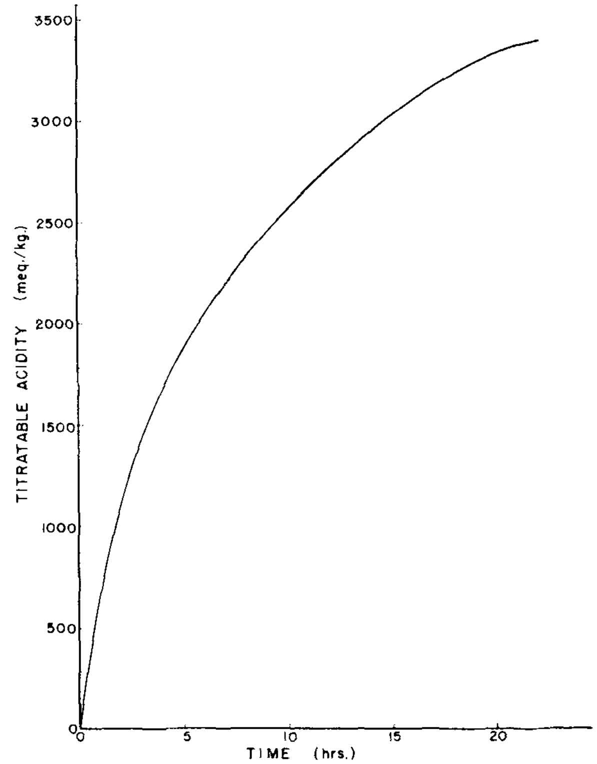

FIG. 8. Effect of increasing reaction time on the titratable acidity of autoxidizing methyl oleate. Catalyst concentration, 0.1% $Co(OAc)_2$; pressure, 100 p.s.i.a. air; temperature, 100°C.

built up if the reaction is continued long enough. After 22 hrs. about 3,400 meq./kg. of titratable acid was present in the oxidized oleate.

Discussion

The explosions were apparently caused by an accelerated oxidation of the liquid phase rather than by the reaction of gaseous by-products with O_2. In all explosions the liquid sample itself exploded, shattering the glass capsule; in some, mild combustion caused the glass wool to melt. In one experiment the explosion occurred as soon as the pressure was applied; thus a reaction due to by-product oxidation is ruled out unless it occurred extremely fast. It is possible that oxidative products, such as hydroperoxides, were built up in the samples until some critical concentration was reached, resulting in an explosion. It is also possible that the reaction involves a direct union of O_2 with the unsaturated linkage, suggested by Gunstone and Hilditch (5) as the initiating reaction in forming hydroperoxides. The method of carrying out the oxidation doubtless has a significant effect on the course of the reaction. In this work the oleate was essentially in the form of a thin film. Bulk oxidation, using a gas disperser, would perhaps give better yields, but the present state of our knowledge does not permit the extension of pressure oxidation to larger batches with any assurance that they would not explode.

Product analysis, which may throw some light on the peculiarities of pressure oxidation of unsaturated esters, has not been completed. The pressure oxidation of oleic acid is said to give a high yield of lactones (4). The oxidized methyl oleate reacts with neutral hydroxylamine to form hydroxamic acids, a reaction characteristic of both lactones and peresters (3). At the temperatures used some interesterification may take place to produce lactones from hydroxyesters, but peresters are equally likely.

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Melting and Dilatometric Behavior of 2-Oleopalmitostearin and 2-Oleodistearin[1]

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The glycerides, 2-oleopalmitostearin and 2-oleodistearin, which are important components of confectionery fats, were synthesized, and their melting behavior and dilatometric properties were determined.

Each glyceride was found to have four melting points: 18.2, 24.5, 33.0, and 37.4°C. for 2-oleopalmitostearin; 22.8, 30.0, 37.7, and 42.8°C. for 2-oleodistearin. The rate of transformation of the thermodynamically unstable polymorphs at temperatures just below their melting points was found to vary from a few seconds to more than eight days.

For each glyceride, expansivities were determined for three polymorphs and melting dilations for two polymorphs. Mixtures of the two glycerides were examined dilatometrically and found to behave in some respects as single components. The mixtures were readily tempered so that both components were in the same polymorphic form, and expansivities and melting dilations were obtained for mixtures in three polymorphic forms.

Dilatometric data also were obtained for a sample of cocoa butter and a sample of sweet milk chocolate of the coating type.

ON THE BASIS of both direct and indirect proof the triglycerides, 2-oleopalmitostearin and 2-oleodistearin, comprise about 75% of cocoa butter. Several investigators (1,11,17) have found that cocoa butter contains a large proportion of oleopalmitostearin, up to 57%; and recently conclusive evidence

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has been presented (4,15) that this glyceride occurs as the 2-oleate. Hilditch and Stainsby (11) and Meara (17) found that cocoa butter contained 2-oleodistearin to the extent of 19 and 22%, respectively. The melting points of a relatively pure fraction of the oleodistearin which Meara obtained from cocoa butter agree well with those found for synthetic 2-oleodistearin, which was prepared for the current investigation. Chapman *et al.* (4) presented evidence that the physical properties of mixtures of 2-oleopalmitostearin and 2-oleodistearin closely resemble those of the monounsaturated fraction of cocoa butter. Indirect proof of structure can be found in the fact that so far all disaturated triglycerides of vegetable oils which have been well characterized have been found to be of symmetrical configuration.

While there is ample proof as to the amounts and structures of the two major compounds in cocoa butter, there are relatively few data on their physical properties. Apparently only Lutton (14) and Lavery (12) determined the several melting-points of 2-oleopalmitostearin. Lutton found three melting points while Lavery found two melting points and one transition point.

Several investigators (6,12,13,14,16) determined the melting points of 2-oleodistearin but are in disagreement as to the number of melting points and the temperatures at which they occur. Practically no data are available for either compound on the ease of transformation from one polymorphic form to another. Dilatometric data are available only for what appears to be the next-to-highest melting form of 2-oleodistearin (5).

Information on the melting and dilatometric behavior of the two main components of cocoa fat can be put to direct use not only in the tempering and solidification operations in the manufacture of chocolate but also in the development of new cocoa butter-like fats from domestic oils. Such new fats should be compatible with cocoa butter. Also they should contain sizable proportions of the same components or closely-related positional isomers.

To obtain this information the two major components of cocoa butter were synthesized; the melting points of the several polymorphic forms of each compound were determined, together with the ease of transformation from one form to another. Dilatometric measurements were made on each compound and on their mixtures. For purposes of comparison dilatometric measurements also were made on cocoa butter and a sweet milk chocolate.

Experimental

Synthesis of Glycerides. Both the 2-oleopalmitostearin and 2-oleodistearin were prepared from 1-monostearin. The latter was isolated from a mixture of stearins obtained by reacting 100 parts of purified stearic acid with 40 parts of glycerol in the presence of 0.1% sodium hydroxide for 3 hrs. at 200°C. After removal of the catalyst and free glycerol the distearins and tristearin were separated from the mixture by repeated fractional crystallization from a 70% isopropyl alcohol—30% water solution, and the 1-monostearin was further purified by fractional crystallization from hexane (7). The melting point of the final 1-monostearin was 81.2°C., and its purity, according to the periodic acid method of analysis (9), was 99.6%.

The 1-monostearin was converted to 1-stearoyl-3-tritylglycerol by reaction with triphenylchloromethane, using essentially the procedure described by Verkade and van der Lee (19). The purified product melted at 69.9–70.0°C.

The 1-stearoyl-3-tritylglycerol was treated with palmitoyl chloride in the presence of quinoline to obtain 1-stearoyl-2-palmitoyltritylglycerol, which after purification melted at 37.1°C. The latter compound was detritylated, and the palmitoyl group was shifted from the 2- to 3-position to obtain 1-stearo-3-palmitin (19). The purified 1-stearo-3-palimitin melted at 70.7–71.0°C.

Oleoyl chloride was prepared from a highly-purified oleic acid, which was obtained by the fractional crystallization of the methyl esters of olive oil fatty acids essentially according to the procedure of Wheeler and Riemenschneider (20), followed by saponification and acidulation of the methyl oleate fraction.

To prepare the 2-oleopalmitostearin 29 g. (0.0964 mole) of the oleoyl chloride in chloroform solution were added slowly to a solution of 30 g. (0.0503 mole) of 1-stearo-3-palmitin and 33 g. (0.256 mole) of quinoline in chloroform. After refluxing the mixture for 3 hrs., it was allowed to stand over-night at room temperature. It was then dissolved in ethyl ether and washed successively with 0.5 *N* sulfuric acid, 5% potassium bicarbonate solution, and water. After drying over anhydrous sodium sulfate, the solution was filtered and the solvents were evaporated under reduced pressure. The reaction product was dissolved in petroleum ether, put on a column of activated alumina (twice the weight of the glyceride), and eluted with petroleum ether, which removed any diglyceride (4). The petroleum ether was evaporated, and the 2-oleopalmitostearin was then crystallized several times from acetone to obtain the purified compound melting at 37.4°C.

The 2-oleodistearin was prepared from the 1-stearoyl-3-tritylglycerol by procedures similar to those used in the preparation of the 2-oleopalmitostearin. In the preparation of 2-oleodistearin the intermediates, 1,2-distearoyl-tritylglycerol and 1,3-distearin, melted at 47.9–48.0 and 78.1°C., respectively. Analytical data for the two glycerides, 2-oleopalmitostearin and 2-oleodistearin, are recorded in Table I.

Melting Points. The several melting points of both 2-oleopalmitostearin and 2-oleodistearin were determined by the capillary tube method. Crystals of each glyceride, as obtained by fractional crystallization from a solvent, were sealed in glass tubes measuring about 1 mm. in diameter. Melting points were ob-

TABLE I

Analytical and Physical Data for 2-Oleopalmitostearin and 2-Oleodistearin

Property	2-Oleopalmitostearin	2-Oleodistearin
Melting point,[a] °C.		
Form I	37.4	42.8
Form II	33.0	37.7
Form III	24.5	30.0
Form IV	18.2	22.8
Density, g./ml. at 50°C.	0.88624	0.88502
Refractive index, $n^{45/D}$	1.45431	1.45452
Iodine value		
Found	29.0	28.5
Calc.	29.5	28.5
Hydroxyl value	0	0

[a] Capillary tube method.

tained in the conventional manner both before and after tempering the crystals. The samples were further examined over short temperature intervals by the "thrust-in" technique, that is, the samples were solidified and tempered at various temperatures for various periods of time and then thrust into a constant temperature bath maintained just above or below the temperature where melting was thought to occur. The temperature and length of time required to transform a lower melting polymorph into one having a higher melting point was noted.

Dilatometric Measurements. The dilatometric examinations were carried out in dilatometers of the gravimetric type with mercury as the confining liquid. The construction of these dilatometers, their method of use, and methods employed in making the various calculations have been described previously (2,3) except as noted.

In obtaining data on the highest melting polymorph of each glyceride, some evidence of vacuole formation was encountered. The effects of this phenomenon were eliminated by repeated partial melting and solidification of the sample during the early stages of tempering.

The dilatometric curve for the lowest melting form represented in Figures 1, 2, and 3 was obtained mostly by a "thrust-in" technique. Generally, for each point shown on the curve, the sample was melted completely, solidified by rapid chilling, and placed in the dilatometer bath at the indicated temperature. Then the mercury-containing flask attached to the dilatometer was loosened and lowered a few centimeters so that mercury could drop from the dilatometer into the flask as the sample expanded, but mercury could not be sucked from the flask into the dilatometer as the tempering effect became significant and the sample began to contract.

To obtain data on the next-to-highest melting polymorph of each glyceride, techniques which differed slightly with the different samples were employed. With 2-oleopalmitostearin the samples in the dilatometers were melted, then cooled for 10 min. in a water bath at 0°C., transferred to a water bath at 22°C., and held at this temperature for 30 min., transferred back to the water bath at 0°C. for 5 min., after which the samples were transferred to the dilatometer bath, the temperature of which had been lowered to −37.7°C. For measurements between −37.7°C. and 28.4°C. the temperature of the dilatometer bath was increased step by step and held at each test temperature for 20 min. Above 28.4°C. the samples were melted and retempered for each determination, and the "thrust-in" technique was employed.

To obtain data on the next-to-highest melting polymorph of 2-oleodistearin, the melted samples were solidified in ice water, held at 25°C. for two days, and placed in the dilatometer bath at the lowest test temperature. The samples were held at each test temperature for 30 min. Measurements below room temperature were made in one run, and measurements above room temperature were made in another.

For mixtures of 2-oleopalmitostearin and 2-oleodistearin the samples in the dilatometers were melted, placed in ice water for 30 min., and then tempered for 1 day at 25°C. Then a short segment of the dilatometric curve for each mixture was determined. The samples were held at each test temperature for 30 min. The entire operation was repeated until the complete curves were obtained.

Results and Discussion

Melting Behavior. The melting point of the lowest melting polymorph (Form IV) of 2-oleopalmitostearin was found only under somewhat unusual conditions. Not only was it necessary for the melt to be quickly chilled, but the time at the chilling temperature had to be of short duration. When the sample in a small capillary tube was heated thoroughly to above 60°C., plunged for 2 seconds in a bath at 0°C., and then put into a bath at 18.2°C., the sample melted and remained melted for 1 or 2 seconds. Tests showed that, with the capillaries employed, the 2 seconds at 0°C. completely solidified the sample, and the melting observed at 18.2°C. was not the result of a core of hot melt dissolving a film of solid fat on the inner wall of the capillary. Such a situation occurred only on reducing the chilling time at 0°C. to about 0.5 second. Holding the quick-chilled sample at 0°C. for short periods of time more than 2 seconds and then thrusting the sample into a bath at 18.2°C. produced a pronounced change in translucency but not complete melting. As the tempering time at 0°C. increased beyond about 40 seconds, the change in translucency became more difficult to detect.

When a sample of 2-oleopalmitostearin was heated to above 60°C., solidified by quick chilling to 0°C., and held at 0°C. for 30 min., Form III was obtained. It melted completely at 24.5°C. However, when the sample tempered for 30 min. at 0°C. was further tempered for 20 min. at 22°C., melting no longer occurred at 24.5°C. When a quick-chilled sample was melted at 24.5°C., then resolidified at 22.3°C., and held at this temperature for 2 min., it would no longer melt at 24.5°C.

Tempering a quick-chilled sample of 2-oleopalmitostearin at 22°C. so that it no longer melted at 24.5°C. produced Form II; the polymorph melted at 33.0°C. Form II also was obtained on cooling the melt to room temperature, about 26°C.

Form I, or the highest melting polymorph of 2-oleopalmitostearin, melting point 37.4°C., was usually but not always obtained on crystallization of the glyceride from a solvent such as acetone. On one occasion the crystals obtained from acetone melted at 32.8°C. Tempering these crystals at 32°C. gradually raised the melting point to 36.8°C. in the course of 4.5 days. Melted samples solidified at 27°C. and tempered for 5 days at 32°C. melted over the temperature range 36.5–37.4°C.

For 2-oleopalmitostearin the three melting-points found by Lutton (14) are in good agreement with three of the four melting-points found in the current investigation. He did not report a melting point at 24.5°C. The three melting-points for 2-oleopalmitostearin reported by Lavery (12) do not agree closely with melting points found in the current investigation; the difference is at least 2° in each case.

The melting behavior of 2-oleodistearin was similar to that of 2-oleopalmitostearin. The lowest melting polymorph, or Form IV of 2-oleodistearin, exhibited a fleeting melting-point, staying completely liquid for 1 or 2 seconds, when a melted sample was quickly solidified by thrusting it for 2 seconds into a bath at 0°C. and then thrusting it into a bath at 22.8°C. Tempering the quickly solidified solid at

TABLE II

Expansivity and Melting Dilation of 2-Oleopalmitostearin, 2-Oleodistearin, and Mixtures Containing Them

Compound or mixture	Temperature interval, °C.	Polymorphic form	Coefficient of expansion, ml./g./°C.	Melting dilation, ml./g.	Dilatometric melting point,[a] °C.
2-Oleopalmitostearin	—38 to +20	I	0.000272	0.1230	37.25
	—38 to +10	II	0.000424	0.1009	33.2
	—38 to + 3	III	0.000507	0.0757	24.2
	37 to 63	Liquid	0.000883	....	......
2-Oleodistearin	—38 to + 3	I	0.000281	0.1129	42.6
	—38 to — 5	II	0.000475	0.0932	37.7
	—38 to 0	III	0.000528	....	......
	42 to 63	Liquid	0.000873	....	......
Mixture	—38 to 0	I	0.000262	0.1159	35.5
2-Oleopalmitostearin, 90%	—38 to + 5	II	0.000423	0.0972	32.25
2-Oleodistearin, 10%	—35 to —10	III	0.000479	0.0774	24.75
	35 to 60	Liquid	0.000867	....	......
Mixture	—38 to + 3	I	0.000235	0.1194	42.2
2-Oleopalmitostearin, 10%	—38 to + 5	II	0.000479	0.0952	37.1
2-Oleodistearin, 90%	—38 to —10	III	0.000524	....	......
	42 to 60	Liquid	0.000872	....	......
Mixture	—38 to 0	I	0.000233	0.1148	36.1
2-Oleopalmitostearin, 72.5%	—38 to + 5	II	0.000434	0.0942	31.6
2-Oleodistearin, 27.5%	—38 to —10	III	0.000600	0.0741	25.9
	36 to 60	Liquid	0.000881	....	......
Cocoa butter	—38 to — 5	I	0.000462	0.0961	34.1
	—38 to — 5	II	0.000627	0.0657	27.3
	40 to 60	Liquid	0.000867	....	......
Milk chocolate, coating type	—38 to — 5	I	0.000206	0.0306	35.7, 33.0 [b]
	....	II	....	....	27.0, 24.5 [b]
	40 to 60	Liquid	0.000381	....	......

[a] Point of complete melting unless otherwise stated.
[b] Point of mid-melting.

0°C. for periods of time more than 2 and under 60 seconds caused the sample to undergo only a change in translucency on being thrust into a bath at 22.8°C.

Heating a sample of 2-oleodistearin to more than 60°C., then quickly solidifying and tempering it for several minutes at 0°C., produced Form III, melting point 30.0°C. On tempering Form III for just 80 seconds at 27.8°C., it was converted largely into Form II, melting point 37.7°C. Form II also was obtained on melting and heating the glycerides to more than 60°C., solidifying it at 26°C., and storing it overnight at this temperature.

On tempering Form II of 2-oleodistearin for 8 days at 35°C., the melting point gradually increased from 37.7 to 42.0°C. A sample of this glyceride completely in the highest melting form, Form I, was found to melt at 42.8°C.

The melting points found for 2-oleodistearin are in fair agreement with those reported by Daubert and Clarke (6) and would also be in fair agreement with those reported by Malkin and Wilson (16) except for the fact that the latter found melting points at both 41.5 and 43.5°C. instead of a melting point at about 42.8°C. Lutton (13) found three definite melting-points. In a later article (14) four are listed, one of which was associated only with crystals precipitated from solvent. Lavery (12) found melting points at about 36.8 and 41.8°C. and a solid-to-solid transformation at 22.4°C.

Filer *et al.* (8) characterized polymorphs of 2-oleodistearin melting at 43.0 and 37.6°C., which melting points correspond almost exactly with the melting points of Forms I and II, respectively. The polymorph melting at 43.0°C. was shown to be the *beta* form, and that melting at 37.6°C. was the *beta* prime form.

In addition to the melting points reported for the two glycerides, transition points probably exist at which visual melting cannot be made to occur. Lutton (14) claims both glycerides undergo a reversible transformation between sub-*alpha* and *alpha* forms below 0°C. In the current investigation it was found that, on solidifying 2-oleodistearin at room temperature, holding it over-night at this temperature, then heating it gradually to its melting point, 37.7°C., a change in appearance occurred at 35°C. The sample became whiter and more nearly opaque.

Dilatometric Behavior. The volumetric expansions *vs.* temperature for three of the polymorphs of 2-oleopalmitostearin are shown graphically in Figure 1. The coefficients of expansion in the solid and liquid states, the volume changes accompanying melting, and the dilatometric melting points obtained from these curves are recorded in Table II, together with data obtained from the dilatometric curves in the other figures.

A dilatometric curve for Form IV of 2-oleopalmitostearin could not be obtained because the size of the sample in the dilatometer, several grams, made impossible the extremely rapid rate of solidification

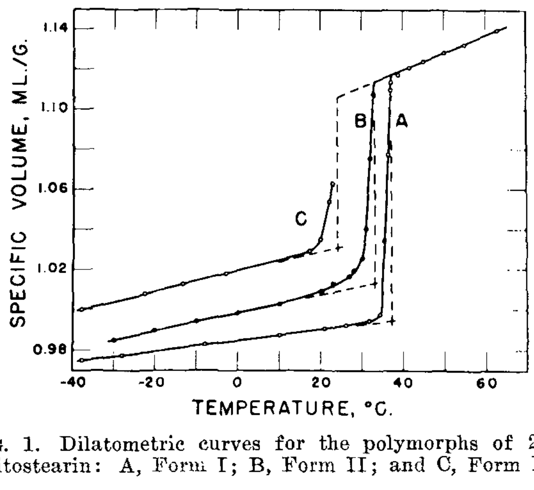

FIG. 1. Dilatometric curves for the polymorphs of 2-oleopalmitostearin: A, Form I; B, Form II; and C, Form III.

and heating required to obtain data on this polymorph. Even Form III was so unstable that the complete curve could not be obtained by the "thrust-in" technique. No difficulty was encountered in obtaining a curve for Form II, which converts to the thermodynamically stable polymorph, Form I, at a relatively slow rate.

Dilatometric curves for three of the polymorphs of 2-oleodistearin are given in Figure 2. Here also the curve for Form III, Curve C, is incomplete because of the marked lack of stability. The discontinuity in Curve C at 2°C. was caused by the partial transformation of Form III to Form II under the particular test-conditions employed. Under other test-conditions this discontinuity could appear at a higher or lower temperature.

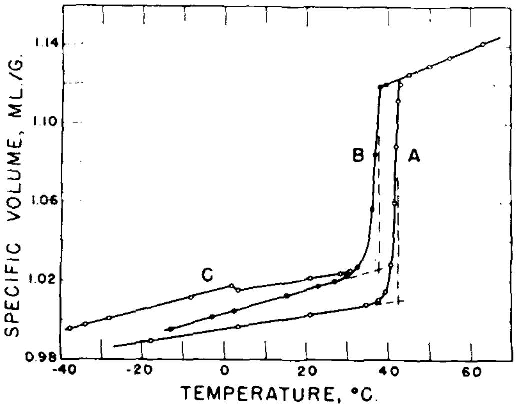

FIG. 2. Dilatometric curves for the polymorphs of 2-oleodistearin: A, Form I; B, Form II; and C, Form III.

As mentioned earlier, the only dilatometric data heretofore available for the two glycerides were data obtained by Craig *et al.* (5) for one polymorph of 2-oleodistearin. They apparently obtained data on the polymorph identified here as Form II, Curve B of Figure 2. Their calculated value for the melting dilation was 0.0925 ml./g., which is in good agreement with the value of 0.0932 ml./g. listed in Table II. Their calculated expansibility was 0.00060 ml./g./°C., which is somewhat higher than the value of 0.000475 ml./g./°C., recorded in Table II.

Dilatometric data were obtained for three mixtures of 2-oleopalmitostearin and 2-oleodistearin (Table II). The set of dilatometric curves obtained for a mixture of 72.5% 2-oleopalmitostearin and 27.5% 2-oleodistearin is given in Figure 3. The ratio of the two glycerides in this mixture is the same as that found in cocoa butter.

The curves in Figure 3 are typical of those found for the three mixtures with one exception. A complete curve could not be obtained for Form III of the mixture containing 90% 2-oleodistearin and 10% 2-oleopalmitostearin. While it is a recognized fact that the rate of polymorphic transformations in a glyceride are retarded by the addition of other glycerides, the presence of 10% of 2-oleopalmitostearin in the 2-oleodistearin did not slow the rate of transformation in the latter sufficiently to allow measurements to be made by the "thrust-in" technique.

Dilatometrically the mixtures behaved like single compounds. The melting ranges were short, and the mixtures could be tempered like single compounds. The melting point of each mixture was within about one degree of that predicted by the Hildebrand solubility equation (10).

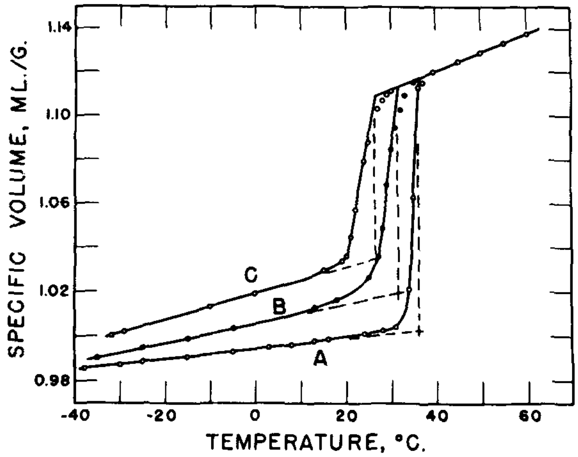

FIG. 3. Dilatometric curves for the composition 72.5% 2-oleopalmitostearin and 27.5% 2-oleodistearin: A, both glycerides in Form I; B, both glycerides in Form II; and C, both glycerides in Form III.

According to the Hildebrand equation, an eutectic mixture of the two glycerides should contain 78% 2-oleopalmitostearin, which is close to the 72.5% mixture represented in Figure 3. To what extent an eutectic actually formed and its composition will be determined in another investigation.

In Figure 3 the plotted points for the upper portions of the melting segments of the curves show that melting became more gradual as the liquid line was approached, indicating the presence of a small amount of higher-melting solids. The occurrence of a small amount of polymorphic transformation at the melting temperatures may have been responsible. On the other hand, the observed behavior may have been caused by the limited segregation of components during solidification and tempering.

For purposes of comparison, dilatometric curves were obtained for a sample of commercial cocoa butter, Figure 4. Curve A was obtained on chilling the melted sample to O°C., holding the sample at this

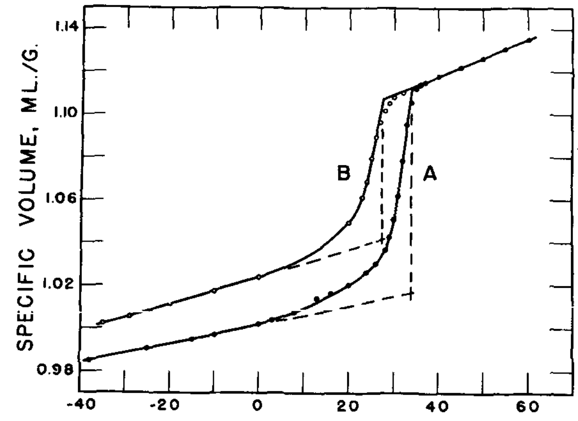

FIG. 4. Dilatometric curves for a sample of commercial cocoa butter: A, the two major glycerides, 2-oleopalmitostearin and 2-oleodistearin, in Form I; B, the two major glycerides in Form II.

temperature for 30 min., and tempering for 15 days at 25°C. Curve B was obtained on chilling the melted sample at 0°C., holding it at this temperature for 30 min., and putting it in the dilatometer bath held at about −38°C.

The approximately 14% of diunsaturated glycerides in cocoa butter (17) melt at temperatures below those of the two main components. According to the curves in Figure 4, melting starts at about 0°C. From Curve A, Figure 4, it was calculated (18) that this sample of cocoa butter after being well-tempered contained the following percentages of liquid:

Temperature, °C.	Content of liquid, %
0	0.6
5	1.9
10	4.1
15	6.8
20	10.8
25	16.7
30	36.1
34.1	100.0

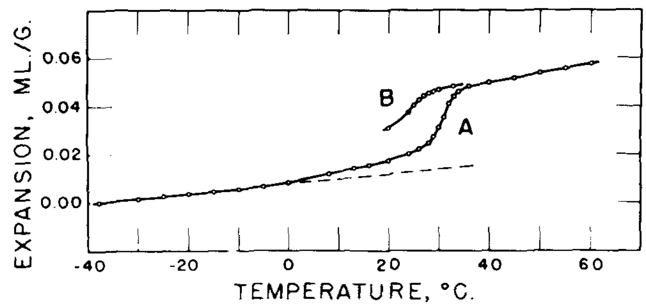

FIG. 5. Dilatometric curves for a sample of sweet milk chocolate, coating type: A, the two major glycerides of cocoa fat in Form I; B, the two major glycerides in Form II.

Dilatometric curves also were obtained for a sample of commercial, sweet milk chocolate, coating type (Figure 5). To obtain Curve A the chocolate, as received from the manufacturer, was tempered for several months at room temperature. A small block of this chocolate was then sealed in a dilatometer, and measurements were made without the usual melting, solidification, and tempering treatments in the dilatometer. To obtain Curve B a tempering procedure similar to that used to obtain Curve B for cocoa butter was employed.

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A Small Laboratory Model, Wiped-Surface Heat Exchanger for Chilling and Texturating Shortenings[1]

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A miniature Votator type, wiped-surface heat exchanger for chilling and texturating shortening has been described.

The package unit, mounted on a table about 2 x 4 ft. in dimensions, fitted with casters for mobility, needs only to be plugged into an electrical outlet for operation, operates at fat feed-rates of 10 to 80 lbs. per hour, with an integrated ¾ ton Freon refrigeration unit capable of holding the chilling unit to ±1°F. in the range −30°F. to +60°F., 4-step pulley agitator drives on both chilling and texturating units giving 330 to 1170 r.p.m. speed range, aluminum alloy chilling and texturating units, fat and air Flowrators with needle valve control, free-swinging wiper blades, and a few other minor features that lead to smooth operation.

The unit has been found useful in our laboratory to process various types of shortenings for research purposes, to supply special shortenings to prospective customers for evaluation, and to check the operation of the plant Votators.

IN THE MID-1930's the closed continuous internal chiller and plasticizer, known as the Votator, came into use and has substantially replaced the chill roll. Bailey (1), Dawson (2), Fincher (3), James (5), Joyner (6), McMichael (7,9), and Slaughter (9) have given excellent descriptions of the various systems used for chilling and texturating plastic fats, including margarine.

In 1957 Steffen and Vander Wal (10) described a batch procedure for plasticizing small samples of fats in the laboratory, using a refrigerated Kitchen-Aid Mixer (Model K-4-B). More recently, in 1959, Harrington, Bates, and Stingley (4) described a batch laboratory-size plasticizer, which will chill and plasticize a 1,300-g. sample in 4 min., giving a product very similar in consistency and performance to a plant plasticized product.

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