A STUDY OF THE MANUFACTURE OF WATER ICES AND SHERBETS

A. C. DAILEBEG

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A. C. DAHLBERG

ABSTRACT

The interest recently manifested by manufacturers of ice cream in improved methods of making water ices and sherbets and the lack of investigation of the manufacture of these products prompted this study.

The hardness of water ices and sherbets could be made comparable to that of ice cream at the same temperature by the proper percentages of overrun and sugar. For ordinary conditions it was found that the overrun should be 20 to 30 per cent and the percentage of sugar 30 to 33.

The unfrozen syrup in water ices and sherbets drained out during storage unless some stabilizing substance was added to hold it in place. Gums and gelatin have been used for this purpose.

India gum, which is extensively used at the present time as a stabilizer, prevented most of the syrup from draining out of the frozen product, but it did not prevent the crumbly body which is undesirable.

Gelatin, by forming a jelly in the water ices and sherbets, made a smooth product, free from crumbliness and drainage of syrup; but it was difficult to prevent excessive incorporation of air during the freezing process. When used in conjunction with low-grade india gum, the action of the gelatin and gum was lessened to a large extent by the precipitation of one stabilizer by the other. Gelatin and gum tragacanth or high-grade india gum did not precipitate each other, but these gums in solution could be readily whipped.

Agar in concentrations of approximately 0.2 per cent gave all of the desirable action of gelatin, but the product was difficult to whip. Milk proteins, gelatin, gum tragacanth, or a high grade of india gum were used in conjunction with agar to permit the incorporation of the proper amount of air. This combination gave the most satisfactory results.

Excessive hardening of water ices and sherbets at their exposed surfaces during storage was caused by crystallization of sucrose from
the supersaturated solution accompanied by subsequent increased proportions of ice. This condition was prevented by substituting corn sugar, which is chiefly dextrose, for 20 to 25 per cent of the sucrose. This mixture of sugars was more soluble than sucrose alone and was not supersaturated until the temperature was below $-20^\circ$ C. ($-4^\circ$ F.).

INTRODUCTION

Water ices and sherbets are made by manufacturers of ice cream to satisfy the consumers' demand for frozen products which differ considerably in their characteristics from normal ice cream. Water ices and sherbets are coarser in texture, less rich, more watery to the taste, sweeter, and higher in fruit acid than normal fruit ice cream. They are especially advantageous for adding variety to brick and fancy molded ice cream combinations.

Much confusion exists concerning the definitions as well as the desirable characteristics of water ices and sherbets. Frandsen and Markham (1919)\(^1\) in their book on ice cream differentiate between water ices and sherbets on the presence or absence of egg albumen or gelatin. They state, "The water ices are made from fruit juices diluted with water, sweetened, and frozen. Sherbets have practically the same composition but contain in addition egg albumen and sometimes gelatin." Milk sherbets apparently are not the same as ordinary sherbets because they state, "Milk sherbets are made by substituting whole or skim milk for the water called for in the ordinary recipe." The definitions given by Fisk (1923) in his book on ice cream agree with those given above.

It is self evident to those familiar with the ice cream trade that if these definitions are acceptable little or no water ice is manufactured. Water ices made without stabilizer are commercially impossible to market because the syrup drains out of the product on standing, leaving a crumbly, hard, somewhat tastless substance to be eaten for water ice. The ice cream industry does not usually distinguish between water and milk sherbets.

The following definitions are more applicable to present trade conditions and define the terms as used in this publication.

A water ice is a semi-frozen product made of water, sugar, and fruit juice which may or may not contain added color, flavor, fruit acid, and stabilizer.

\(^1\text{Refers to Literature Cited, page 30.}\)
A sherbet is a semi-frozen product made of the same ingredients as a water ice except that it also contains milk or milk products. The term semi-frozen is used to describe the products because all of the water in these products, together with that in ice cream, is never completely frozen.

According to these definitions, water ices and sherbets as defined by Frandsen and Mørkham and by Fisk are all water ices, while the milk sherbets of these authors are the real sherbets. These definitions permit a clear cut distinction between the two products in question, namely, the presence or absence of milk or milk products. They also permit the commercial manufacture of water ices because these products can contain substances to make them retain their desirable characteristics during the time required for their marketing.

SCOPE OF THE INVESTIGATION

Several difficulties are encountered in the commercial manufacture of water ices and sherbets. There is a tendency for the unfrozen syrup to drain out of the semi-frozen product during storage in the hardening room even tho the temperature is held below —17.7°C. (0°F.). When this occurs, the water ice or sherbet becomes exceedingly hard in the upper part of the container and very soft, mushy, and sweet in the lower part. The usual practice among ice cream manufacturers partially to prevent this condition is to use a substance known to the trade as india gum. This gum, according to information received from certain dealers, is a low grade of gum tragacanth. It swells in the water making the product so viscous that movement of unfrozen water is interfered with. Sufficient india gum to handle the situation completely cannot be used because it would make a sticky, viscous product which is undesirable. Furthermore, the product usually becomes too hard to serve in one to two weeks time and its body is too crumbly. Gelatin has also been used either alone or in combination with the india gum.

It was the intent of this investigation to devise methods of preparing water ices and sherbets which would enable a manufacturer to control the characteristics of the body and texture. Several conditions must be satisfied. The syrup in the products must be held in place as long as the temperature of the product is not above the temperature at which ice cream can be stored. The hardness of the products must be such that they can be readily dish and eaten at the same temperature at which ice cream can be dish and eaten.
The body of the products must not be crumbly so that they can be served in cones or dishes. The texture must not be very coarse, yet it should not be as smooth as ice cream. The products must melt at ordinary room temperature and return to a liquid condition. Lastly, these characteristics in the freshly frozen products must remain during their usual period of storage which varies from one to four weeks.

Stabilizers used for this purpose must either be rather free from odor and flavor or have a desirable flavor and odor. They must not be injurious to health, altho there is no reason why they must be digestible and beneficial to health.

**EXPERIMENTAL**

**INFLUENCE OF PERCENTAGE OF SUGAR UPON THE HARDNESS OF WATER ICES**

Several investigators have found that the hardness of ice cream decreases with increased sugar content, a fact well recognized by most ice cream manufacturers. It has been shown by the author (1925) that the sugar in ice cream prevents the complete freezing of the water by forming a very concentrated solution and in this manner alters the hardness and smoothness. The sugar (sucrose) content of water ices and sherbets is usually about double that of ice cream so that sugar may be an important factor in determining their body and texture.

A series of water ices were prepared containing 26, 28, 30, 32, 34 and 36 per cent of cane sugar. The syrup was held in position in the frozen product by the use of sufficient gelatin to form a weak jelly in the hardening room. The percentage needed for the grade of gelatin used in the experiments was found to be 0.9 calculated as percentage of gelatin in the water, disregarding the sugar. This determination was made by the method previously described by the author (1925). The water ices were frozen in a specially constructed battery of three 1-gallon freezers and complete freezing data were recorded.

The water ices were taken in the freezers into the hardening room where they were transferred to 1-quart brick molds. After one to two weeks storage at a temperature of approximately $-17.7^\circ$C. ($0^\circ$F.) tests for hardness were made in duplicate on each lot of water ice. Ice cream manufacturers store their products at various temperatures and all water ices, sherbets, and ice creams are not of the
proper hardness for dishing and eating at the same temperature. Hence, it was desirable to determine the hardness of the water ices at various temperatures. Changes in the hardness of water ices, sherbets, or ice creams take place rather slowly and considerable time must be allowed for the samples to adjust themselves to new temperatures. After a few tests an arbitrary standard was adopted of allowing not less than four hours for the 1-quart brick of water ice to become adjusted to each change in temperature of 3.0°C. (5.4°F.) or less.

When hardness tests were to be made the refrigerator was cooled to $-23.0^\circ$C. ($-9.4^\circ$F.) or less and the temperature in the brine stored in the refrigerator was several degrees lower. Several hours later the first tests were made. As the temperature of the room increased tests were made when the temperature changed about $2.5^\circ$C. It was always necessary to keep the refrigerator door slightly ajar part of the time to raise the temperature rapidly enough to permit six tests to be made over a period of 48 hours.

The hardness tests were made with the apparatus devised by Perkins (1914) for determining the hardness of fats. Some improvements in this apparatus might be made to adapt it to water ices and ice cream, but fairly satisfactory results can be obtained with the standard equipment. In accordance with the recommendation of Perkins, the results were calculated to give the number of grams required to displace 1 cubic millimeter of the tested substance, in this case water ice.

The first half of Table 1 gives the influence of the percentage of sugar upon the hardness of water ices with high percentages of overrun at temperatures ranging from $-10.2^\circ$C. ($+13.7^\circ$F.) to $-23.0^\circ$C. ($-9.4^\circ$F.). The result of many trials showed that the proper hardness of water ices for dishing with an ordinary automatic ice cream disher gave tests of 1 to 2 grams. Products with very low overrun, 15 to 20 per cent, can be dished when somewhat softer, while products with high overrun, such as these particular water ices, should be dished when somewhat harder. Hardness varying from 0.7 to 3.0 grams will cover the entire group of ice cream products. Considering a hardness of 2.0 grams to be proper for these water ices, the table shows that when the sugar content was 26 per cent, which is too low for commercial use, and the overrun 70 to 90 per cent, the temperature for good dishing was approximately $-17.0^\circ$C. The correct dishing tem-
perature for water ices containing 36 per cent sugar and 70 to 90 per cent overrun was $-23.0^\circ\text{C.}$ which is much too cold for practical purposes. An increase of 10 per cent in the sugar content of water ices decreased this temperature for good dishing $6^\circ\text{C.}$ (10.8°F.).

Less overrun was needed to harden the water ices to permit the use of 30 to 33 per cent sugar. It was not practical to hold the overrun below 50 per cent with gelatin as the stabilizer. India gum was used alone in place of gelatin. One per cent of this gum was needed to fix the syrup so that it would not drain out in the hardening room, but the product containing this quantity of gum possessed some undesirable characteristics from a commercial standpoint. However, a product with a low overrun was obtained. The second part of Table 1 gives the average hardness of two different batches of water ices with overruns varying from 12.6 to 16.0 per cent and sugar contents ranging from 26 to 36 per cent. Most of these water ices were much too hard for dishing when compared with ice cream and needed to be melted too much before they could be dished properly. The data show that the effect upon hardness of an increase of 1 per cent in the sugar content of these water ices can be counteracted by a decrease in storage temperature of 0.5° to 1.0°F.

**Influence of Percentage of Overrun Upon Hardness of Water Ices**

Variations in the sugar content of water ices used in these experiments did not exert sufficient influence upon the hardness of the ices to use such variations as a method of control. Variations in the percentage of air incorporated in the water ices apparently exerted a great influence over hardness and a study was made of this factor.

The percentage of overrun was determined in the usual manner by weighing a unit volume, about 1 pint, of the water ices before and after freezing. The percentage of overrun was calculated as the percentage which the decrease in weight due to the incorporation of air was of the weight of the product after the air had been incorporated.

Considerable difficulty was experienced in obtaining any desired overrun from 10 to 100 per cent until this work was nearly completed. After all other investigations had been completed, the data on the hardness of all the water ices containing 33.0 per cent of cane sugar were brought together and summarized in Table 2. Some special batches were also prepared to furnish data to complete the table.
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<th>Water Ice's, Percentage of Overrun</th>
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<td>24.0</td>
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TABLE 2.—Influence of Percentage of Overrun Upon Hardness of Water Ice's.

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<th>Hardness at 10°C</th>
<th>Hardness at 12°C</th>
<th>Hardness at 14°C</th>
<th>Hardness at 16°C</th>
<th>Hardness at 18°C</th>
<th>Hardness at 20°C</th>
<th>Hardness at 22°C</th>
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TABLE 1.—Influence of Percentage of Sugar Upon Hardness of Water Ice's.
The data presented include 35 different batches of water ices upon which hardness tests were made in duplicate or triplicate. Some evident discrepancies in the data were caused by the difficulty of obtaining close-checking freezings which were often made months apart and because various stabilizers had to be used to obtain the different percentages of overrun. A discussion of the stabilizers will be given later.

For the purpose of comparison the hardness of ice cream is also given. This ice cream was made to contain 12 per cent of milk fat and 10 per cent of milk solids not fat. The sugar contents were 13, 15, and 17 per cent, respectively, while the overrun was held as near 80 per cent as possible. The mix was pasteurized at 62.7°C. (145°F.) for 30 minutes and homogenized without cooling at 2,000 pounds pressure. It is probable that the ice cream with 13 and 15 per cent sugar was slightly harder than average commercial ice cream because it required a temperature from −12.5°C. (+9.5°F.) to −10.0°C. (+14.0°F.) to soften the ice cream enough for good dishing.

The data clearly show that to obtain a water ice that will be of proper hardness to dish at a temperature of −15.5°C. (+4.1°F.) the overrun should be between 20 and 30 per cent when the sugar content of the water ice is 33 per cent; especially considering the fact that a product with a lower overrun is too heavy and cold for most satisfactory consumption. It will also be noted from Table 2 that if the temperature of dishing can be held fairly constant the percentage of overrun can be increased 10 or 20 per cent without softening the body very much. However, if the temperature rises a few degrees the product with high overrun rapidly becomes too soft to serve and this is another reason why the overrun should be held relatively low in commercial practice.

When sherbets are made, the addition of milk products slightly increases the hardness so that sherbets should be whipped to have about 5 per cent more overrun than water ices. This statement is approximately correct when the sherbets contain either 10 per cent ice cream mix, 50 per cent of milk, or 50 per cent of skimmilk. It is recommended that water ices should be frozen to yield 20 to 25 per cent overrun and sherbets should be frozen to yield 25 to 30 per cent overrun. Water ices and sherbets made with these overruns and with 33 per cent sugar were in excellent condition for dishing at −15.5°C. A higher overrun was satisfactory when a lower temper-
ature of dishing was desired or when the sugar content was reduced to 28 or 30 per cent.

The influence of the percentage of sugar and overrun upon the hardness of water ices is compared graphically with the hardness of ice cream in Chart I. The chart shows how much harder or softer water ices can be made than ice cream by varying these two factors. It will be observed that very hard water ice could be dished at a very narrow range of temperature, while the water ice which compared most favorable with the hardness of ice cream could be dished over a rather wide range of temperature. The water ice with the highest overrun became too soft to handle when the ice cream was still in satisfactory dishing condition. The chart clearly illustrates how the hardness of water ices can be varied by the percentages of sugar and overrun to obtain the desired hardness in the finished product.

INDIA GUM AND GUM TRAGACANTH AS STABILIZERS

The extensive use of india gum in the manufacture of water ices and sherbets prompted a study of this stabilizer first. Three different grades of india gum\(^2\) and a high grade of gum tragacanth\(^3\) were secured for these tests. Another lot of very low-grade india gum which had been purchased for use in water ices was also used in the experiments. These various lots of india gum will be designated as Grades 1, 2, 3, and 4, respectively.

It was essential to know how much gum was necessary to hold all the syrup in the frozen water ices so that no syrup drained out in the hardening room. This was determined by preparing water ices with varying quantities of the different gums and placing them in 1-quart brick boxes in the hardening room. The brick boxes had covers on two sides, and since one cover was placed at the bottom, examination of the top and bottom of the water ices could be readily made after the water ices had been stored in the hardening room. Tests of two different series of water ices made with gum tragacanth and with Grades 1, 2, 3, and 4 of the india gum showed that approximately 0.5, 0.5, 0.6, 0.7, and 1.0 per cent, respectively, of the various

\(^2\)These samples of india gum were secured thru the courtesy of S. Gumpert Co., Brooklyn, N. Y.
\(^3\)The gum tragacanth was secured from Arthur H. Thomas Co., Philadelphia, Pa.
Chart I.—Comparison of Hardness of Ice Cream with Water Ices of Varying Percentages of Sugar and Overrun.
stabilizers were necessary to hold all of the syrup so that it would not drain out of the hardened water ices. The highest grade of india gum was just as efficient as the gum tragacanth and could be purchased at a much lower price.

Several observations of practical importance were made. When the percentage of gum used was sufficient to hold the syrup intact, the water ices were still crumbly in texture. The drainage of the syrup out of water ices caused them to become very hard in the upper portion of the container and very cold to eat. This effect was doubtless due to the increased proportion of actual ice to the total weight of the water ices due to the removal of the unfrozen portion. The lower grades of gum imparted a peculiar flat taste to water ices and sherbets that was detected in concentrations of 0.5 per cent and was easily recognized in concentrations of 0.7 per cent or more. This flavor was objectionable and necessitated the use of extra fruit flavor and acid to obtain the desired results.

The overrun which could be obtained with these various gums depended upon the quality of the gum. Using the same order as previously given in arranging the gums, namely, gum tragacanth, india gum Grades 1, 2, 3, and 4, water ices containing 0.7 per cent of each gum could be whipped according to the average of several trials to 83, 64, 59, 51, and 12 per cent of overrun, respectively. High grade gum permitted the incorporation of much air in water ices, whereas low grade gum had very little effect. The crumbliness of water ices and sherbets was increased with increased percentages of overrun.

Mixtures of high and low grade gums were made by trial until overruns between 12 and 51 per cent were obtained. Much difficulty would be encountered by ice cream manufacturers in controlling overrun in this way because no method is available for determining the whipping properties of a gum except by trial. Each lot of gum may be different and new tests in the freezer would need to be made each time gum was purchased. However, it may be possible for manufacturers of india gum and gum tragacanth to place upon the market gums of known whipping properties.

**GELATIN USED ALONE OR IN COMBINATION WITH GUM AS A STABILIZER**

Gelatin is not used as extensively as india gum in the manufacture of water ices and sherbets probably because it permits the easy in-
corporation of air to such an extent that the frozen products are too light in body. It appeared reasonable that mixtures of gelatin and gum might be made that would control the overrun and prevent crumbliness thru the formation of jelly by the gelatin.

Water ices prepared using gelatin as the only stabilizer need just enough gelatin to form a weak jelly in the product during the freezing process. Distinct traces of jelly formation should be evident in the product before freezing if it is held at 4.4C. (40°F.) for several hours. In these experiments with gelatin as the sole stabilizer 0.9 per cent gelatin in the water, or 0.59 per cent expressed on the basis of the total mixture, was used. Water ices prepared in this manner whipped readily to 100 per cent overrun and difficulty was encountered reducing the overrun to 50 per cent. In spite of the high overrun, these water ices were smooth and not crumbly, thus showing that a gel formation in the water ices would eliminate crumbliness and produce a smooth product. This effect was not obtained if the quantity of gelatin used was insufficient to form a gel. Furthermore, no syrup ever drained out of the water ices in the hardening room if a gelatin gel was present.

An attempt was made to control the overrun thru combinations of the gelatin with gums. In the first trials 0.7 per cent gelatin was combined with 0.35 per cent gum tragacanth and an overrun of 135 per cent was obtained. The gelatin was then combined with the lowest grade of india gum which gave low overruns. It was learned that only 0.2 per cent gelatin was sufficient to give enough overrun with this low-grade gum. Consequently, 0.2 per cent gelatin was used in all the water ices in the experiment, together with the low-grade gum in percentages varying from 0.2 to 1.0 per cent. In this manner overruns varying from 33 to 65 per cent were obtained. The greater the percentage of gum, the lower the overrun would be when this low-grade gum was used. The quality of the water ices made in this manner was very unsatisfactory. Syrup drained out of them in the hardening room when the percentage of gum was below 0.6 and the body was crumbly in every instance. The quantity of gelatin was not sufficient to form a gel and prevent the crumbly body.

It was believed that the crumbly body could be eliminated if sufficient gelatin were used, but it was necessary to hold down the overrun by the use of a larger percentage of low-grade gum. In another series of tests all water ices contained 0.5 per cent of gum and
the gelatin content was varied from 0.2 to 0.8 per cent. The overrun increased with increased quantities of gelatin and ranged from 29 to 86 per cent. Strange as it seemed to be, none of these mixes showed traces of gel formation during aging, and the water ices were crumbly and syrup leaked from them in the hardening room. These results clearly show that, altho the extent of overrun can be controlled by the proper relation of gelatin to low-grade india gum in water ices, the stabilizing ability of both is impaired by their presence together in the same solution.

The reason for the neutralizing effect of gelatin and india gum upon each other as stabilizers was studied. The presence of citric acid, normally used in water ices and sherbets, had no effect upon the stabilizing action of gelatin and gum alone or in combination with each other. Likewise, heating the water ices to 60°C. (140°F.) had no effect upon the stabilizing action of gelatin and gum alone, but in combination with each other a dense white precipitate collected on the surface of the water ices when both gum and gelatin were present. This precipitate was also noticeable even tho the water ices were not heated, but it did not come to the surface. No attempt was made to learn the reason for the mutual precipitation of gelatin and india gum in the same solution. Certain facts were noted. The gelatinizing power of the gelatin was reduced to less than half its normal strength by the very low-grade india gums and the stabilizing value of the gums were similarly destroyed. No precipitation occurred when gelatin and gum tragacanth or high-grade india gum in solutions were brought together and all the stabilizers functioned normally in water ices.

It is apparent that gelatin should not be used in combination with low-grade india gum to control the overrun because of the precipitation of these two stabilizers. Gelatin can be used with high-grade india gum without destroying the strength of the stabilizers, but high-grade india gum or tragacanth give sufficient overrun without gelatin and with gelatin the overrun becomes excessive. These statements are true for the particular stabilizers used in these experiments and which are now being sold for use in the manufacture of ice cream. They may not be true for all gelatins and gums.

AGAR USED ALONE OR IN COMBINATION WITH INDIA GUM OR GELATIN

The results obtained with gelatins and gums in water ices and sherbets clearly indicated that the crumbly condition could be
eliminated by gel formation. This observation also held true to a lesser degree with ice cream. An attempt was made to use some other gelatinizing material in place of gelatin with the hope that it might not whip readily.

Agar was tried because of its availability and great gelatinizing strength. Tests were first made to determine the minimum quantity of agar needed to form a weak gel at 4.4° C. (40°F.). It was found that 0.2 per cent formed a gel and traces of gel could be observed in a solution of 0.15 per cent concentration. Agar exhibited the same characteristics as gelatin in that the temperature required for gelatinization depended upon the concentration of the solution. This was fortunate, because an agar solution of approximately 0.20 per cent could then be used with complete satisfaction, so far as gelatinization was concerned, as a gel would be present in the frozen water ices and would be absent when they were melted at ordinary room temperatures. The ability to melt completely to the liquid condition at temperatures above those maintained in refrigerators is a desirable characteristic of good water ices and sherbets.

Water ices were prepared using 0.10, 0.15, 0.20, 0.25, and 0.30 per cent of agar. It was found that 0.10 per cent was not sufficient to prevent entirely the drainage of unfrozen syrup in the hardening room and the water ices were slightly crumbly. Water ices with 0.15 and 0.20 per cent agar were very satisfactory in these respects because the agar had gelatinized in both cases. Increased quantities of agar produced a product with too stiff a gel for commercial purposes. The overrun obtained varied around 10 per cent. Since this overrun can be obtained with plain sugar solutions, it is evident that agar cannot be of any value in permitting the incorporation of air. In spite of its low protective colloid action in holding air in the syrup solution, it has nevertheless a great effect in making water ices smooth and not crumbly. This latter action is much greater than for similar quantities of gelatin, altho gelatin is a protective colloid of much merit. These facts further substantiate the previous contention of the author (1925) that the action of gelatin in ice cream is due to the formation of a gel.

Agar used alone in water ices made them smooth, not crumbly, and prevented the drainage of unfrozen syrup out of the water ices in the hardening room. Furthermore, water ices containing sufficient agar or gelatin can be subjected to changes in temperature without affecting their hardness or texture more than the same temperature
variations would have altered ice cream. Some other ingredient was necessary to permit the incorporation of air in larger amounts to prevent the body from being too hard and to prevent the water ice from being too cold to eat.

Obviously, gelatin might be used to increase the overrun. Water ices made with 0.15 per cent of agar and 0.2 per cent of gelatin gave overruns varying from 35 to 40 per cent. A decrease of 0.05 in the percentage of gelatin reduced the overrun about 15 per cent, while increases in the percentage of gelatin increased the overrun. Gelatin and agar both form gels so that the action of one is augmented by the other.

Agar and gum can also be satisfactorily used in combination because they do not react together in the same solution as do gelatin and gum. India gum of high quality or gum tragacanth must be used with the agar because the gum must be easily whipped in solution. Two water ices were prepared using 0.2 per cent agar and 0.5 per cent gum tragacanth. They were whipped to 50 and 53 per cent of overrun and made very satisfactory water ices, except that the body was a trifle soft due to excessive overrun. When the percentage of gum tragacanth was reduced to 0.2, the overrun was decreased to 46 per cent. Sufficient overrun could always be obtained when gum tragacanth was used in combination with agar.

Tests were also made using the highest grade of india gum available which was in reality a lower grade of gum tragacanth. When 0.15 per cent agar and 0.20 per cent of india gum of highest grade were used together, an overrun of 20 to 25 per cent was obtained. Another lot of agar was used which tended to hold down the overrun to a greater extent, showing that agar is not uniform in this property. However, by the use of an additional quantity of gum the proper overrun could again be obtained. Since an overrun of 20 to 25 per cent on water ices gave the best body, this combination of stabilizers was especially suitable. The action of agar being due to gel formation and the action of gum being due to soaking up the water and increasing the viscosity, these two stabilizers supplemented each other in an effective manner. The gum also gave substance to the body of the water ices so this combination proved to be the most satisfactory of any tried.

Sherbets were also satisfactorily prepared using agar alone and in combination with gum as the stabilizers. It is probable that most sherbets are now commercially manufactured from water ices to
which some ice cream mix has been added. When an overrun between 20 and 25 per cent was desired, it could be obtained by using 0.15 or 0.20 per cent of agar in a water ice which contained 10 per cent of ice cream mix by weight. It is preferable that the ice cream mix should be only the milk products without added sugar and stabilizer, because it is easier to calculate the ingredients and make comparable sherbets. Furthermore, the gelatin may precipitate the india gum if a rather low grade has been used. The overrun could be readily increased to 30 or 35 per cent by the use of 0.20 to 0.40 per cent of high grade india gum or gum tragacanth. Very high quality sherbets were also made by substituting milk to the extent of 25 to 50 per cent of the entire water ice for part of the water that would normally have been used. The use of milk had the advantage over ice cream mixes in that it was lower in cost so that it could be readily used in varying quantities to control the overrun without seriously altering the cost of the mix. Experimental sherbets with 25 per cent of milk whipped to 20 per cent overrun, while those of 50 per cent of milk were made to contain slightly over 30 per cent overrun. Substitution of skim milk for whole milk increased the overrun but resulted in a product of less desirable flavor.

Powdered agar was preferable to granulated or shredded agar because it dissolved so much more quickly. The agar was stirred into approximately 50 times its weight of boiling water. If the water was boiling when the agar was added, it completely dissolved in less than five minutes, providing the water was kept boiling slowly and continuously after the agar had been added. The boiling does not injure the gelatinizing strength of the agar providing no acid is present in the water. If fruit acid, fruit juices, etc., are present when the agar is in the boiling water, the agar will be hydrolyzed and its action greatly inhibited or completely destroyed. The hot agar solution was poured into the water ices or sherbets when the latter were being rapidly stirred. Unlike gelatin, agar acts quickly and aging of the water ices or sherbets containing this stabilizer was unnecessary. Good agar should be practically free from both flavor and odor and it has little or no effect in this respect upon water ices and sherbets.

India gum or gum tragacanth could be most satisfactorily added to the water ices or sherbets after they had been thoroughly mixed with part or all of the sugar. The sugar with gum was slowly poured into
the water ices or sherbets when the latter were being stirred rapidly. The low grade india gums used in these experiments were brownish yellow in color and had a distinct flavor. Gum tragacanth or high-grade india gums were pure white in color and almost free from flavor. The low-grade gums when dissolved in gelatin solutions formed a white precipitate which was especially noticeable if the solutions were hot. The low-grade gums in syrup solutions could not be whipped to contain more than 10 to 15 per cent overrun, whereas gum tragacanth under similar conditions permitted the incorporation of more than 50 per cent overrun.

**FORMATION OF HARD SPOTS ON THE SURFACE OF WATER ICES AND SHERBETS**

Throughout most of this investigation the excessive hardening of water ices and sherbets at the surface was never encountered because they were always kept covered in the hardening room. When water ices and sherbets were exposed to the air light-colored spots appeared, first barely visible to the unaided eye and later so numerous and large that the entire surface was covered. As the spots grew in size eccentric rings were formed and the spots were slightly elevated on the surface of the water ices or sherbets. The spots also grew into the water ices or sherbets to the extent of one-fourth to one-half inch. These spots were so hard that they resembled solid ice in their character. They could not be broken with the ordinary spoon or knife, but when they were completely removed the frozen product beneath them was in good condition. The extreme hardness of the surface of water ices or sherbets which have undergone this change has caused this material to be referred to occasionally as “petrified ice.”

It was usually necessary to start the formation of these spots in some artificial way or they would not appear on a can of water ice or sherbet the surface of which had been completely covered with parchment paper at the time of drawing from the ice cream freezer. These spots could be started by removal of the parchment paper to expose the water ices or sherbets to drying. The spots would appear in less than one day if the surface of the water ice or sherbet had been cut with a knife or spoon. After the surface had been cut in this way wrapping a cut surface with parchment paper would not seriously interfere with the hardening at the cut surface. From a practical standpoint these observations would show that this difficulty of ex-
cessive hardness would usually develop after the water ices or sherbets were in the retailers cabinet or in partially filled cans and bricks in the manufacturers refrigerator.

Microscopic examination showed these hard spots to contain, in addition to ice crystals, numerous crystals which appeared to be much like those which were later obtained from super-saturated sucrose solutions at a temperature of −15 to −20°C. Pure ethyl alcohol was cooled in the hardening room to −17.7°C., or thereabouts, and small quantities of the hard spots were placed in the alcohol. The water and ice mixed with the alcohol leaving behind the crystals of cane sugar. It was then learned that the hard spots could always be started by placing a crystal of cane sugar upon the surface of the water ices or sherbets, but they could not be produced by an ice crystal. Chemical analyses of some of the hardened areas gave a total sugar content of 38.29 per cent when only 33 per cent sugar had been used in the water ice upon which the hardening had taken place.

The evidence clearly showed that the highly concentrated sugar solutions which have been previously demonstrated by the author (1925) in ice cream were not stable in water ices or sherbets. The sugar crystallized out increasing the freezing point of the solution which in turn permitted the formation of more ice. This process continued until all, or nearly all, of the sugar had become crystals and the water frozen into ice. Wherever this crystallization occurred the water ices or sherbets became excessively hard due to the absence of unfrozen syrup and the presence of ice and sugar crystals.

The first attempt to avoid this hardening was thru the use of protective colloids. It was learned that gelatin, gum tragacanth, or agar had no appreciable effect upon the crystallization of the sugar. The use of additional milk or ice cream mix in the sherbets likewise did not materially alter the rate of sugar crystallization. Ice cream mixes were prepared using plain cream containing 30 per cent fat, gelatin, and 30 per cent of cane sugar. Crystallization of the sugar began in less than one week after freezing which showed that protective colloids were of little consequence, whereas the relation of the lactose to sucrose was important.

Corn sugar was mixed in various proportions with cane sugar and frozen in solution as water ices. Corn sugar was selected in prefer-

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4The corn sugar used in these experiments was "refined cerelose" manufactured by the Corn Products Refining Co. of New York.
ence to other sugars for several reasons. It is now being used to replace a portion of the sucrose in some brands of ice cream so it is available to ice cream manufacturers at the present time. A sufficient quantity could be used without imparting an off flavor in the product. It contains about 91 to 92 per cent of dextrose, or 99.5 per cent on the dry basis according to Cathcart (1924), and it has about two-thirds the sweetening effect. The dextrose sugar is especially soluble, and is very apt to form supersaturated solutions rather than crystallize from the solution. When 20 to 25 per cent of sucrose was replaced by corn sugar the water ices or sherbets would not show crystallized sugar in less than seven to ten days no matter how they were treated. Even then this crystallization occurred to so small an extent that the slightly hardened spots could be readily dished up with a spoon and eaten. In many instances water ices were cut, seeded with sucrose, and exposed to the air in the hardening room for two weeks without any crystallization of sugars, but the surface did dry enough to become heavily coated with a thick syrup. For all practical purposes the substitution of 20 to 25 per cent of corn sugar for sucrose made a water ice or sherbet in which crystallization of sugars would seldom occur. The total percentage of sugars in the water ices or sherbets which gave most satisfactory results was 7 per cent of corn sugar and 25 per cent of cane or beet sugar. It is advantageous to hold the percentage of corn sugar as low as possible because of its reduced sweetening power and greater effect in lowering the freezing point.

An explanation of the combined effect of dextrose and sucrose in preventing crystallization of the sucrose was sought. The substitution of dextrose for a portion of the sucrose caused a slightly lower freezing point than if sucrose was used alone. The difference was only about 0.5°C. which alone could not explain the retarded sugar crystallization.

Cane sugar is dissolved in water to the extent of approximately 65 per cent when the water ices are held at −17.7° C. (0°F.). Aqueous solutions of sucrose in concentrations of 65 per cent at approximately −17.7° C. were uniformly crystallized by seeding with a single sucrose crystal in less than 12 hours. Crystallization occurred to such an extent within one day that the surface of the solution became as hard as ice, and in one case within one week the water had frozen to so great an extent that the glass container was cracked by the force of the expanding ice. Sucrose solutions of similar concentra-
Chart II.—The Relation of Maximum Solubility of Sucrose and of Sucrose and Dextrose in the Same Solution to Temperature and Freezing Point.
tions when held at the same temperature failed, without seeding, to form any crystals in two weeks.

Aqueous solutions containing 65 per cent sugars of which 16.2 per cent was dextrose and 48.8 per cent was sucrose failed to give any crystals after two weeks in the ice cream hardening room. One of these solutions was heavily seeded with dextrose and sucrose crystals, and it was shaken daily to induce crystallization without any success. Cooling this solution to as low a temperature as $-25^\circ$C. ($-13^\circ$F.) failed to induce sugar crystallization within a period of 12 hours.

The investigations of Jackson and Silsbee (1924), giving the solubility relationships of sucrose, dextrose, and invert sugars, served admirably to help explain the prevention of crystallization of sucrose by substitution of dextrose for part of the sucrose. These authors showed that, altho the presence of dextrose or invert sugar decreased the solubility of sucrose and vice versa, the total solubility of the two sugars in the same solution was much greater than the solubility of either sugar alone. Dextrose and invert sugar have nearly the same solubility relation to sucrose, so they might have been used interchangeably in this investigation. From the data presented by Jackson and Silsbee the three solubility lines for sucrose and dextrose in the same solution have been plotted and shown in Chart II. Data are available for temperatures ranging from 50$^\circ$ to 0$^\circ$C., but the solubilities have been extended in the chart to $-25^\circ$ C. to include all temperatures used in this investigation.

The author recognizes that this procedure may have introduced some error, but facilities for maintaining sugar solutions at constant temperatures to determine the solubilities below 0$^\circ$ C. were not available. It will be observed from Chart II that the maximum solubility of dextrose in saturated sucrose solutions decreased with decreased temperature until at approximately $-17.7^\circ$ C. (0$^\circ$F.) the solubility of dextrose was about 18 per cent. The maximum solubility of sucrose in saturated dextrose solutions increased with decreased temperature until at $-17.7^\circ$ C. the solubility of sucrose was about 50 per cent. The combined total maximum solubility of both dextrose and sucrose decreased with decreased temperature until at $-17.7^\circ$ C. this solubility was about 67 per cent. These figures show a ratio of 1 part of dextrose to 2.8 parts of sucrose to obtain maximum solubility at $-17.7^\circ$C. and they agree closely with those obtained in this investigation which showed that 1 part of dextrose to 3.0 or 3.5 parts of sucrose gave best results in water ices.
For the purpose of illustrating further the solubility and crystallization relationships the solubility of sucrose and the freezing point of sucrose solutions are also graphically represented in Chart II. The solubility of sucrose from 50 to 0°C. was obtained from the Landolt-Börnstein tables (1923), but the cryohydric point of a sucrose solution as given by these authors could not be used to give a point on the line below 0°C. because this point did not agree with the other solubility data or with the results of this investigation. Consequently, the data presented by Kremann and Eitel (1923) were used which gave the cryohydric point of a sucrose solution as one containing 62.5 per cent sucrose at $-14.5^\circ$C. This concentration and temperature is in harmony with the other solubility table of Landolt and Börnstein and the results of this investigation. The line in Chart II representing the freezing point of sucrose solutions was constructed from the data of Kremann and Eitel, and the last point in the line was taken from the data obtained in this investigation that a 65 per cent sucrose solution began freezing at $-17.7^\circ$C.

A study of Chart II shows that the line representing the freezing point of sucrose solutions crosses the line representing the solubility of sucrose at a temperature of $-14.5^\circ$C. and a concentration of 62.5 per cent. This means that if a water ice made with sucrose is cooled below this temperature sucrose will eventually crystallize and form a hard surface on the frozen product. Kremann and Eitel also considered the lowering of freezing point due to the citric acid content of water ices, but the concentration of acid they used was much greater than that of commercial water ices or sherbets in this country. They stated that sugar crystals in water ices would not appear at a temperature of $-17.0^\circ$C. or above, whereas the author had no difficulty in obtaining sucrose crystals in seven days when the refrigerator was held at this approximate temperature and the solution was seeded with sucrose. This discrepancy was probably due to the fact that Kremann and Eitel used excessive quantities of citric acid and in the present investigation 0.2 per cent acid was considered as sufficient for any commercial conditions. The line representing the freezing point of sucrose solutions crosses the line representing the maximum solubility of sucrose and dextrose in the same solution at a temperature of $-19.5^\circ$C. and a sugar concentration of 66.5 per cent. This temperature for pure dextrose-sucrose solutions is about 0.5° C. too high because, as previously shown, the substitution of dextrose in place of part of the sucrose as used in this investigation reduced the freezing
point to this extent. Hence this solution, in the absence of any citric acid, would form sugar crystals at a temperature below \(-20.0^\circ\text{C.}\) \((-4^\circ\text{F.})\). The presence of small amounts of citric acid in commercial water ices would alter the temperature of sugar crystallization much less than \(0.5^\circ\text{C.}\) and can be ignored.

In summarizing the results, it can be stated that the hardening of water ices or sherbets at the surface during storage in the refrigerator can be completely prevented or reduced to so small an amount that it will not be troublesome. The water ice should contain 1 part dextrose or invert sugar to 3.0 or 3.5 parts sucrose. In the manufacture of water ices 7 per cent of corn sugar and 25 per cent of cane or beet sugar gave excellent results, although a slightly greater proportion of corn sugar can be used. When the frozen product was stored in an ice cream hardening room held at approximately \(-17.7^\circ\text{C.}\) \((0^\circ\text{F.})\), no sugar crystallization occurred, but if the temperature was maintained at \(-20.5^\circ\text{C.}\) to \(-23.3^\circ\text{C.}\) \((-5\text{ to } -10^\circ\text{F.})\), a small amount of crystallization was sometimes detected after the cut surface of a water ice had been exposed to the air from seven to ten days.

**FORMULAE FOR WATER ICES AND SHERBETS**

The results of this investigation which are of special practical importance are brought together in recommended formulae for water ices and sherbets so that they may be readily available. The formulae give only the basis for the mixture and do not attempt to specify flavors and fruit juices to give the water ices or sherbets their characteristic flavors. The figures are given on the basis of 100 pounds of mix which is about 10½ gallons. The mix has a specific gravity of approximately 1.14 at 10°C. and weighs 9.5 pounds per gallon. The specific gravity varies greatly, depending largely upon the percentage of sugar and the temperature.

**WATER ICE**

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Description</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cane sugar</td>
<td>..........................</td>
<td>25.0 pounds</td>
</tr>
<tr>
<td>Corn sugar</td>
<td>.........................</td>
<td>7.0 pounds</td>
</tr>
<tr>
<td>Agar</td>
<td>.........................</td>
<td>0.2 pound (3.2 ounces or 90.6 grams)</td>
</tr>
<tr>
<td>Gum tragacanth or high-grade</td>
<td>India gum</td>
<td>0.4 pound (6.4 ounces or 181.2 grams)</td>
</tr>
<tr>
<td>Water, fruit, fruit acid, flavor, and color</td>
<td>..........</td>
<td>67.4 pounds</td>
</tr>
<tr>
<td>Overrun 20 to 25 per cent—Total yield 13 gallons</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
SHERBET USING MILK

Cane sugar ........................................ 25.0 pounds
Corn sugar ........................................ 7.0 pounds
Agar ................................................. 0.2 pound (3.2 ounces or 90.6 grams)
Gum tragacanth or high-grade india gum 0.2 pound (3.2 ounces or 90.6 grams)
Whole milk .......................................... 50.0 pounds
Water, fruit, fruit acid, flavor, and color 17.6 pounds
Overrun 25 to 30 per cent—Total yield 13.5 gallons.

SHERBET USING ICE CREAM MIX

Cane sugar ........................................ 25.0 pounds
Corn sugar ........................................ 7.0 pounds
Agar ................................................. 0.2 pound (3.2 ounces or 90.6 grams)
Gum tragacanth or high-grade india gum 0.2 pound (3.2 ounces or 90.6 grams)
Ice cream mix, without sugar or gelatin. 10.0 pounds
Water, fruit, fruit acid, flavor, and color. 57.6 pounds
Overrun—25 to 30 per cent—Total yield 13.5 gallons.

The mixture should be prepared by first weighing most of the water or all of the milk, if any is used, leaving out enough water to dissolve the agar and to allow for fruit juices, etc. The sugars should be thoroly mixed with the powdered gum tragacanth or high-grade india gum and slowly poured into the water while the water is being agitated rapidly. Powdered agar is preferable to granular or shreds because it can be more readily dissolved. The powdered agar should be poured into 50 times its weight of boiling water while the water is being agitated rapidly. The water with agar should continue to boil for about five minutes when the agar will be completely dissolved. The hot agar solution should be added to the mix as if it were a hot gelatin solution. The gelatinization strength of agar is reduced by boiling in acid solutions, but it is only slowly altered by boiling in water, so it is important that fruit acid should be added to the mix after the agar. All other ingredients used should be added to the mix at this time and the total weight brought up to the required amount with water, making allowance for the fruit and fruit acids or juices which are usually added at the freezer.

There is no necessity of aging water ices or sherbets made with agar and gum as stabilizers because the action of each takes place within a few minutes. Evidence of a weak gel formation should be readily observed at once if sufficient agar has been used, since agar solutions set at 40° to 42° C. and since the temperature of the cold mixes is much lower.
Ices and sherbets should be frozen to the same stiffness as ice cream in the freezer which will give the product when drawn from the freezer a temperature of $-4^\circ$ to $-5^\circ$ C. (24.8 to 23°F.). If the product is drawn from the freezer at too warm a temperature there is a tendency toward crumbliness, coarseness, and the syrup is more apt to settle out in the hardening room. The overrun which should be obtained on each product is given in the formulae, altho it may be stated that during much of this work the author favored a product with 5 to 10 per cent additional overrun. The higher overrun causes the product to be slightly softer than average ice cream. It is desirable to reduce the sugar content to 28 or 30 per cent in water ices with the higher overrun to harden the product, even tho the flavor is slightly injured by the decrease in the sugar content. The overrun can be controlled by the amount of mix placed in the freezer, the hardness to which the product is frozen, the length of time it is whipped, and the amount of gum tragacanth, milk, or ice cream mix used in the formula. Sherbets containing milk whip easily and the overrun can be reduced by using a lower-grade gum or less of the high-grade gum. Difficult whipping of water ices or sherbets containing ice cream mix can be improved by using a better grade of india gum or gum tragacanth or by increasing the quantity of high-grade gum used to as much as 0.5 per cent. A few trials may be necessary to so adjust the ingredients that a proper overrun is obtained.

The question will naturally be raised concerning the probable adoption by the ice cream industry of the mixture of sugars and stabilizers recommended as a result of this investigation. There can be no question concerning the superiority of the body, texture, and keeping quality of water ices or sherbets made according to the new method. The adoption of these practices will depend upon several factors. First it will be necessary for the manufacturers of the products concerned to study the problem and present the facts to the ice cream industry. Secondly, the ice cream manufacturers who now have a large water ice or sherbet trade will then be able to try the methods, with the help of interested manufacturers of agar, gums, and corn or invert sugar, and adapt them to their factory conditions. The consuming public is buying more water ices and sherbets than ever before and an improved product should be made to stimulate this interest of the public.

Not only should the body, texture, and keeping quality be improved, but the flavor can and is being improved by the more ex-
tensive use of pure fruits for both flavor and acid. When the first orange water ices and sherbets were prepared in this investigation they were flavored with artificial orange and given the proper acid taste with citric acid. Few people cared to eat them. The desirability of the product as a food pleasing to the appetite was completely changed by the use of fruit. Fresh oranges and lemons were used to the extent of 2 dozen oranges and 2 dozen lemons for each 100 pounds of finished product. The heavy-skinned naval oranges were selected and the outer surface of the peel was grated off to serve as the principal source of orange flavor. The grated peel was stirred directly into the water ices but the coarsest particles of peel were later strained out by a coarse metal strainer. The juice of all the lemons and oranges was used and 0.1 per cent of dry citric acid crystals (6 ounces of a 25 per cent acid solution for each 100 pounds of finished product) added. No other flavor was added. The fresh fruit almost doubled the cost of the ingredients to make the water ice or sherbet, but the finished product was well worth the extra cost.

CONCLUSIONS

The hardness of water ices and sherbets was influenced to the largest extent by the percentage of air which they contained and to a lesser extent by the percentage of sugar. Variable overruns affected the hardness to the greatest extent at relatively warm storage temperatures. The effect of air was physical. Water ices or sherbets at any given temperature contained unfrozen syrups with the same concentration of sugar. Hence, increased percentages of sugar increased the quantity of unfrozen syrup and made the product softer. A hardness of water ices and sherbets comparable to that of ice cream at the same temperature was obtained with 20 to 30 per cent overrun and 30 to 33 per cent sugar. Any increase in the overrun above these limits should be accompanied by a decrease in the percentage of sugar unless a comparatively soft product is desired.

The unfrozen syrup in water ices or sherbets drained out during storage unless some stabilizing material was present to prevent it. Gums and gelatin have been used commercially for this purpose. India gum or gum tragacanth prevented most of the syrup from settling out of the water ices or sherbets during storage, but a product made with these gums as the only stabilizer possessed a crumbly body and became excessively hard throughout as a result of alternate
hardening and softening. A low grade of india gum produced a low overrun, but a high grade of india gum or gum tragacanth permitted a high overrun. Obviously, the overrun could be controlled by mixtures of gums.

When sufficient gelatin alone was used to form a jelly in the water ices or sherbets, it was difficult to obtain an overrun low enough to give proper hardness to the product. The gelatin held the syrup in place, gave a smooth enough texture, prevented crumbliness, and made a product that would withstand heat shocks very well. A low grade of india gum could not be used to reduce the overrun because the gelatin and gum in the same solution precipitated each other. It is not advisable, therefore, to use gelatin and gum together as stabilizers in the same water ice or sherbet. A high grade of india gum or gum tragacanth was used with gelatin without precipitation, but such a combination only increased the overrun.

All of the benefits of gelatin were secured thru the use of agar in concentration of 0.2 per cent which was sufficient to form traces of jelly at 4.4°C. (40°F.). The water ices and sherbets containing agar alone gave very low overruns. The addition of milk solids, gelatin, gum tragacanth, or a high grade of india gum permitted the incorporation of a sufficient quantity of air. The gums slightly improved the body as well as increased the overrun; so their use was especially advantageous in water ices. Agar was not precipitated by any of the gums.

The excessive hardening of water ices and sherbets at their exposed surfaces during storage in the hardening room or retailer's cabinet was caused by crystallization of sucrose from a supersaturated solution. When water ices or sherbets were cooled to −14.5°C. (+6.0°F.), the unfrozen water contained 62.5 per cent sucrose and was saturated in respect to sugar. Below −14.5°C. the syrup was supersaturated in respect to sugar and crystallization could be started by exposure to air, shocking the solution by cutting the water ice or sherbet with a knife or spoon, or by seeding with sucrose crystals. Since ice cream refrigerators are below this temperature crystallization may occur in commercial water ices or sherbets at any time. As crystallization of the sucrose took place more water froze into ice due to the increased freezing point of the solution and the water ice or sherbet became exceedingly hard. Dextrose and sucrose have a greater combined solubility than either alone, so it was possible to substitute a portion of the cane or beet sugar with corn sugar, thereby increasing the
total solubility to such an extent that supersaturation did not occur above −20° C. (−4.0°F.). This temperature is sufficiently low to permit the storage of water ices or sherbets made with both sugars in ice cream refrigerators with little or no crystallization of either sugar.

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