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ALPHA HYDRATE AND BETA ANHYDRIDE LACTOSE CRYSTALS IN SANDY ICE CREAM

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ALPHA HYDRATE AND BETA ANHYDRIDE LACTOSE CRYSTALS IN SANDY ICE CREAM

C. W. DECKER,* W. S. ARBUCKLE AND W. H. E. REID

Sandiness in ice cream is a problem which has warranted much study. The factors causing sandiness, methods of preventing this defect, and the physico-chemical properties of lactose have been studied by several investigators.

These investigators have confined their studies primarily to commercially prepared lactose. The problem of isolating and identifying the particular crystals causing sandiness in ice cream is one which requires further investigation.

Several workers (1),† (2), (3), (4) have reported that the alpha hydrate form of lactose is responsible for sandiness in ice cream. The optical properties have not been determined directly on the sandy material as isolated from ice cream, and the possibility exists of the beta anhydride form of lactose being present although its presence has never been reported.

The petrographic microscope proved to be an excellent instrument for study of the sandy material in ice cream. A method for extracting the crystalline substances causing sandiness in ice cream was devised by modifying the technique of several other workers. This material in a purified form was then examined under the petrographic microscope and the optical data obtained. Several other studies were made on the crystalline material including chemical tests and polarimeter determinations. Commercial samples of ice cream were studied microscopically for sandiness and photomicrographs made of sections of sandy ice cream.

Since the possibility exists that both alpha hydrate lactose and beta anhydride lactose may be present as sandy material in ice cream, the

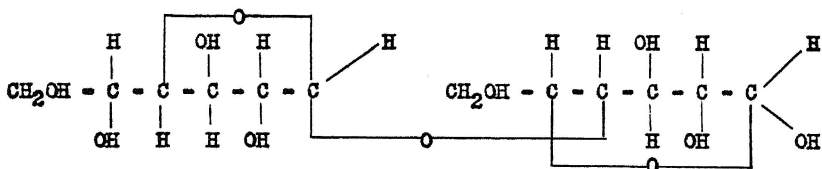
*The data presented in this bulletin were taken from a paper submitted by the senior author in partial fulfillment of the requirements for the degree of Master of Arts in the Graduate School of the University of Missouri, 1939.

†See List of References on pages 21 and 22.

difference in the properties of the two forms are shown by their structural formulas.

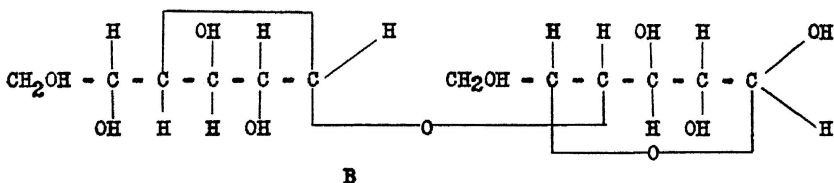
ALPHA LACTOSE

A - glucose - B - Galactoside



BETA LACTOSE

B - glucose - B - galactoside



The difference structurally between the two forms is merely an interchange of the "H" and "OH" on the last carbon atom from the alpha to the beta position.

REVIEW OF LITERATURE

Bothel (1) was one of the first to point out this defect as being due to the presence of lactose crystals. He stated that sandiness is the defect in ice cream which may be detected in the mouth as though sand were present.

Zoller and Williams (2) first isolated sandy material from ice cream and identified it chemically and crystallographically as alpha lactose.

Troy and Sharp (3) explained the formation of "sandy" ice cream as being due to the presence of sufficient non-frozen water to permit the alpha hydrate lactose to crystallize. By rapidly freezing out the water from ice cream, the lactose solution may be concentrated to a region where the crystallization of lactose does not occur (super-saturation). Alpha lactose crystals may form in such ice cream if sufficient ice is melted to dilute the syrup to a range of concentration where crystallization can take place.

Lactose crystals have been shown by Hormberger and Cole (4) to be from 16 to 30 microns in size when they can be detected as sandiness in the mouth.

Causes of Sandiness in Ice Cream and Methods of Preventing It

Temperature fluctuations or "heat shocking" have been shown by several investigators (4), (5), (6), (7), (8), (9) to be an important factor in promoting sandiness. Temperature fluctuations may take place at any time after the ice cream is placed in the hardening room and until it reaches the consumer.

Nut meats have been shown by Reid (6) and Dahle (7) to aid lactose crystal formation and induce sandiness. Reid demonstrated that by washing the nut meats in warm water and autoclaving them at steam pressure or soaking them in gelatin solution previous to their use in ice cream, that sandiness could be delayed. Dahle used a syrup solution in which to soak the nut meats and delay sandiness.

Whitaker (8) shows that moisture is taken up by the nut meats and advances the assumption that unsoaked nut meats may take up water in the ice cream thus decreasing the unfrozen part of the ice cream and consequently increasing the lactose concentration and tendency to crystallize. He also found that: (a) rapid hardening delays crystallization, (b) hand packed ice cream developed crystals more rapidly than factory filled packages, (c) mechanical shock is not an important factor in producing sandiness, (d) the presence of inert nuclei was an important factor in promoting sandiness, (e) lactose crystallization was materially retarded by pasteurizing at 175-180 degrees Fahrenheit, and (f) the longer the ice cream was in the freezer the sooner lactose crystallized.

Hormberger and Cole (4) report that lactose crystallization was not prevented by the presence of sucrose, dextrose, or gelatin or by combinations of these substances within the limit of concentrations normally used in ice cream.

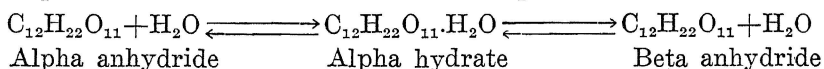
Sommer (26) gives a review of serum solids content permissible in ice cream mixes if sandiness is to be avoided. Dahle (5) reports the serum solids content permissible in ice cream mixes.

Several investigators (4), (10), (11) have advocated the use of low-lactose products as a source of serum solids in the ice cream mix as a means of preventing sandiness.

Physico-Chemical Properties of Lactose

Troy and Sharp (3) report that Erdmann in 1855 called attention to another form of lactose which we now know as the beta anhydride form.

Later studies by Hudson (12), (13) showed the third form to be a mixture of alpha hydrate and beta anhydride lactose. He advanced the following equation to show the dynamic equilibrium reaction and to explain the mutarotation between the Alpha and Beta form:



Hudson (14) found that alpha-lactose was less soluble than beta-lactose. He reported a solubility of 5.0 parts per 100 parts water at 0 degrees Centigrade for beta-lactose. As the alpha-lactose crystallizes out as a hydrate containing one molecule of water, some of the beta-lactose must change to alpha to restore this ratio.

Herrington (15) studied the vapor tension of alpha hydrate-anhydride systems of lactose. Crystal size was of little importance in determining the rate at which moisture is taken up by the alpha anhydride lactose. He also found that some reaction other than loss of water of hydration must occur on continued heating of lactose.

Factors Affecting Natural Constants of Lactose

Herrington (16) showed that salts and acids had an important influence upon the mutarotation velocity of lactose. This phenomenon is attributed to general acid and base catalysis. The catalytic effects of other salts was not studied but were thought to be of importance only in such concentrated products as milk powder and ice cream.

Herrington (17), in a further study of the influence of other substances upon the equilibrium rotation of lactose, found that in glycerol solutions the equilibrium mixture of the high and low rotating forms of lactose contains more of the high rotating component than is found in aqueous solutions, and that the specific rotation of lactose is increased in glycerol solutions.

Herrington (18) showed that many salts may combine with lactose. The existence of such molecular compounds is possibly a factor contributing to the stability of such supersaturated solutions of lactose as are found in ice cream and in milk powders. Lactose is more soluble in molar solutions of calcium chloride or of calcium nitrate than in pure water and an increase in the concentration of salt brings further increase in the solubility of lactose.

Hunziker and Nissen (19) found that the presence of sucrose diminished the solubility of lactose. This decrease is slight in dilute sucrose solutions as are present in ice cream mixes, but becomes greater as the sucrose concentration increases, as in sweetened condensed milk, amounting to approximately 15 per cent of the lactose

solubility in aqueous solutions. They also stated that the colloids do not have any material influence on the solubility of lactose.

Factors Influencing the Change From Alpha to Beta-Lactose

Troy and Sharp (3) state that between pH values of 2.0 and 7.0, the rate of change of alpha-lactose into the beta-lactose is at a minimum. The rate approached infinity at pH 0.0 and 9.0. The effect of pH on the rate of change of alpha to beta-lactose was shown to influence the rate of solution of alpha-lactose and the effect of pH on the rate of change of beta to alpha-lactose was shown to influence the rate of precipitation of alpha-lactose. They found relatively concentrated sucrose solutions to have little effect on the rate of change of the two forms of lactose into each other.

Whitaker (8) states that rapid hardening delays crystallization, and a rapid attainment of low temperatures is desired as the rate of conversion of beta to alpha is decreased under such conditions.

Factors Affecting the Crystalline Habit of Alpha-Lactose

Hunziker and Nissen (20) describe the change in lactose crystal shapes in the presence of 14 per cent sucrose solution to be slight. The crystals are shorter and thicker but possess full development. The crystals resemble short truncated pyramids with flat rhomboid base and apex. Crystallographically the crystals are the same but the crystal habit appears to be altered.

Hormberger and Cole (4) report that the rate of crystal growth was found to be an essential factor in the development of sandiness, and was influenced by lactose concentration, storage temperatures, fluctuations in storage temperatures, and methods of freezing.

Herrington (21) found that the crystallizing habit of alpha hydrate varied greatly under different conditions of crystallization. The precipitation pressure and the ratio of the actual concentration to the solubility, was the principal factor governing the crystalline habit of lactose. He states that sucrose has a precipitating effect upon lactose and influences its crystallization. He reports that both alpha hydrate and beta anhydride will form needles if crystallization is sufficiently rapid, but may be distinguished by the fact that the alpha hydrate prisms are always straight while those of beta anhydride are curved. Under favorable conditions he was able to grow lactose crystals with 13 faces and as high as 16 although crystals with more than 13 faces are extremely rare.

Crystallographic and Optical Properties of Alpha- and Beta-Lactose

Groth (22) gives the data on alpha hydrate lactose to be: monoclinic-sphenoidal; cleavage in three directions nearly at right angles. He also includes the refractive indices.

Zoller and Williams (23) identified sandy material in ice cream as alpha-lactose by making photomicrographs utilizing a petrographic microscope. They found the typical tomahawk-shaped alpha hydrate lactose crystal to be present.

Wherry (24) determined the crystallographic features and optical data of alpha- and beta-lactose, and sucrose. He found the following values:

Substance	A (alpha)	B (beta)	Γ (gamma)	2E
a—lactose	1.517	1.553	1.555	33½°
b—lactose	1.542	1.572	1.585	120°
sucrose	1.540	1.567	1.572	79°

He states that beta lactose crystallographically belongs to the holoaxial-polar (sphenoidal) class of the monoclinic system.

Hunziker and Nissen (20) give the crystallographic features of alpha-lactose. Lactose crystals belong to class C_2 and are monoclinic sphenoidal and have only one axis of symmetry. They have trapezoidal side faces and rhombic tops and bottoms. The full developed lactose crystal has in addition beveled faces at the base which may terminate in a sharp edge giving the crystal a distinct tomahawk appearance.

Williams and Peter (25) report having found a new diamond-shaped lactose crystal which they identified as alpha-lactose. They give the cause of its formation, the high viscosity of the solution, due in particular to the concentration of the sucrose in the crystallizing medium, which in turn is caused by a freezing out of part of the water.

PROCEDURE

A high serum solids ice cream was made containing 15.5 per cent serum solids, 13 per cent fat, 14 per cent sugar, and 0.30 per cent gelatin, and frozen in a Vogt continuous freezer.

The ice cream was then divided into two groups. The first group was placed in the hardening room at -10 degrees Fahrenheit immediately after being frozen, and held at this storage temperature for twelve months at the conclusion of which time the ice cream was considered only slightly sandy.

The second group was placed in a dispensing cabinet at 6 and 10 degrees Fahrenheit respectively for three months, and subsequently in the hardening room at -10 degrees Fahrenheit where pronounced sandiness developed in five months.

Samples of the second group were used as the source of sandy material for this investigation. Heat shocking the samples by exposing them at room temperature for twenty minutes every second day for ten days aided in promoting sandiness.

Isolation of Sandy Crystals from Ice Cream

Using a modification of the method of Zoller and Williams (2), the ice cream was allowed to melt at a temperature of 72 degrees Fahrenheit and was then placed in 50 cubic centimeter centrifuge tubes which were centrifuged at 2500 revolutions per minute for 15 minutes. The sandy material was deposited at the base of the tube beneath a separate layer of liquid and fat. The supernatant liquid was decanted off leaving the sandy material at the base of the tube.

A saturated solution of lactose was prepared at a temperature of 72 degrees Fahrenheit. About 35 cubic centimeters of this saturated lactose solution was used to wash the sandy crystals, and the tubes were again centrifuged. This procedure was repeated with ether and fifty per cent acetone.

The sandy crystals were then sufficiently pure to be used in making chemical tests and to study with the petrographic microscope.

Chemical Tests

Chemical tests were made on the sandy material to determine the particular sugar or sugars responsible for sandiness in ice cream.

Fehlings solution was used to determine the reducing properties of the sandy material. The addition of several cubic centimeters of Fehlings solutions to the solution of the sandy material in a test tube produced a yellow precipitate and upon further heating changed to a red precipitate which indicated the presence of a reducing sugar.

Seliwanoff's reaction was used to determine the presence of a keto-hexose sugar. The addition of Seliwanoff's reagent to the sandy material gave a positive reaction for this test indicating that the sandy material was a keto-hexose sugar.

The Osazone tests were used in making a positive identification of the sugar. Yellow crystalline bodies called osazones are formed from certain sugars and in general each individual sugar gives rise to an osazone of a definite crystal form, which is typical for that sugar.

The Use of the Petrographic Microscope in Obtaining Optical Data

Differentiation of the two forms of lactose can be made with a petrographic microscope since their structural formula, optical and crystallographic properties differ. The petrographic microscope utilizes polarized light and may be used to identify crystals through optical means. The refractive indices were obtained by using the oil immersion method. Oils with a different refractive index were graduated to cover a definite range. A slide was prepared by immersing the powdered crystalline substance to be studied in the drop of oil of known refractive index and covering it with a cover slip. When using the petrographic microscope a white line called the Becke line outlines the crystal. As the focus is raised the line moves toward the substance having the higher index. A succession of oils may be necessary before a match is obtained. As the crystal and the oil approach a match the crystal fades out and at a match the white line may split into a yellow and a blue line, one moving toward the oil and the other toward the mineral. Estimations are made of the third place value.

By utilizing polarized light, optical data were obtained. Between crossed nicols the optical angle ($2V$), interference figures, interference colors, dispersion of the indices, optical character and crystal form were determined.

Positive identification of the crystal material was acquired by utilizing the complete optical data.

Examination of Sandy Ice Creams

Several sandy samples of ice cream were obtained and examined. The sandy material was isolated by the method (2) previously described and examined with the petrographic microscope. Comparisons were made of the sandy material from these samples with the sandy material of the original high serum solids ice cream from which the chemical and optical data were obtained. Methods of manufacture, composition of the mix, temperature and length of time of holding, subjection to heat shocking varied with the several samples. Consequently, the type and rate of crystal formation differed.

Photomicrographs of Sections of Ice Creams

Sections of ice creams showing sandiness were prepared with a microtone in a hardening room at a temperature of -14 degrees Fahrenheit and photomicrographs obtained.

EXPERIMENTAL DATA

When the Osazone test was applied to the isolated sandy material, lactosazones, Figure 1, were formed showing that the sandy material consisted of lactose and that sucrose was not present.

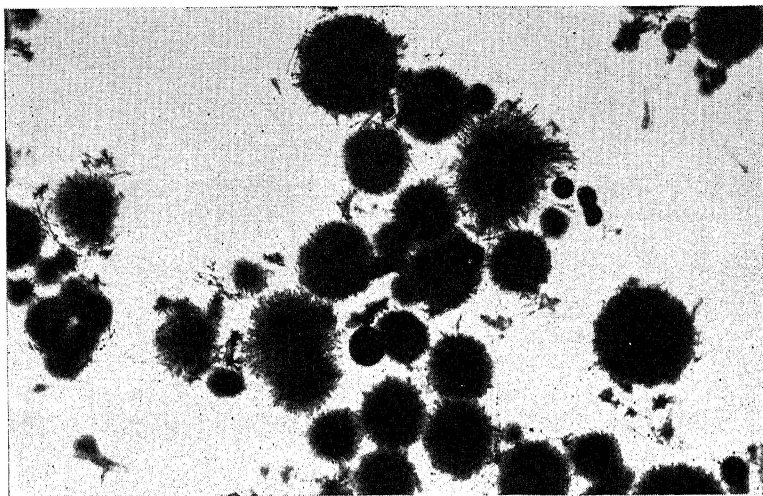


Fig. 1.—Lactosazones from Sandy Ice Cream Material (Magnification 100x)

Optical Differentiation of Alpha and Beta Lactose

Table 1 presents optical data on alpha- and beta-lactose present in sandy material isolated from a high serum solids ice cream. The alpha-lactose was designated as the tomahawk type of crystal, and beta-lactose as the columnar type. Comparisons of the tomahawk type and the columnar type of crystals were made with the optical data obtained by Wherry (24) for alpha- and beta-lactose.

TABLE 1.—OPTICAL PROPERTIES OF ALPHA LACTOSE AND BETA LACTOSE*

Name of Substance	Refractive Indices	Optical Character	Optical Angle 2E	Crystal Form	Dispersion	Interference Color	Source of Optical Data
Alpha lactose	a-1.517 b-1.553 g-1.555	negative	33½°	Mono clinic	Violet Red	1st to 2nd order	Wherry
Tomahawk type crystal	a-1.518 b-1.553 g-1.556	negative	34°	Mono clinic	Violet Red	1st to 3rd order	
Beta lactose	a-1.542 b-1.572 g-1.585	negative	120°	Mono clinic	Violet Red	1st to 3rd order	Wherry
Columnar type	a-1.542 b-1.572 g-1.582	negative	...	Mono clinic	Violet Red	1st to 3rd order	

*It is interesting to note here that several references (2), (22), (26) on the optical properties of alpha lactose give the refractive index value beta (B)=1.542 while Wherry (24) gives B=1.553. The value we found for the columnar crystal agrees with that of Wherry.

Figure 2 shows the alpha hydrate form of lactose, ($C_{12}H_{24}O_{12}$) or ($C_{12}H_{22}O_{11} \cdot H_2O$), as a tomahawk or wedge-shaped crystal. This is a typical crystal shape of the alpha form although slightly modified forms may be produced depending upon the conditions under which crystallization occurs. The dark bands are interference colors produced between crossed nicols.

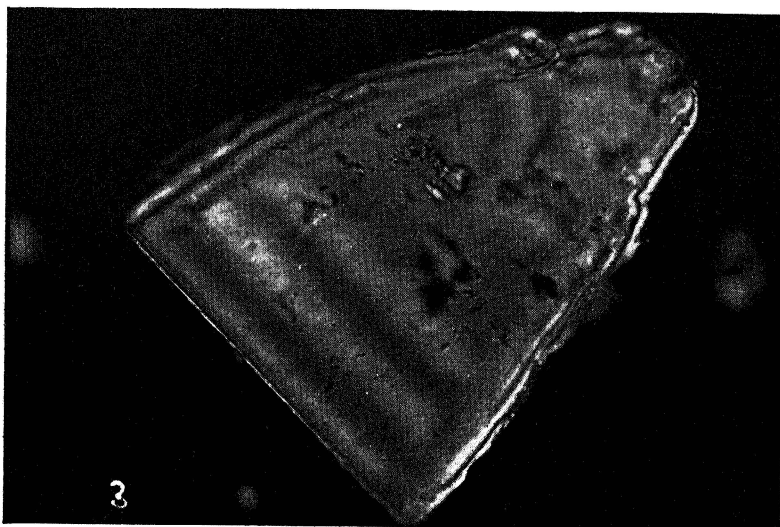
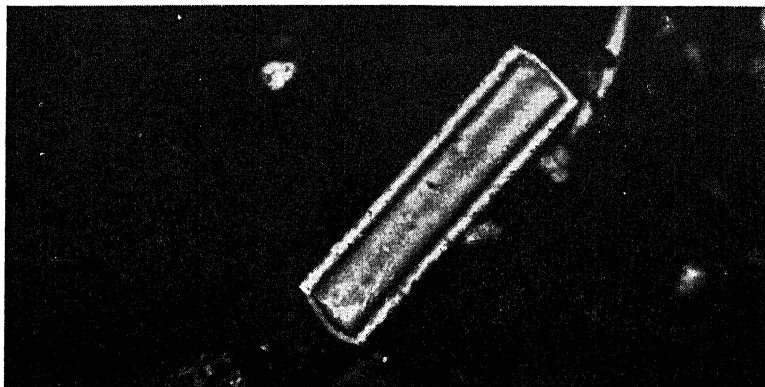


Fig. 2.—Alpha Lactose (Tomahawk Type) Sand Crystals (Magnification 450x)

Form	Monoclinic	Optical character	negative
Refractive Indices	$a=1.518$	Optical angle.....	$2E \dots 34^\circ$
	$b=1.553$	Dispersion.....	violet red
	$c=1.556$	Interference colors.....	1st to 3rd order

Figure 3 shows the beta anhydride form of lactose as it appears in the sandy ice cream material. It is a long columnar type of crystal which distinguishes it from the wedge-shaped alpha type. The columnar type is an anhydride ($C_{12}H_{22}O_{11}$) and has a higher index of



(Upper Section, Magnification 450x)



(Lower Section, Magnification 200x)

Fig. 3.—Beta Lactose (Columnar Type) Sand Crystals

Form	Monoclinic	Optical character.....	Negative
Refractive Indices	a=1.542	Optical angle.....	2E.....34°
	b=1.572	Dispersion.....	violet red
	c=1.582	Interference colors.....	1st to 3rd order

refraction than the alpha type. The cleavage is at right angles in some of the better types of crystals. A variation in the size of the crystals may be observed.

Figure 4 shows the relative proportion of alpha to beta lactose crystals isolated from sandy material in its original state. The large irregular shaped crystals are alpha lactose and are comprised of masses of smaller crystals.

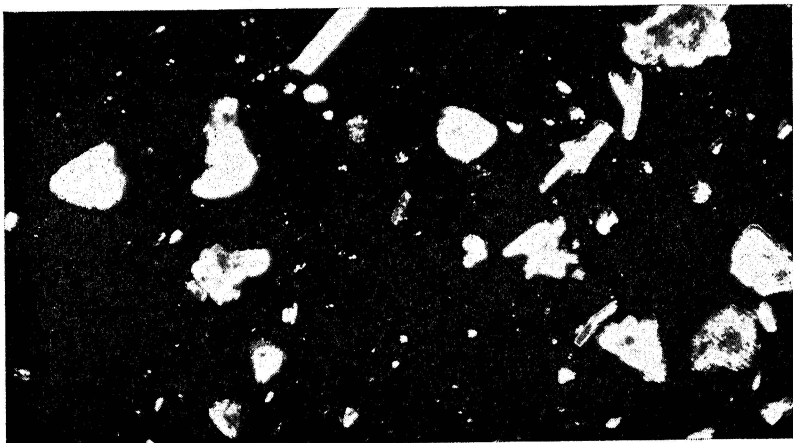
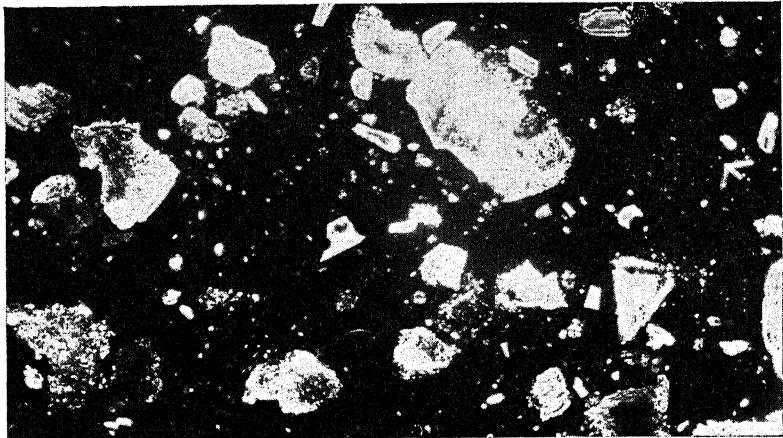


Fig. 4.—Relative Proportion of Alpha and Beta Lactose in Sandy Material (Magnification 100x)

Figure 5 illustrates the variations in size of beta lactose crystals. These photomicrographs were taken of microscopic fields in which the beta form of crystal predominates.

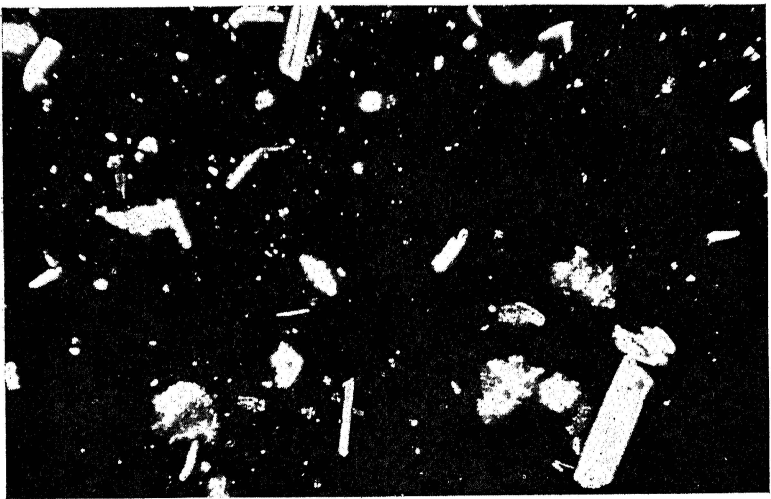
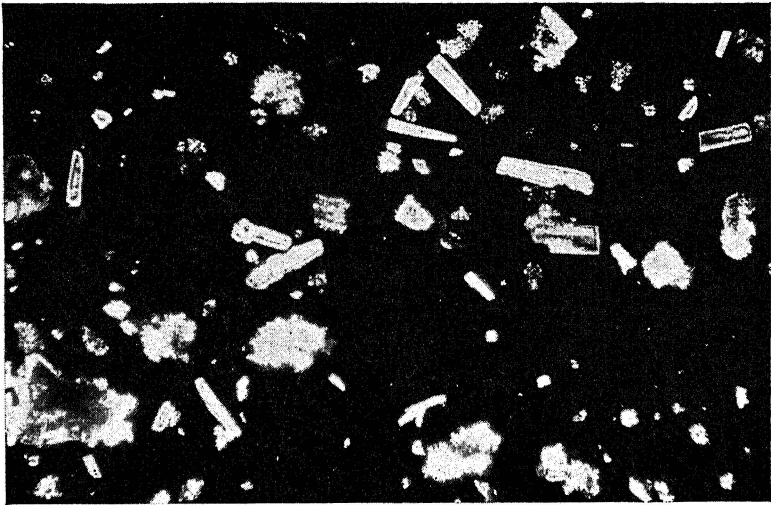


Fig. 5.—Beta Lactose Showing Variation in Size of Crystals (Magnification 100x)

Figure 6 shows an alpha and beta lactose crystal. These crystals are not attached physically. However, the contrast between the two different types of crystals can readily be observed. The edges of the alpha lactose crystals are slightly irregular due to the action of centrifuging and washing, whereas commercially prepared lactose and lactose recrystallized from aqueous solutions have smoother crystal faces and edges.

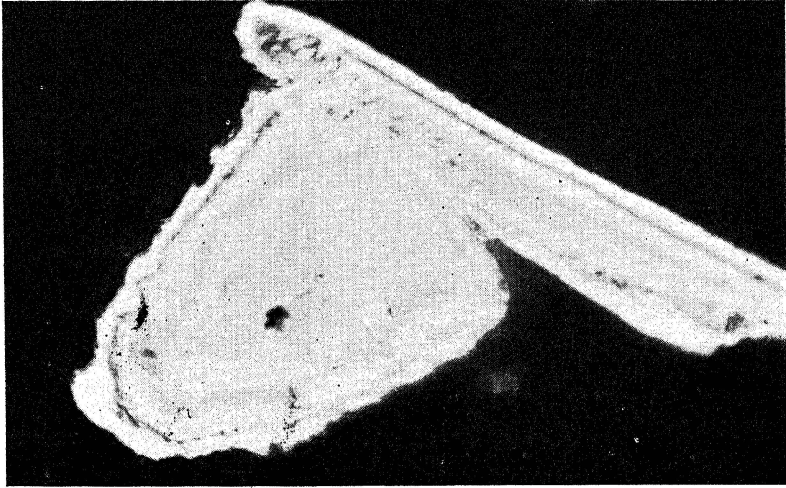


Fig. 6.—Alpha and Beta Lactose Sand Crystals (Magnification 450x)

Figure 7 shows commercially prepared lactose. The tomahawk form of crystal is predominant and the crystal faces and edges are clearly outlined. There is a greater uniformity in the size of the crystals than in the lactose present in the sandy material. There are no beta anhydride lactose crystals present. The more perfect

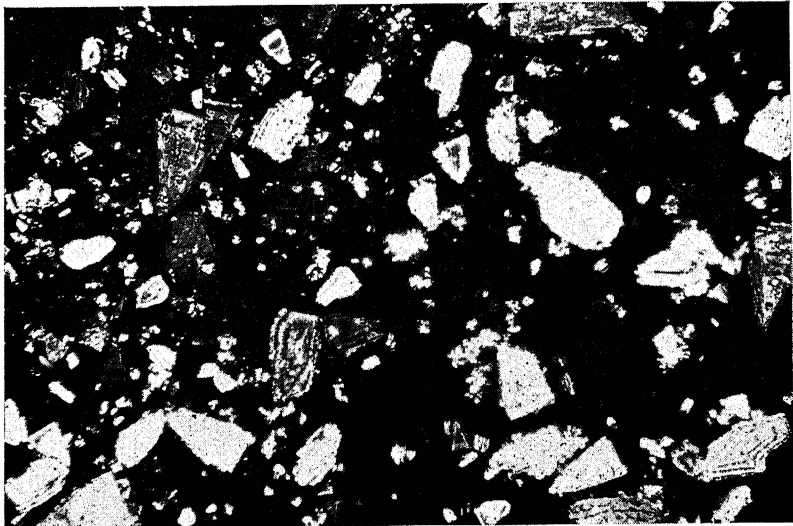


Fig. 7.—Commercially Prepared Lactose Showing Alpha Form (Magnification 100x)

crystal form of the commercially prepared lactose compared with the lactose of the sandy material removed from ice cream shows the effect of the various factors such as the concentration of the various ingredients in the ice cream mix, heat shocking, and the method of isolation upon the crystal form of the sandy material.

TABLE 2.—COMMERCIAL ICE CREAMS OF DIFFERENT FLAVORS SHOWING PRESENCE OF ALPHA AND BETA LACTOSE CRYSTALS

Sample No.	Flavor	Type of Freezer	Serum Solids per cent	Age of Ice Cream Weeks	Alpha Lactose	Beta Lactose
1	Vanilla	Vogt	12.00	12	+	+
2	Butterscotch	Batch	11.00	4	+	+
3	Black Walnut	Vogt	11.00	12	+	+
4	Nut	Batch	12.00	4	+	+

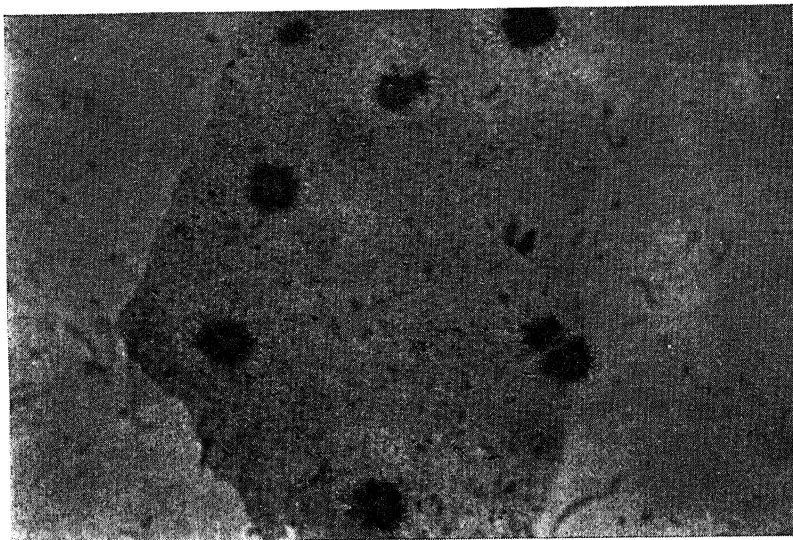


Fig. 8.—Alpha Lactose Crystals in the Process of Formation (Magnification 100x)

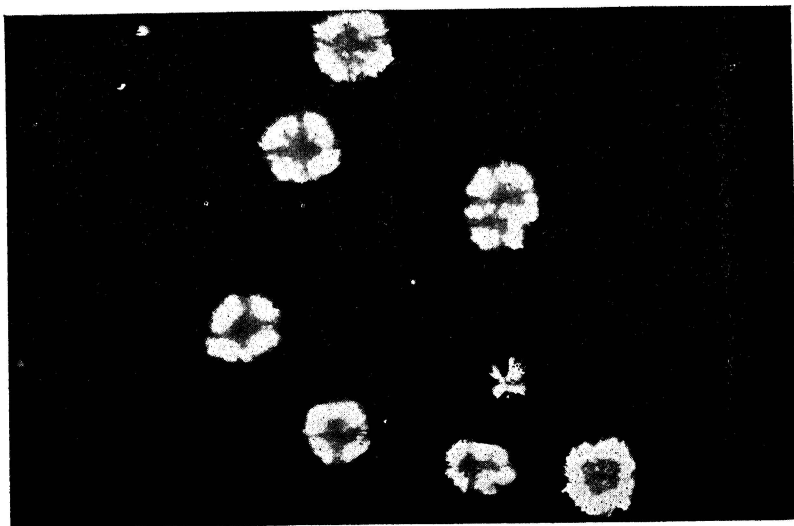
Table 2 shows the presence of both alpha and beta lactose in sandy ice cream obtained in the local market. Sample 1 showed typical lactose crystal formation of both types, while in samples 2, 3, and 4, the alpha lactose crystals were less numerous and in the process of formation. Sandiness had not developed as fully in the latter three samples as in sample 1. An average of three or four beta lactose crystals were present in each field.

Figure 8 shows alpha lactose crystals in the process of formation in samples 2, 3 and 4, Table 2. The grouping of the needle-like

crystals into a cluster in the formation of a larger crystal of alpha lactose is clearly shown.



Plane-Polarized Light (Magnification 100x)



Between Crossed Nicols (Magnification 100x)
Fig. 9.—Formation of Alpha Lactose Crystals on a Nut Particle.

Figure 9 shows the formation of alpha lactose crystals on a nut particle.

Figure 10 was taken at a temperature of -14 degrees Fahrenheit with an oil of 1.41 refractive index and shows the texture of sandy ice cream and the presence of an alpha lactose crystal as it occurs in

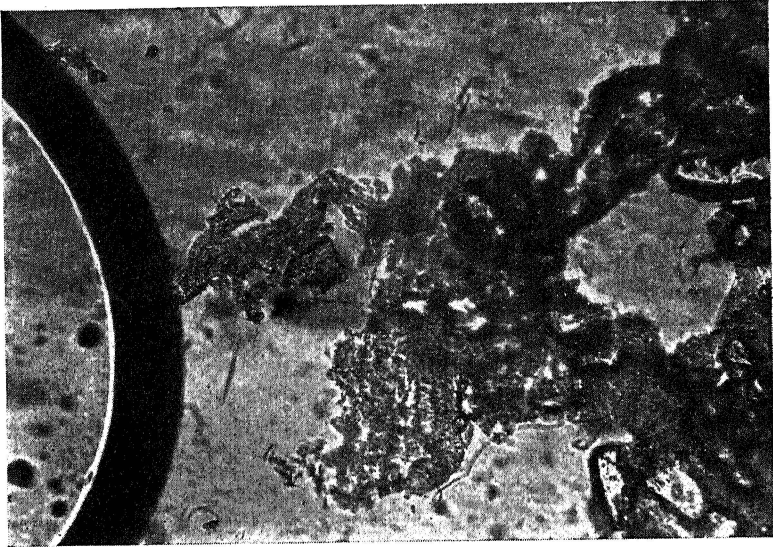


Fig. 10.—Presence of An Alpha Lactose Crystal in a Section of Sandy Ice Cream (Magnification 100x)



Fig. 11.—Presence of Beta Lactose in Sandy Ice Cream (Magnification 100x)

the ice cream. The texture of the ice cream shows the effect of heat shocking.

Figure 11 shows the presence of crystals of beta lactose which appear in the center of the photomicrograph. The effect of heat shocking on the texture of the ice cream can also be observed.

DISCUSSION

It is thought that the immediate factor causing the formation of sandiness in the samples of high serum solids ice cream held in the dispensing cabinet three months and subsequently placed in the hardening room for five months was the storage temperature difference between the dispensing cabinet at 6 and 10 degrees Fahrenheit and the hardening room -10 degrees Fahrenheit. This temperature difference can be interpreted as a mild form of heat shock and provides the condition necessary to cause the formation of sandiness in the high serum solids ice cream.

The presence of beta anhydride lactose in sandy material isolated from ice cream may be the result of a combination of several factors. The establishment of an equilibrium between beta and alpha lactose takes place very slowly at hardening room temperatures. It is then quite apparent that the factors promoting sandiness, and in particular, the formation of both alpha and beta lactose may be as follows: (a) the storage period of 8 months, (b) the temperature difference of a dispensing cabinet at 6 and 10 degrees Fahrenheit and the hardening room temperature of -10 degrees Fahrenheit, and (c) the high serum solids content of 15.5 per cent.

The sandy material isolated from several commercial samples of ice cream showed a different stage of crystal development than the sandy material found in the high serum solids ice cream. The crystals isolated from the commercial samples of ice cream showed the formation of alpha lactose crystals, and under favorable conditions for crystal growth and the promotion of sandiness, these crystals could be grown to the size and shape of the alpha lactose crystals present in the sandy material isolated from the high serum solids ice cream.

CONCLUSIONS

1. The optical data relating to the two forms of lactose present in the sandy material agreed with Wherry's (24) data for alpha hydrate and beta anhydride lactose.

2. Alpha hydrate and beta anhydride lactose, with the alpha form predominating, were found to be the only substances occurring as sandy material isolated from high serum solids ice cream.

3. Sandy material isolated from commercial ice creams showed alpha hydrate lactose crystals in the process of formation. The almost total absence of beta lactose crystals was apparently due to the fact that insufficient time had elapsed to permit this type of crystal to form and develop.

4. Pronounced sandiness formed in the high serum solids ice cream held in a dispensing cabinet at 6 and 10 degrees Fahrenheit for 3 months and subsequently maintained at -10 degrees Fahrenheit for five months. However, sandiness did not develop in the high serum solids ice cream which was held at -10 degrees Fahrenheit for 12 months because of the fact that it was held at a uniformly low temperature contrasted with the ice cream subjected to variable temperatures.

5. When using a modification of the technique of Zoller and Williams (2) and the optical data determined on commercially prepared lactose by Wherry (24) it was possible to identify the substances present in sandy material from ice creams as alpha and beta lactose.

6. Commercial ice creams in which sandiness had developed sufficiently to be detectable in the mouth showed alpha lactose crystals in the process of formation.

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