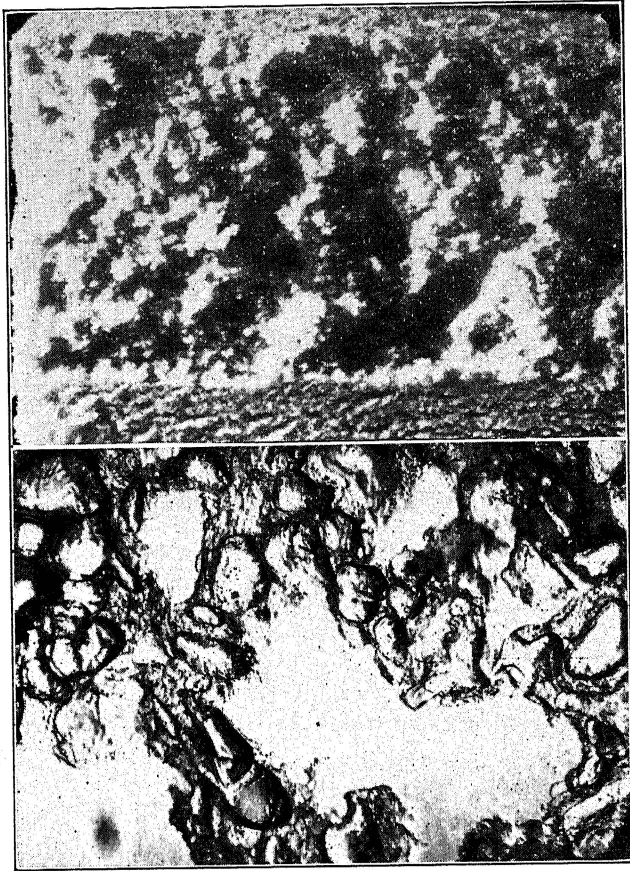


Fig. 3.—Photographs showing macroscopic and microscopic appearance of medium coarse, open texture in ice cream.

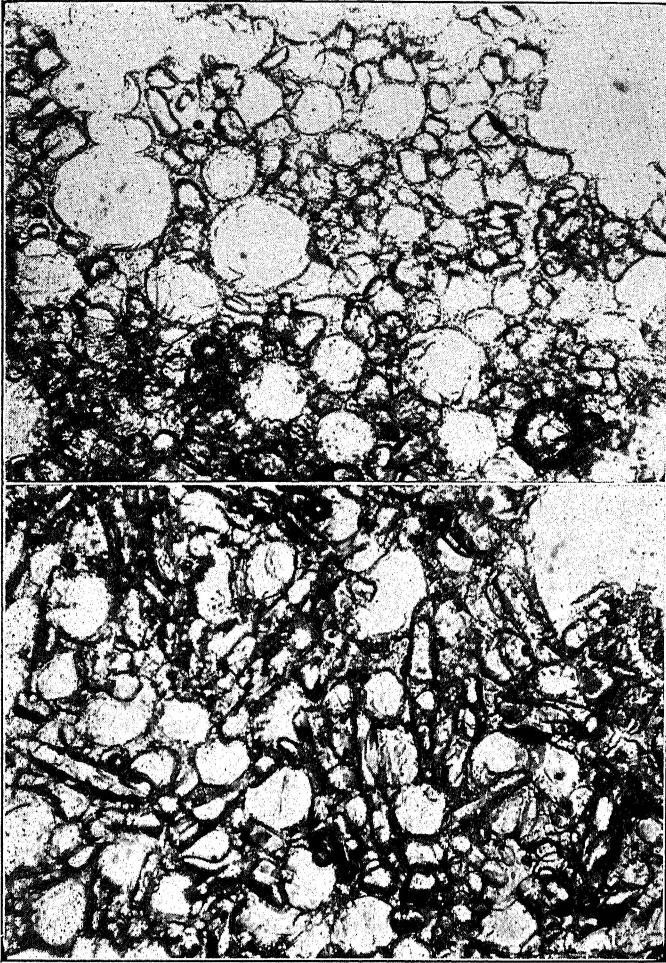


Fig. 4.—A comparison of the microscopic texture of an ice cream before and after heat shocking.

Identification of the Ice Crystal

Thin sections of ice cream examined under high power magnification revealed in detail the materials that Reid and Hales (1934) and Keller (1934) pointed out to be present in the structure of ice cream: namely, ice crystals, air cells, and unfrozen materials. The use of immersion liquids of different refractive indices, prepared with the use of a Abbe refractometer by procedures outlined by Fry (1933) and Winchell (1931), revealed that the unfrozen ma-

terial surrounding the ice crystals varied as the composition of the mix was changed. Hydrofluoric acid with a calculated refractive index of 1.310 at -26.1 degrees Centigrade (-15 degrees Fahrenheit) was used to determine the principal indices of the ice crystals. Fig. 5 shows the structure of an ice cream magnified 250 times.

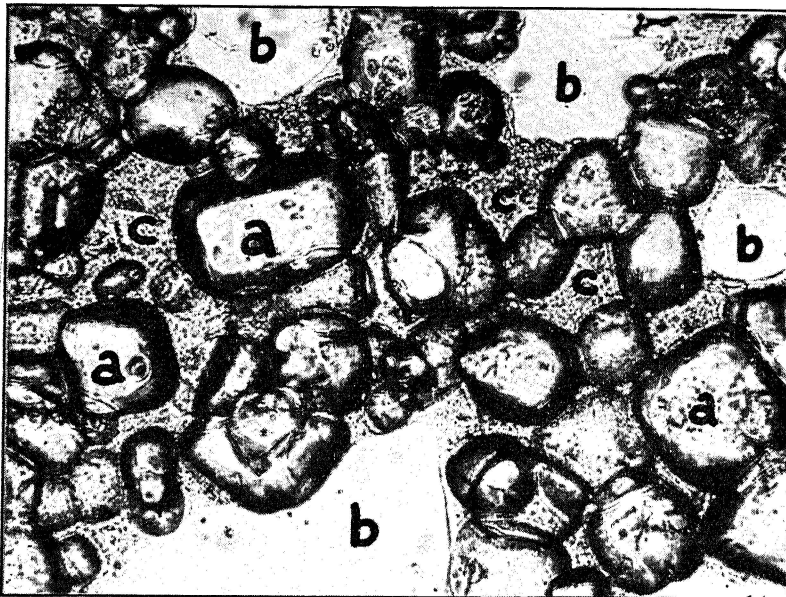


Fig. 5.—Photomicrograph showing detailed internal structure of ice cream. (X 250).

- a. Ice crystals
 - Form—anhedral—uniaxial—positive
 - Refractive indices
 - Omega 1.309
 - Epsilon 1.313
 - Optical character—positive
 - Bi—refrignce—low
- b. Air cells
 - Refractive index
 - Much lower than that of any immersion liquid used
- c. Unfrozen material
 - Refractive index variable but approximately 1.460

From the optical data presented in Figure 5 it is logical to conclude that water freezes out of the ice cream mix in the form of pure ice, and that the unfrozen material surrounding the ice crystal may be an important factor in influencing organoleptic observations of texture. Varying the composition of the mix causes the refractive index of the unfrozen material to change. This would indicate that the material may become more viscous, or less viscous, and vary in amount, thus greatly influencing organoleptic observations.

Accuracy of Texture and Structure Examination

In order to determine the accuracy and statistical significance of the method followed in securing texture and structure measurement, in this investigation, a statistical analysis was made of the measurements of a number of identical control samples of ice cream. Table 3 presents this analysis.

The standard deviation from the mean for each of the factors studied, showed that measurements for identical samples of ice cream agreed very closely, especially for the ice crystal size and distance between crystal factors, and thus indicates that statistically the method of measurement used to determine texture and structure was highly accurate.

TABLE 3.—STATISTICAL ANALYSIS OF METHOD USED TO DETERMINE TEXTURE AND STRUCTURE

Mix No.	Average Ice Crystal Size		Average Diameter (Microns)	Average Air Cell Diameter (Microns)	Distance Between Cells (Microns)	Unfrozen Material Between Crystals (Microns)
	Length	Width				
2	6.43	x 4.88	5.65	18.62	13.65	.60
6	5.84	x 4.62	5.23	16.84	14.87	.82
10	6.01	x 4.53	5.22	10.85	10.72	.75
38	5.85	x 4.99	5.42	13.30	11.92	.76
43	5.61	x 4.45	5.03	12.05	10.04	.71
2a	6.22	x 4.71	4.47	11.92	11.51	.68
6a	5.92	x 4.90	5.41	14.61	12.82	.64
10a	6.62	x 4.01	5.32	17.40	10.58	.72
38a	6.00	x 4.21	5.10	13.12	13.04	.80
43a	5.82	x 4.88	5.35	15.01	12.13	.74
Mean			5.32 ± .037	14.37 ± .74	12.12 ± .45	.72 ± .02
Standard Dev.			.17 ± .026	2.34 ± .52	1.43 ± .32	.06 ± .01

The literature reveals that there is considerable disagreement as to the effect of various mix components upon the texture of ice cream. In order to secure detailed information upon this matter, a number of ice creams were examined to determine the effect of a number of variables upon texture.

The Effect of Fat Content Upon Texture and Structure

To determine the effect of fat content upon the structure and texture, a series of ice creams was prepared under strictly controlled conditions in which the fat content was the only variable. The fat ranged from 10 to 16 per cent which is the normal range used in the industry. The measurements secured from ice creams containing variable increments of fat are presented in Table 4.

There is a high negative correlation between the fat content and ice crystal size, and a high positive correlation between the fat content and the distance between crystals. The average diameter of the ice crystal had a negative change of approximately .46

TABLE 4.—EFFECT OF FAT CONTENT OF ICE CREAM UPON TEXTURE AND STRUCTURE

Mix No.	Fat Per Cent	Ice Crystals Size Microns		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
		Average Dimension	Average Diameter		Cells Microns	Crystals Microns	
1	10.00	8.26 x 6.08	7.17	21.00	11.85	.49	Smooth Close
2	12.00	6.43 x 4.88	5.65	18.62	13.65	.60	Smooth Close
3	14.00	5.69 x 4.66	5.17	24.00	16.34	.75	Smooth, Close Sl. Crumbly
4	16.00	4.72 x 3.80	4.26	14.04	8.05	.68	Smooth Sl. Soggy Sl. Crumbly
Correlation of fat to texture and structure measurements.....			-.97±.02	-.56±.23	-.31±.30	+.94±.04	
Change in texture and structure measurements for each one per cent increase in fat content			-.4564	+.0361	

microns for each one per cent increase in fat content. Dahlberg (1925) and Sommer (1938) pointed out increased fat content produced a smaller ice crystal due to mechanical obstruction. This obstruction to crystal growth causes the water to freeze out of the mix in smaller ice crystals, instead of allowing it to collect on crystal nuclei during the agitation in the freezing process to form larger crystals.

Apparently there is very little relationship of air cell size and distribution of air cells to the per cent of fat in ice cream. It should be stated that the air cell measurements presented in this investigation are of those cells of microscopic size, and possibly a higher proportion of larger cells, not in this range, were not measured. Also a smooth effect produced by the fat was noted by organoleptic observations but could not be measured microscopically.

The Effect of Serum Solids Content Upon Texture and Structure

A series of ice creams, in which the serum solids content was the only variable, was prepared to determine the effect of serum solids upon the structure and texture. The serum solids content ranged from 9 to 15 per cent. Table 5 shows the effect of variable increments of serum solids upon texture and structure measurements.

There is a high negative correlation of serum solids content of ice cream to average ice crystal diameter, the average air cell size and the distance between cells, and a high positive correlation to the distance between crystals.

The ice crystal size, the air cell size, the distance between cells, and distance between crystals changed -4.336 , -1.1524 , $-.0768$, and $.1645$ microns, respectively, for each one per cent increase in serum solids content.

Increased serum solids, like fat content, produced a smaller ice crystal, due to mechanical obstruction, and also the hydrophylic nature of proteins of milk influence the formation of water into ice crystals and provides a physical condition in the ice cream mix that is favorable for smaller air cell formation with a greater distribution. The pronounced increase in distance between crystals may be caused by the addition of serum solids which lowers the freezing point of the mix, resulting in a greater amount of unfrozen material between the crystals. This factor along with the smaller ice crystal size and slightly smaller air cell size, caused the ice creams of higher serum solids content to be ranked as more smooth by organoleptic observations.

TABLE 5.—EFFECT OF SERUM SOLIDS CONTENT OF ICE CREAM UPON TEXTURE AND STRUCTURE

Mix No.	Serum Solids Per Cent	Ice Crystal Size Microns		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
		Average Dimension	Average Diameter		Cells Microns	Crystals Microns	
5	9.00	6.37 x 4.80	5.58	17.66	16.54	.70	Sl. Crumbly Smooth
6	11.00	5.84 x 4.62	5.23	18.84	14.87	.82	Smooth
7	13.00	4.37 x 3.51	3.94	15.80	11.66	1.40	Smooth
8	15.00	3.58 x 2.77	3.22	10.32	12.40	1.60	Very Smooth
Correlation of serum solids to texture and structure measurements			-.97±.02	-.90±.06	-.90±.06	+.97±.02	
Change in texture and structure measurements for each one per cent increase in serum solids content			-.4336	-1.1524	-.0768	+.1645	

The Combined Effect of Fat and Serum Solids Upon Texture and Structure

In order to determine the combined effect of fat and serum solids content upon structure and texture an arrangement of mixes was made which totaled from 21 to 27 per cent of combined fat and serum solids. Table 6 gives the combined effect of fat and serum solids upon texture.

A high negative correlation of combined fat and serum solids content to the average ice crystal size, the average air cell size, and the distance between cells, and a high positive correlation to the distance between crystals, is shown in Table 6. As suggested by many investigators, Table 6 shows that fat and serum solids are more effective in combination than they are alone in reducing ice crystal size. The effect upon the other texture and structure factors considered was approximately the same as the average of the effect of the fat and serum solids considered separately.

Ice crystal size, air cell size, distance between cells, and distance between crystals changed $-.5820$, $-.9523$, $-.1464$, and $-.1899$, respectively, for each one per cent increase in combined fat and serum solids content.

The Effect of Sugar Content Upon Texture and Structure

The effect of sugar content in a 11.00 per cent serum solids content mix upon texture and structure is shown in Table 7.

It can be seen from Table 7 that there is a high negative correlation between sugar content and ice crystal size, and a high positive correlation between sugar content and distance between crystals. The ice crystal size and distance between crystals changed $-.2870$, and $.0657$, respectively, with each increase of one per cent sugar content of the mix. It is apparent that the sugar content does not have as much effect upon reducing the ice crystal size as does the fat or serum solids content. A low correlation existed between the sugar content and air cell size and distribution. Table 8 shows the effect of variable increments of sugar upon texture and structure in an ice cream mix containing 13.50 per cent serum solids.

Variable increments of sugar in ice cream, containing 13.50 per cent serum solids, show a like effect in reducing the ice crystal size as that produced by sugar in lower serum solids content ice creams. Also, little relationship of sugar content to air cell size and distribution exists. However, the change in distance between ice crystals for each one per cent increase of sugar content in a 11.00 and a 13.50 per cent serum solids ice cream was $.0657$ and $.15438$ microns,

TABLE 6.—THE COMBINED EFFECT OF FAT AND SERUM SOLIDS CONTENT UPON TEXTURE AND STRUCTURE

Mix No.	Fat Per Cent	Serum Solids Per Cent	Ice Crystal Size Microns		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
			Average Dimension	Average Diameter		Cells Microns	Crystals Microns	
1	10.00	11.00	8.26 x 6.08	7.17	21.00	11.85	.49	Smooth Sl. Crumbly
A	11.00	12.00	6.17 x 4.71	5.41	19.13	11.78	.73	Smooth
7	12.00	13.00	4.37 x 3.51	3.95	15.80	11.66	1.40	Smooth
B	13.00	14.00	4.01 x 3.21	3.61	15.61	11.58	1.51	Very Smooth Sl. Soggy
Correlation of Combined fat and serum solids to texture and structure measurements				-.92±.05	-.92±.05	-.99±.01	+.97±.02	
Change in texture and structure measurements for each one per cent increase of combined fat and serum solids content				-.5820	-.9523	-.1464	+.1899	

TABLE 7.—THE EFFECT OF SUGAR CONTENT UPON TEXTURE AND STRUCTURE

Mix No.	Serum Solids Per Cent	Sugar Per Cent	Ice Crystal Size Microns		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
			Average Dimension	Average Diameter		Cells Microns	Crystals Microns	
9	11.00	12.00	6.75 x 5.10	5.92	16.13	9.30	.62	Smooth Sl. Crumbly Med. Resistance
10	11.00	14.00	6.01 x 4.53	5.27	10.85	10.72	.75	Smooth Med. Resistance
11	11.00	16.00	5.37 x 3.99	4.68	11.71	8.46	.84	Smooth Lacks Resistance
12	11.00	18.00	4.88 x 3.55	4.21	11.92	10.64	1.05	Smooth Lacks Resistance Sl. Soggy
Correlation of per cent sugar to texture and structure measurements				-.97±.02	-.65±.20	+.63±.20	+.96±.02	
Change in texture and structure measurements for each one per cent increase in sugar content				-.2870	+.0657	

TABLE 8.—THE EFFECT OF SUGAR CONTENT UPON TEXTURE AND STRUCTURE

Mix No.	Serum Solids Per Cent	Sugar Per Cent	Ice Crystal Size		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
			Microns			Cells Microns	Crystals Microns	
			Average Dimension	Average Diameter				
13	13.50	12.00	6.33 x 4.90	5.61	9.34	11.31	1.03	Smooth Sl. Coarse Med. Resistance
14	13.50	14.00	4.28 x 3.55	3.91	14.32	12.60	1.12	Smooth Med. Resistance
15	13.50	16.00	4.63 x 3.72	4.17	18.64	10.25	1.45	Smooth Lacks Resistance
16	13.50	18.00	4.23 x 3.46	3.84	13.10	13.25	2.06	Smooth Sl. Soggy
Correlation of sugar to texture and structure measurements				-.77±.13	-.34±.29	+.34±.29	+.94±.04	
Change in texture and structure measurements for each one per cent increase in sugar content				-.2491	+.1543	

TABLE 9.—THE EFFECT OF SUGAR AND GELATIN IN HIGH SERUM SOLIDS ICE CREAM UPON TEXTURE AND STRUCTURE

Mix No.	Serum Solids	Composition in Per Cent		Ice Crystals Size		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
		Sugar	Gelatin	Microns			Cells Microns	Crystals Microns	
				Average Dimension	Average Diameter				
17	15.50	12.00	0.20	4.75 x 4.28	4.51	9.34	7.22	1.03	Med. Smooth Sl. Crumbly
18	15.50	14.00	0.30	4.00 x 3.63	3.81	14.32	8.40	1.12	Smooth Mellow
19	15.50	16.00	0.40	4.21 x 3.72	3.96	8.64	6.83	1.38	Smooth Mellow
20	15.50	18.00	0.50	3.82 x 3.22	3.52	13.10	6.50	1.96	Smooth Sl. Crumbly Sl. Soggy
Correlation of combined sugar and gelatin to texture and structure measurements				-.82±.11	-.26±.31	-.64±.20	+.95±.03		
Change in texture and structure measurements for each one per cent increase in sugar and gelatin content				-.1267	+.1457		

respectively, indicating that sugar is more effective in producing a change in the distribution of the ice crystals, and also the amount of unfrozen material surrounding the crystals in the ice creams of higher serum solids content.

The Effect of Sugar and Gelatin Content Upon Texture and Structure

Table 9 shows texture and structure measurements on high serum solids ice cream containing variable increments of sugar and gelatin.

In a high solids content ice cream there was only a fairly high negative correlation of the sugar and gelatin content to ice crystal size, and there was a low correlation to air cell size and distribution. However, there existed a high positive correlation of sugar and gelatin to the distance between cells. For each increase of one per cent sugar and gelatin, there was a $-.1267$ and a $.1457$ micron change in ice crystal size and distance between crystals, respectively. This indicates that sugar is not very effective in reducing ice crystal size in the presence of high serum solids and gelatin; however, considerable increase in distance between crystals is manifested under these conditions. It seems that after the ice crystals reach a certain degree of smallness, further changes of the components of the mix have little influence upon ice crystal size.

The Effect of Acidity Upon Texture and Structure

The effect of acidity upon the texture and structure of ice creams was studied by preparing a series of mixes containing 11 per cent and also 15 per cent serum solids that ranged from .08 to .24 per cent acidity. The acidity was adjusted by the use of a combined magnesium oxide and calcium oxide standardizer. Table 10 shows the effect of variation in acidity upon texture and structure measurements.

TABLE 10.—THE EFFECT OF ACIDITY UPON TEXTURE AND STRUCTURE

Mix No.	Acidity Per Cent	pH	Ice Crystal Size		Average Air Cell Size	Distance Between	
			Microns			Cells	Crystals
			Average Dimension	Average Diameter	Microns		
21	0.24	6.70	5.07 x 4.25	4.66	10.40	11.80	1.08
22	0.18	6.90	4.25 x 3.74	3.99	12.20	8.60	1.13
23	0.12	7.50	4.43 x 4.92	4.67	12.40	9.90	1.26
24	0.08	7.65	4.88 x 3.87	4.37	11.80	7.70	1.05

From this table it appears that the ice crystals are larger at the higher acidity, and that their size is reduced by a limited acidity

adjustment, and that upon further acidity adjustment the ice crystals again increase in size. Apparently the smaller ice crystals are formed when the pH of the mix is adjusted near the neutral point, with calcium and magnesium standardizers as an acidity adjusting agent.

For a further study of the effect of acidity upon texture and structure an examination was made of 15 per cent serum solids content ice cream. These observations are shown in Table 11.

The effect of acidity, in high serum solids content ice cream, upon texture and structure was similar to that in lower solids ice creams. Here again, the smaller ice crystals occurred in the ice creams having a pH value near the neutral point. The ice crystals were larger in the more acid or the more basic mixes. The same acid standardizer was used in this series of mixes.

The Effect of Variable Sources of Serum Solids Upon Texture and Structure

As serum solids is the constituent that contributes most to the acidity of the ice cream mix, a series of mixes were prepared containing portions of dry milk powder and another powder product of standardized acidity content.

TABLE 11.—THE EFFECT OF ACIDITY UPON TEXTURE AND STRUCTURE

Mix No.	Serum		pH	Ice Crystal Size Microns		Average Air Cell Size Microns	Distance Between Cells Crystals Microns	
	Solids Per Cent	Acidity Per Cent		Average Dimension	Average Diameter		Microns	Microns
25	15.00	0.24	6.60	4.42 x 4.00	4.21	13.90	11.90	1.13
26	15.00	0.18	6.90	3.63 x 3.40	3.51	10.90	8.10	.98
27	15.00	0.12	7.65	4.29 x 3.66	3.97	15.60	10.40	1.18
28	15.00	0.08	8.50	4.38 x 3.70	4.04	13.50	7.80	.84

Table 12 shows a comparison of the effect of different sources of serum solids upon texture and structure. The data indicate that there was very little difference in the effect of milk powder and mineralized milk solids as a source of serum solids upon texture and structure of ice cream. It seems that probably any effect produced was mostly due to the acidity possessed by the different products. The higher acidities encountered in this series of mixes resulted in a slightly larger ice crystal with a smaller amount of unfrozen material visible around the crystals.

TABLE 12.—THE EFFECT OF VARIABLE SOURCES OF SERUM SOLIDS UPON TEXTURE AND STRUCTURE

Mix No.	Composition in Per Cent			Acidity Per Cent	pH	Ice Crystal Size		Average Air Cell Size Microns	Distance Between Cells Microns	Distance Between Crystals Microns
	Dry Milk Solids	Mineralized Milk Solids	Milk and Cream			Average Dimension	Average Diameter			
33	3.00	6.00	6.00	.19	6.75	4.30 x 3.89	4.08	7.57	10.53	1.35
34	6.00	3.00	6.00	.21	6.70	4.18 x 3.45	3.81	8.06	6.06	1.55
35	9.00	0.00	6.00	.31	6.45	4.39 x 4.32	4.35	8.07	6.76	.77
36	7.50	0.00	6.00	.28	6.50	4.97 x 4.26	4.55	8.40	6.06	.61
37	4.50	4.50	6.00	.23	6.83	4.30 x 4.20	4.29	8.40	5.53	1.21

The Effect of Overrun Upon Texture and Structure

Apparently overrun is an important factor influencing the texture and structure of ice cream. Therefore, series of mixes with 85 to 130 per cent overrun were prepared and their texture and structure characteristics were studied as shown in the following table.

TABLE 13.—EFFECT OF OVERRUN UPON TEXTURE AND STRUCTURE

Mix No.	Overrun Per Cent	Ice Crystal Size		Average Air Cell Size	Distance Between	
		Microns			Cells	Crystals
		Average Dimension	Average Diameter	Microns	Microns	Microns
29	85.00	6.27 x 5.12	5.69	16.52	16.20	1.12
30	10.00	5.39 x 4.73	5.06	14.23	12.30	1.00
31	115.00	5.04 x 4.41	4.72	10.90	9.30	.85
32	130.00	4.98 x 4.31	4.64	10.40	9.40	.70
Correlation of overrun to texture and structure measurements			— .88±.06	— .91±.09	— .86±.09	— .97±.02
Change in texture and structure measurements for each 10 per cent increase in overrun			— 0.162	— 1.024	— 1.161	— 0.069

There is a fairly high negative correlation of the per cent overrun to the ice crystal size, the air cell size, the distance between cells, and the distance between crystals. In this series of mixes it was found that for each 10 per cent increase in overrun, there was a change in ice crystal size, air cell size, distance between cells, and distance between crystals of -0.162, -1.024, -1.161, and 1.069 microns, respectively. It can be seen that overrun is important in influencing the size and distribution of air cells and ice crystals.

The Effect of Combined Disaccharide and Monosaccharide Sugars Upon Texture and Structure

Zoller (1924) stated that sugars tend to hold a portion of their water with great resistance because of their molecular structure. It is reasonable to suppose that variable increments of a monosaccharide sugar in combination with a disaccharide sugar should have an effect upon the texture of the resulting ice cream due to the considerable difference in the molecular structure of these sugars.

Since corn sugar is used extensively in the industry to replace portions of sucrose in the manufacture of ice cream, the effect of variable increments of corn sugars upon texture was studied.

Table 14 gives the effect of variable increments of dextrose in an ice cream containing 11 per cent serum solids upon texture and structure measurements.

TABLE 14.—THE EFFECT OF VARIABLE INCREMENTS OF DEXTROSE UPON TEXTURE AND STRUCTURE

Mix No.	Composition		Ice Crystal Size Microns		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
	Sucrose Per Cent	Dextrose Per Cent	Average Dimension	Average Diameter		Cells Microns	Crystals Microns	
38	14.00	0.00	5.85 x 4.99	5.42	13.30	11.92	.76	Smooth Mellow
39	10.00	2.00	5.25 x 4.26	4.75	11.27	7.00	.61	Medium Smooth Sl. Crumbly
40	10.00	4.00	5.46 x 4.45	4.95	10.96	5.00	.79	Medium Smooth Very Sl. Crumbly
41	10.00	6.00	6.95 x 5.97	6.46	15.35	13.80	.96	Medium Smooth
42	10.00	8.00	6.38 x 5.39	5.88	14.32	13.41	1.25	Med. Resistance Very Sl. Coarse
Correlation of Dextrose to texture and structure measurements				+ .70±.17	+ .80±.12	+ .81±.12	+ .99±.01	
Change in texture and structure measurements for each one per cent increase in Dextrose content				+ .2474	+ .6770	+ 1.1890	+ .1058	

TABLE 15.—THE EFFECT OF VARIABLE INCREMENTS OF CERELOSE UPON TEXTURE AND STRUCTURE

Mix No.	Composition		Ice Crystal Size Microns		Average Air Cell Size Microns	Distance Between		Organoleptic Observations
	Sucrose Per Cent	Cerelose Per Cent	Average Dimension	Average Diameter		Cells Microns	Crystals Microns	
43	14.00	0.00	5.51 x 4.45	4.98	8.41	8.80	.71	Smooth Mellow
44	10.00	2.00	6.04 x 5.03	5.53	16.70	8.71	.68	Medium Smooth Sl. Crumbly
45	10.00	4.00	6.38 x 5.39	5.88	13.12	10.51	.81	Medium Smooth
46	10.00	6.00	6.36 x 5.26	5.81	12.74	10.69	.86	Medium Smooth
47	10.00	8.00	6.89 x 5.88	6.36	14.80	13.18	.94	Lacks Resistance Very Sl. Coarse
Correlation of Cerelose to texture and structure measurements				+ .79±.13	— .76±.14	+ .96±.03	+ .99±.01	
Change in texture and structure measurements for each one per cent increase in Cerelose content				+ .1253	— .7403	+ 1.0847	+ .0432	

A fairly high correlation exists between added percentage dextrose to texture and structure measurements.

Added increments of dextrose caused positive changes in the ice crystal size, the air cell size, the distance between cells, and the distance between crystals. This change was .2474, .677, 1.189 and .1058 microns, respectively, for each one per cent increase in dextrose content. The influence of added increments of dextrose upon texture apparently lies in the difference in amount of the water of the mix held by its molecular structure as compared to that of sucrose. Organoleptic observations also show these texture changes.

Table 15 shows the effect of variable increments of cerelose upon the texture and structure. The positive correlation of the per cent cerelose to the ice crystal size was somewhat less than the correlation to the distance between cells and distance between crystals which were rather high. There was a significant negative correlation between added percentage of cerelose and the air cell size of the resulting ice cream.

For each one per cent increase in cerelose content, there was a change of .1253, -.7403, 1.084, and .0432 microns in the ice crystal size, the air cell size, the distance between cells, and the distance between crystals, respectively. These measurements clearly indicate that added increments of the cerelose product produced less change in texture and structure than did added increments of dextrose.

The Effect of Added Increments of Dextrose Accompanied by Increased Serum Solids Upon Texture and Structure

Sommer (1938) stated that not only does the addition of serum solids in the mix replace water, but also reduces the amount of free water by virtue of the fact that the milk proteins hold water as water of hydration. Therefore, if the change in texture and structure, produced by adding dextrose to the ice cream mix, is the result of the lack of the water holding capacity of the dextrose molecule as compared to the sucrose molecule, it is logical to expect that if added increments of dextrose are accompanied by added serum solids, the water of hydration holding capacity of the milk proteins present would counter-balance any texture change that might be produced by the dextrose.

Table 16 gives the effect of variable increments of dextrose accompanied by increased serum solids upon texture and structure. This series of mixes contained 13 per cent serum solids. This table also shows that there is a very low correlation of the dextrose con-

tent of a 13 per cent serum solids mix to the ice crystal size, the air cell size, and the distance between cells. However, there was a high positive correlation to the distance between crystals. These dates indicate that the effect of dextrose upon texture and structure is the result of the lack of the water holding characteristics of the dextrose sugar molecule.

TABLE 17.—STATISTICAL RELATION OF VARIOUS MIX COMPONENTS TO THE TEXTURE AND STRUCTURE OF ICE CREAM

Mix Component (X)	Ice Crystal Size (Y)		Air Cell Size (Y)	
	Correlation	Regression Equation	Correlation	Regression Equation
Fat	-.97±.02	$Y = -.4564X + 11.4932 \pm .24$	-.56±.23	
Serum Solids	-.97±.02	$Y = -.4336X + 9.6560 \pm .21$	-.90±.06	$Y = -1.1524X + 28.978 - .53$
Combined Fat and Serum Solids	-.92±.05	$Y = -.5820X + 18.9980 \pm .20$	-.93±.05	$Y = -.9523X + 40.680 - .51$
Sugar—11% Serum Solids	-.97±.02	$Y = -.2870X + 9.3250 \pm .01$	-.65±.20
Sugar—13% Serum Solids	-.77±.13	$Y = -.2491X + 8.1070 \pm .57$	-.34±.30
Overrun	-.87±.06	$Y = -.0162X + 7.2800 \pm .19$	-.91±.06	$Y = -.1024X + 23.98 - .87$
Dextrose	+.73±.17	$Y = +.2474X + 4.2600 \pm .56$	+.50±.12	$Y = +.6770X + 9.55 - .09$
Cerelose	+.79±.13	$Y = +.1253X + 5.2740 \pm .62$	-.76±.14	$Y = -.7403X + 18.04 - .91$
Dextrose—10% Fat, 13% Serum Solids	+.24±.31	-.25±.31
Sugar and Gelatin	-.81±.11	$Y = -.1267X + 5.8900 \pm .1179$	-.26±.31
Mix Component (X)	Distance Between Air Cells (Y)		Distance Between Crystals (Y)	
	Correlation	Regression Equation	Correlation	Regression Equation
Fat	-.31±.30	-.94±.04	$Y = +.0361X + .16 - .35$
Serum Solids	-.90±.06	$Y = -.7680X + 23.11 + .04$	-.97±.02	$Y = +.1645X - .84 - .23$
Combined Fat and Serum Solids	-.99±.04	$Y = -.1464X + 15.20 \pm .06$	-.97±.02	$Y = +.1899X - 3.52 - .25$
Sugar—11% Serum Solids	+.63±.20	-.96±.02	$Y = +.0657X - .17 - .11$
Sugar—13% Serum Solids	+.34±.30	-.94±.04	$Y = +.1544X - .90 - .12$
Overrun	-.86±.09	$Y = -.1161X + 24.70 \pm .78$	-.99±.02	$Y = +.0069X - 1.66 - .00$
Dextrose	+.81±.12	$Y = +1.1890X + 3.85 \pm 1.15$	-.99±.00	$Y = +.1058X + .37 - .09$
Cerelose	+.96±.03	$Y = +1.0850X + 5.35 \pm .22$	-.99±.00	$Y = +.0432X + .60 - .01$
Dextrose—10% Fat, 13% Serum Solids	-.49±.33	-.99±.00	$Y = +.1027X + .62 - .02$
Sugar and Gelatin	-.64±.20	-.95±.04	$Y = +.1457X - .89 - .11$

Statistical Relationship of Various Mix Components to Texture and Structure

Table 17 presents a statistical analysis of the relationship of various mix components upon texture and structure of ice cream.

Table 17 also gives the correlation of the various factors studied to texture and structure measurements. The regression equations presented enable the calculation of texture and structure measurements for any percentage of mix components within the conditions of this investigation. In order to present in a summarized form the effect of the various mix components studied upon texture and structure, Table 17 was prepared.

TABLE 18.—THE EFFECT OF ONE PER CENT INCREASE OF MIX COMPONENT UPON TEXTURE AND STRUCTURE

	Crystal Size Microns	Air Cell Size Microns	Distance Between Cells Microns	Distance Between Crystals Microns
Fat	—4564	+0361
Serum Solids	—4336	—1.1524	—7680	+1645
Fat and Serum Solids	—5820	.9523	—1464	+1899
Sugar—11% Serum Solids	—2870	+0657
Sugar—13% Serum Solids	—2491	+1544
10% Overrun	—1620	—1.0240	—1.1610	—0690

A combination of fat and serum solids was most effective in reducing ice crystal size. Fat content was somewhat more effective than serum solids content in reducing ice crystal size. Sugar was the next component in importance in reducing crystal size, while overrun was less effective.

Overrun, combined fat and serum solids, and serum solids content affected the size and distribution of air cells.

All the factors considered in Table 18 except overrun, caused a greater distribution of ice crystals. A combination of fat and serum solids produced the greatest effect upon ice crystal distribution. Increased overrun causes the ice crystals to form closer together.

Figure 6 gives a visual presentation of the data shown in Table 18. It also gives a visual summarization of the effect of various mix components studied upon texture and structure measurements of ice cream. Results that did not lend well to graphic presentation have been previously discussed and were not included in this figure.

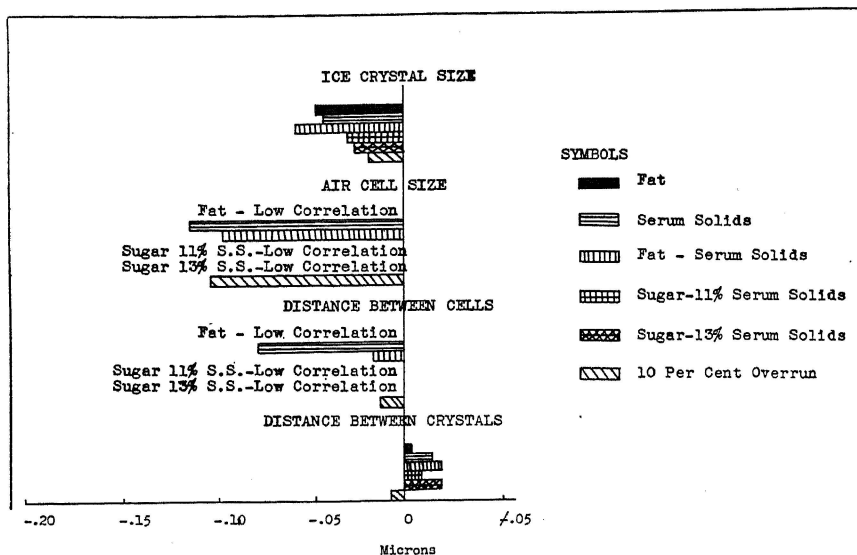


Fig. 6.—Effect of 1 per cent increase of mix components upon texture and structure.

Lactose Crystals in Ice Cream

Lactose crystals sometimes form in ice cream resulting in a texture factor of importance. Therefore, a number of the lactose crystals were studied to investigate their optical properties. Herrington (1934) stated that the crystallization habit of alpha hydrate lactose varied greatly under different conditions. Hunziker and Nissen (1927) described two different shapes of alpha lactose. They stated that one was fully developed having trapezoidal side faces and rhombic tops and bottoms. A modification of this one resembled short stubbed truncated pyramids with flat rhombic base and with apex that lacked full development. They stated that the habit of the crystal was probably the only element of difference between the two crystals, and that the presence of sucrose is an important factor causing the latter to be produced.

Decker, Reid, and Arbuckle (1939) showed photographs of lactose crystals isolated from ice cream that include both alpha lactose crystals described by Hunziker and Nissen (1927) thus showing that these shapes of alpha lactose crystals occur in sandy ice cream.

A series of solutions were prepared with conditions similar to those that exist in ice cream, in order that lactose crystals of various

shapes might be secured. The following were produced: a thin triangular crystal that grew into a fully developed alpha lactose crystal, a thin triangular saw-tooth crystal that developed into lactose crystals and into thick saw-tooth shaped crystals, and a thin diamond shaped crystal that developed a short truncated pyramid shaped crystal.

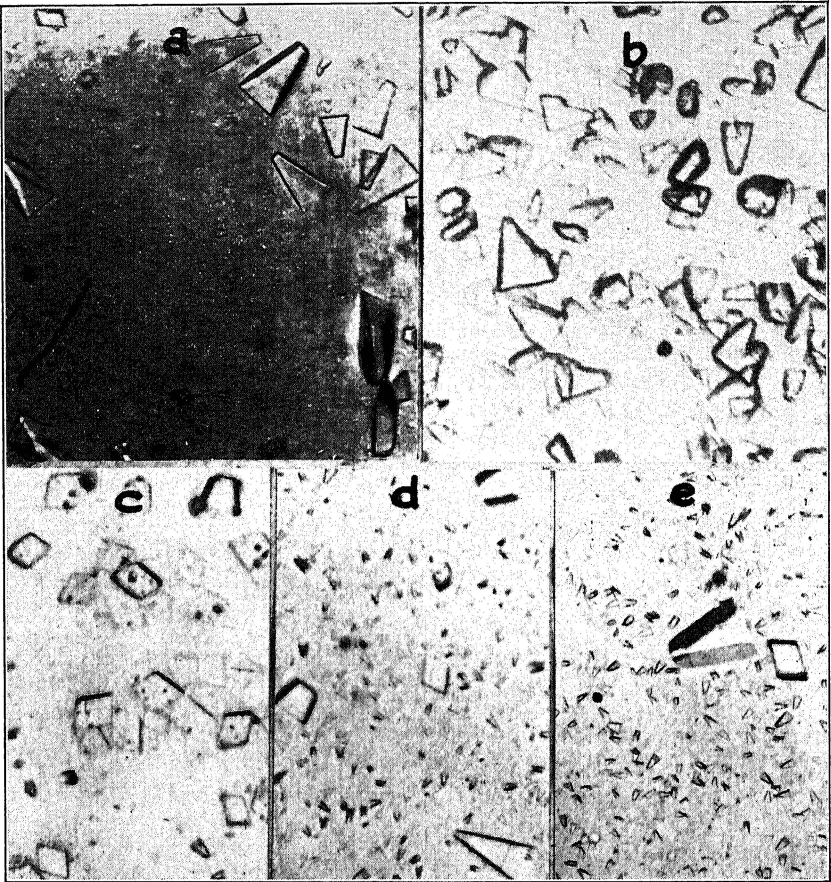


Fig. 7.—Lactose crystals observed. (a) Presents the fully developed lactose crystal and thin triangular crystals that occur in the stages of formation of the fully developed lactose crystal. (b) Saw-tooth lactose crystal. (c) Thin diamond shaped crystals from which the short truncated crystal develops. (d) and (e) Stages in the development of the short truncated crystal.

The crystals of alpha lactose produced in the above series of solutions were isolated and examined with the petrographic microscope to determine their optical properties. Figure 7 shows the stages of formation in the fully developed form of alpha lactose crystal which grew from a thin triangular base. The saw-tooth

crystal also grew from a thin triangular saw-tooth shaped base crystal.

An examination of the different crystals with the petrographic microscope revealed that the optical properties of all the crystals examined were the same. These properties were as follows: form monoclinic; refractive indices, alpha 1.519, beta 1.553, gamma 1.556; optical character negative; extinction angle 24 degrees; optical an-

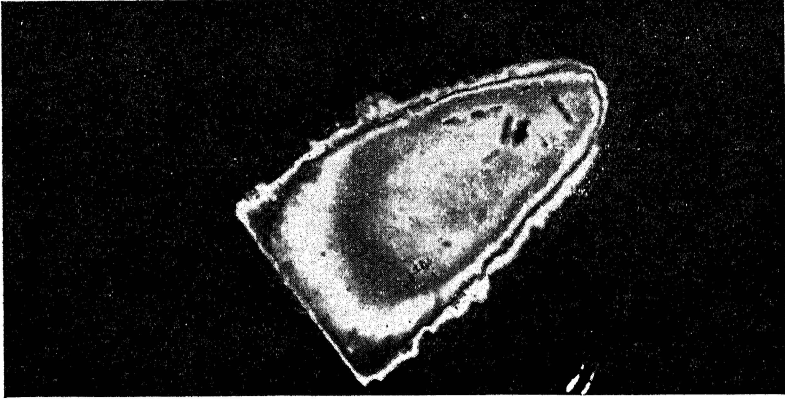


Fig. 8.—Photograph showing the appearance and color order present in the fully developed lactose crystal

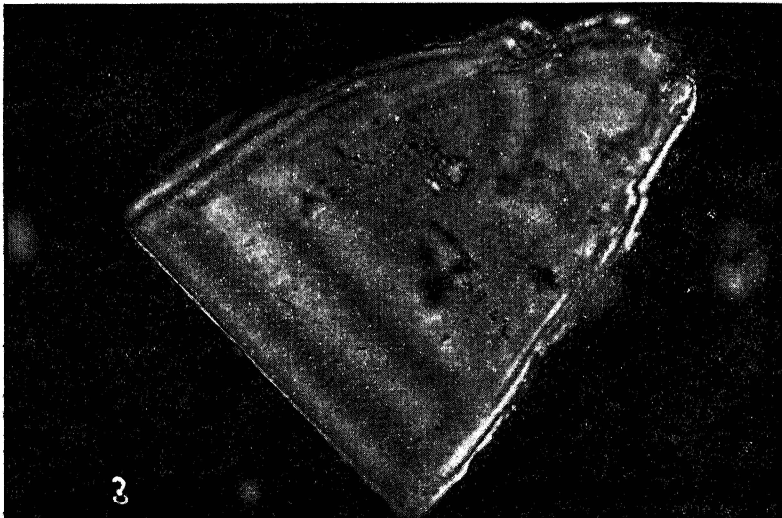


Fig. 9.—Photograph showing the appearance and color order present in the short truncated lactose crystal.

gle 34 degrees; dispersion violet greater than red, and interference colors third order. The crystals observed were the same crystallographically. This is in agreement with the statement of Hunziker and Nissen (1927) that the habit of the crystals was probably the only element of difference.

Figures 8 and 9 show the appearance and color orders of the two type crystals when observed with the petrographic microscope under polarized light.

DISCUSSION

To evaluate the texture of ice cream by microscopic methods it is necessary to consider ice crystal size and structure. Prior to the use of the microscope to examine texture, organoleptic observations were used. Texture determined by these observations is influenced by certain mix components that may act as lubricants and produce a texture change not recognizable or measurable by microscopic methods. The advent of modern freezers made it possible to produce a product of very fine texture. Several factors contributing to texture are so minute that they cannot be detected by organoleptic tests. Microscopic measurements make it possible to detect texture and structure differences that would otherwise be overlooked.

The results obtained in this investigation concerning the effect of fat and serum solids upon ice crystal size are in agreement with those of Dahlberg (1925) and are somewhat contradictory to the conclusions advanced by Cole and Boulware (1940). It was found that fat was more effective in reducing ice crystal size than milk-solids-not-fat, however, the difference was so small it is believed to be of little significance. Apparently the mechanical obstruction of ice crystal formation by the fat is as effective as the water binding properties of the milk proteins in influencing ice crystal size in ice cream.

A greater combined effect of fat and solids-not-fat upon ice crystal size was noted than the effect of either alone. This observation is in agreement with that of many other investigators, and indicates that the mechanical obstruction to ice crystal formation by the fat is coupled with the water binding properties of the milk protein that influence ice crystal formation to produce the decided reduction in ice crystal size.

Microscopic examination revealed that sugar content had but a slight effect in reducing ice crystal size; however, a smoother texture was noted due to the amount of unfrozen material that sur-

rounds the ice crystal in ice creams containing larger amounts of sugar.

It was found that hydrogen, calcium and magnesium ions are conducive to the formation of larger ice crystals. The probable explanation of this is that they promote the clumping of fat and affect the hydration of the milk proteins thus favoring the formation of a larger ice crystal.

Overrun has considerable effect upon texture and structure of ice cream in that increased overrun reduces ice crystal size, decreases air cell size, reduces the distance between cells, and decreases the distance between crystals. It appears that the incorporation of numerous small air cells in high overrun ice cream offers resistance to ice crystal formation and as a result the crystals are smaller and closer together.

The occurrence of a larger ice crystal with the use of a monosaccharide sugar instead of a disaccharide seems to be caused by the difference in water holding capacity as influenced by the hydrogen bondage of the two sugar molecules.

Although several different shapes of lactose crystals have been observed as occurring in ice cream, they are crystallographically the same. Apparently the presence of certain substances in the ice cream mix interferes with lactose crystal formation and results in crystals of variable shapes but they have identical optical properties.

CONCLUSIONS

1. Factors of considerable importance that determine the texture and structure of ice cream are the size of the ice crystal, the size and distribution of air cells, and the amount of unfrozen material present.

2. Optical data indicate that water crystallizes from the mix in the form of pure ice.

3. The effect of various mix components upon texture and structure of ice cream can be determined accurately by microscopic examination.

4. Texture and structure measurements of sections of ice cream may be taken more rapidly and more accurately from photographs than by direct observations at hardening room temperatures.

5. Fat content improved texture chiefly due to the reduction of ice crystal size by mechanical obstruction of crystal formation.

6. Serum solids influenced texture by reducing the ice crystal and air cell size, and by increasing the distribution of air cells and the amount of unfrozen material between crystals.

7. Fat and serum solids had a greater combined effect upon ice crystal size and the amount of unfrozen material between crystals than either fat or serum solids alone.

8. Sugar affects texture by reducing ice crystal size and increasing the amount of unfrozen material between ice crystals.

9. The smallest ice crystals were formed when the pH of the mix was near the neutral point when calcium and magnesium acidity standardizers were used. This indicates that the hydrogen, calcium, and magnesium ions are conducive to the production of larger ice crystals, and that conditions are least favorable for crystal formation when the pH is near the neutral point.

10. Increased overrun decreased ice crystal and air cell size.

11. The texture change resulting from the use of a monosaccharide sugar in combination with a disaccharide sugar is apparently due to the lack of water holding capacity of the monosaccharide sugar molecule.

12. The use of the petrographic microscope to determine the optical properties of the different shapes of lactose crystals that have been observed as occurring in ice cream, revealed that they are crystallographically the same. Apparently the presence of certain substances in ice cream mix interfere with lactose crystal formation to produce the various crystal forms.

BIBLIOGRAPHY

- Alexander, Jerome. 1909 *Function of Gelatin in Ice Cream*. Ice Cream Trade Journal, No. 3, pp. 14-16.
- Alexander, Jerome. 1926 *Influence of Colloids on Crystallization*. Colloid Chemistry, Theoretical and Applied, Vol. 1, p. 624.
- Ambrose, A. S. and Tracy, P. H. 1924 *Factors That Affect Ice Cream Quality*. Ill. Sta. Ann. Rept. 37, pp. 103-104.
- Arbuckle, W. S., Decker, C. W. and Reid, W. H. E. 1939 *The Use of the Petrographic Microscope in Studying the Different Types of Lactose Crystals As They Occur in Sandy Ice Cream*. Journal of Dairy Science, Vol. 22, No. 6, p. 41.
- Baer, A. C. 1916 *Ice Cream Making*. Wisconsin Agr. Exp. Sta. Bul. No. 262.
- Bothel, F. H. 1920 *Facts About Sandy Ice Cream*. Ice Cream Trade Journal, Vol. 16, No. 2, pp. 55-56.
- Brown, R. W. 1913 *Ice Cream Experiments*. Ann. Rept. of Dept. of Agriculture, Ontario, Vol. 1, pp. 92-94.
- Brainerd, W. K. 1915 *Smoothness and Keeping Qualities in Ice Cream As Affected by Solids*. Virginia Agr. Exp. Station Tech. Bul. No. 7.
- Cole, W. C. 1928 *Microscope Exposes Crystallization*. Ice Cream Field, Vol. 13, No. 5, pp. 18, 96, 98.
- Cole, W. C. 1929 *Studies in Freezing Ice Cream*. Ice Cream Field, Vol. 16, No. 5, pp. 15, 44, 102.
- Cole, W. C. 1932 *A Microscopic Study of Ice Cream Texture*. Journal of Dairy Science, Vol. 15, No. 6, pp. 421-427.
- Cole, W. C. and Boulware, J. H. 1940 *Influence of Some Mix Components Upon the Texture of Ice Cream*. Journal of Dairy Science, Vol. 23, No. 2, pp. 140-157.
- Dahlberg, A. C. 1924 *How Ice Cream Looks Under the Microscope*. Ice Cream Trade Journal, Vol. 20, No. 2, pp. 68-69.
- Dahlberg, A. C. 1925 *Factors Influencing the Texture of Ice Cream*. Ice Cream Review, Vol. 9, No. 4, p. 156.
- Dahlberg, A. C. 1925a *The Texture of Ice Cream*. Geneva Exp. Sta. Tech. Bul. 111.
- Dahle, C. D. and Caulfield, W. J. 1926 *Penn. Agr. Exp. Sta., 39th Ann. Rept.* Bul. 204.
- Dahle, C. D. and Bradley, H. H. 1932 *Freezing and Hardening Studies*. Proceedings of the 33rd International Association of Ice Cream Manufacturers, Vol. 2, pp. 28-47.
- Davis, L. M. 1916 *Ice Cream Experiments*. California Agr. Exp. Sta. Rept., p. 48.
- Decker, C. W., Arbuckle, W. S. and Reid, W. H. E. 1939 *Alpha Hydrate and Beta Anhydride Lactose Crystals in Sandy Ice Cream*. Mo. Agr. Exp. Sta. Res. Bul. 302.
- Fry, W. H. 1933 *Petrographic Methods for Soil Laboratories*. U. S. Dept. of Agriculture Tech. Bul. 344.
- Grosse, Aristide V. 1937 *Refractive Indices at Low Temperatures*. Jour. Am. Chem. Soc., Vol. 59, No. 12, pp. 2739-2741.
- Hall, Thomas. 1921 *Microscopic and Thermal Analysis of the Texture of Ice Cream*. Ice Cream Trade Journal, Vol. 17, No. 11, p. 71.
- Harrington, B. L. 1934 *Some Physico-Chemical Properties of Lactose. II. Factors Influencing the Crystalline Habit of Lactose*. Jour. of Dairy Science, Vol. XVII, No. 8, pp. 533-542.
- Holzinger, K. J. 1928 *Statistical Methods for Students In Education*. Boston: Ginn and Company.

- Hunziker, O. F. 1926 *Condensed Milk and Milk Powder* LaGrange, Illinois: 4th Ed., p. 332.
- Hunziker, O. F. 1935 *Condensed Milk and Milk Powder* LaGrange, Illinois: 5th Ed., p. 388.
- Hunziker, O. F. and Nissen, B. H. 1927 *Lactose Crystal Formation*. Jour. of Dairy Science, Vol. X, No. 2, pp. 139-154.
- Keller, W. D. 1934 *Range of the Petrographic Microscope*. The Pan-American Geologist, Vol. 61, pp. 349-354.
- Kucharenko, J. A. 1925 *New Theory of Solution*. Int. Inst. of Agr., Vol. 3, No. 1, pp. 1-9.
- Linderwirth, E. 1926 *Einfluss der Luftzellen auf die Struktur der Rahmeis*. Ztschr. Rahmeis, Vol. 1, No. 2, p. 12.
- Martin, W. H. 1926-1928 *Kansas Agr. Exp. Sta. Bien. Rept.*, p. 97.
- Mortensen, M. 1915 *Mechanism of Overrun in the Manufacture of Ice Cream*. Creamery and Milk Plant Monthly, Vol. 2, No. 1, pp. 21-22.
- Mortensen, M. 1918 *Factors Which Influence the Yield and Consistency of Ice Cream*. Iowa Agr. Exp. Sta. Bul. 180.
- Mortensen, M. 1924 *Some Factors Responsible for Desirable Texture*. Ice Cream Review, Vol. 3, No. 4, p. 146.
- Mortensen, M. 1925 *Air Cells and Ice Cream Texture*. Ice Cream Trade Journal, Vol. 21, No. 1, p. 48.
- Mueller, W. S. and Button, F. C. 1929 *The Use of Dehydrated Egg Products in the Manufacture of Ice Cream*. Journal of Dairy Science, Vol. 12, No. 4, p. 320.
- Munkwitz, R. C. and Meade, Devoe 1929 *Sharp versus Dull Freezer Blades and Freezer Efficiency*. Maryland Bul. 324.
- Ostwald, W. 1897 *Zeitschr. f. physikal chem.*, Bd. 22, S. 289.
- Reid, W. H. E. 1933 *Microscopic Study of the Crystalline Structure of Different Ice Creams*. Abstracts of Papers, 28th Meeting of American Dairy Science Association, Univ. of Ill., p. 32.
- Reid, W. H. E., Cooley, R. J. and Arbuckle, W. S. 1940 *Unpublished data*, University of Missouri.
- Reid, W. H. E., Decker, C. W. and Arbuckle, W. S. 1940. *Unpublished data*, University of Missouri.
- Reid, W. H. E., Drew, R. J. and Arbuckle, W. S. 1939 *The Effect of Composition and Serving Temperature Upon Consumer Acceptance and Dispensing Qualities of Ice Cream*. Mo. Agr. Exp. Sta. Res. Bul. 303.
- Reid, W. H. E. and Hales, M. W. 1934 *The Relation of Freezing Procedure and the Composition of the Mixture to the Physical and Crystalline Structure of Ice Cream*. Mo. Agr. Exp. Sta. Res. Bul. 215.
- Sherfy, Carrie B. and Smallwood, Nell W. 1928 *U. S. Dept. of Agriculture Bibliographical Contributions*, No. 17, pp. 152-154.
- Sommer, H. H. 1938 *Theory and Practice of Ice Cream Making*. 3rd Ed.
- Troy, H. C. and Sharp, P. F. 1930 *Alpha and Beta Lactose in Some Milk Products*. Journal of Dairy Science, Vol. 13, No. 2, p. 140.
- Turnbow, G. D. 1928 *Newer Phases in Processing the Ice Cream Mix*. Ice Cream Field, Vol. 13, No. 4, pp. 17, 110, 126.
- Turnbow, C. D. 1928a *A Few Factors Affecting Crystallization*. Proceedings of the 28th Convention of the International Association of Ice Cream Manufacturers, Vol. 2, p. 86.
- Washburn, R. M. 1910 *The Principles and Practices of Ice Cream Making*. Vermont Agr. Exp. Sta. Bul. 155.
- West, C. D. 1939 By correspondence.

- Williams, O. E. and Peter, P. N. 1930 *A New Form of Lactose Crystal Found in Sandy Ice Cream*. Journal Dairy Science, Vol. 13, No. 6, pp. 471-477.
- Winchell, A. N. 1931 *The Microscopic Character of Artificial Inorganic Solids Substances or Artificial Minerals*. London: Capman and Hall, 2nd Ed.
- Young, S. W. 1911, 1913 *Mechanical Stimulus to Crystallization in Super-cooled Liquids*. Journal Am. Chem. Soc., Vol. 33, No. 2, pp. 148-162; Vol. 35, No. 9, pp. 1067-1078.
- Zoller, H. F. 1921 *Separation of Ice in Freezing Ice Cream Mixes*. Ice Cream Trade Journal, Vol. 17, No. 8, p. 40; Vol. 17, No. 9, pp. 45-47; Vol. 17, No. 10, pp. 50-52.
- Zoller, H. F. and Williams, O. F. 1921 *Sandy Crystals in Ice Cream*. Journal of Agr. Research, Vol. 21, No. 10, p. 791.
- Zoller, H. F. 1924 *Measuring the Refrigeration Used in Making Ice Cream*. Ice Cream Trade Journal, Vol. 20, No. 6, pp. 55-56.