methyl esters. It was a dark brown viscous semi-solid with an iodine value of 98.29.

The total composition of the oil is reported in Table II as calculated from the above fractionation.

Spectrophotometric analysis of the pentane-extracted oil previously mentioned in this paper showed 0.14% of conjugated diene acids and 0.01% conjugated triene acids. After alkali conjugation it was estimated that 1.0% linolenic acid was present. Analysis of the individual fractions by thiocyanogen and iodine value did not indicate more than a trace of linolenic acid. It is possible that the conjugated triene acid was from a fatty acid of different molecular weight than linolenic acid. We, therefore, have indicated linolenic acid as present but not in significant quantities.

TABLE II

Composition of the Mixed Fatty Acids of Buttonweed Seed Oil

| Fatty Acid | Weight Percent |
|---|---|
| Myristic | trace |
| Palmitoleic | 0.6 |
| Palmitic | 12.2 |
| Linolenic | trace |
| Linoleic | 58.0 |
| Oleic | 14.1 |
| Stearic | 0.9 |
| Boiling above $C_{18}$ fatty acids | 9.4 |
| Residue | 4.7 |
| | 100.0 |

The outstanding fact from this analysis is the very high percentage of linoleic acid in the $C_{18}$ fatty acid group. The ease of separation of this fraction from the rest of the product makes this oil an unusually good source of linoleic acid.

### Discussion

The composition of the variety of buttonweed (Abutilon) studied by the authors is considerably different from either cottonseed or soybean oil and disagrees with the work of Jolson in this respect.

The importance of buttonweed seed oil appears to be primarily in its high linoleic acid content. This is particularly valuable because of the ease with which a high concentrate of linoleic acid may be obtained by simple fractionation.

In view of the present shortage of oils the recovery of this oil from seeds which at the present time are either thrown away or sold as screenings to feed mills appears to have considerable promise. The most serious drawback is the relatively low oil content. However, this is largely offset by the low cost of the seed.

### Summary

Buttonweed seeds (Abutilon theophrasti) contain 15 to 17% oil. The oil contains about 58% linoleic acid which may be concentrated by simple fractionation into fractions containing as high as 83% of the acid. This suggests the possible use of the oil as a source of linoleic acid. The seed also has an appreciable sterol content.

### Acknowledgment

The authors wish to acknowledge the work of Betty R. Fisher in carrying out many of the analytical experiments.

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# Dilatometric Investigations of Fats

## III. The Density, Expansibility, and Melting Dilation of Some Simple Triglycerides and Other Fats

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PREVIOUS papers (2, 8) in this series have called attention to the usefulness and the convenience of dilatometry as a tool in phase investigations of fats. However, in general, dilatometric data can be interpreted quantitatively only if one knowns accurately the volume changes associated with phase transformations in the material examined, as well as the thermal expansibility of the material, in both solid and liquid states.

There are data in the literature on the thermal expansibility of liquid commercial fats (9, 15) and also on certain pure liquid triglycerides (7). However, accurate information on the expansibility of fats in the solid state or on the volume changes accompanying melting and polymorphic transformations is wholly lacking. The purpose of this investigation has been to supply such information.

### Samples

Samples of trilaurin, trimyristin, tripalmitin, tristearin, triolein, and trielaidin were prepared from purified fatty acid chlorides, as described previously (1). Neutralization values of the saturated fatty acids and iodine and thiocyanogen values of the unsaturated acids agreed with theoretical values for pure materials within experimental errors of the methods. Previous experience had shown that the method of esterification employed, using acid chlorides in 10% excess over the glycerol, produces a material containing less mono- or diglycerides than can be detected by chemical methods.

The samples of partially hydrogenated and highly hydrogenated cottonseed oil, which had iodine values of 59.5 and 0.85, respectively, were the same as those employed in previous calorimetric investigations (10, 12). The lard used was a commercial sample of prime steam lard which had an iodine value of 66.6.

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[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

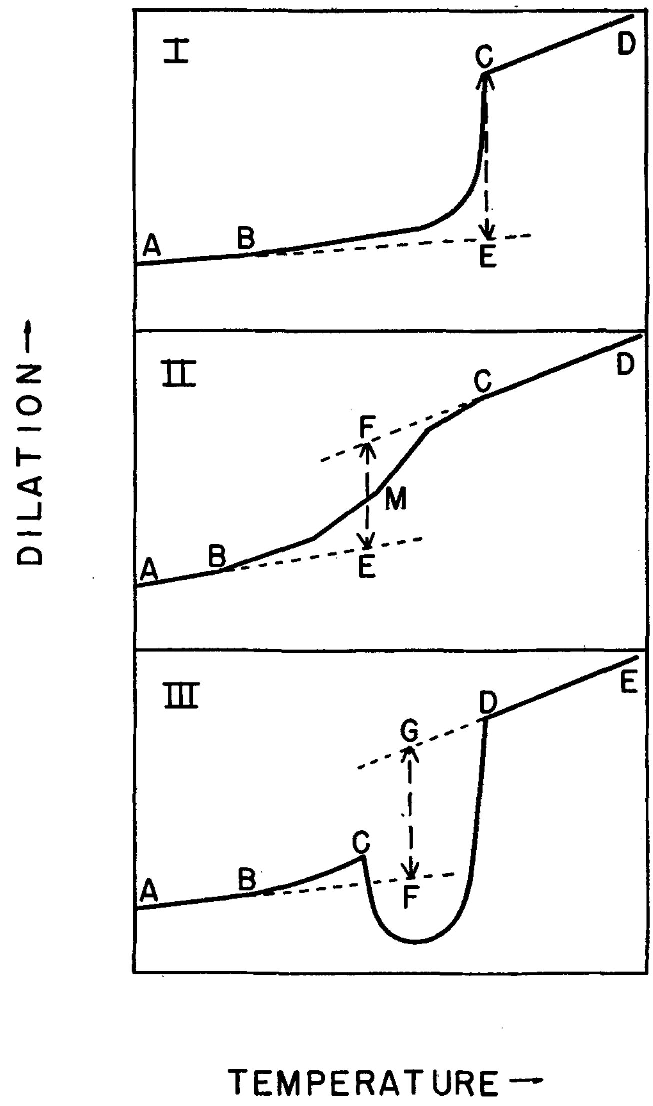

FIG. 2. *Curves illustrating methods of calculating melting* dilation of fat samples.

### Densities

The densities and specific volumes of the different samples, as determined for the liquid state, and as calculated from the dilatometric results for the solid state at *ca.* —38° C. are shown in Table 2. It was known from previous calorimetric examination of the samples that the tristearin, tripalmitin, trimyristin, trilaurin, highly hydrogenated cottonseed oil, and partially hydrogenated cottonseed oil are completely solid at —38° C. and it seems very improbable that the trielaidin, triolein, or lard could have contained any liquid material at this temperature.

The densities of the simple saturated triglycerides in the liquid state at 80° C. and in the most stable solid form at —38° C. are shown graphically as a function of chain length of the fatty acids in Figure 3.

### Expansibility in the Solid State

It was not possible to obtain reliable values for the expansibility of all the samples in the solid state, due to the unfortunate circumstance that the method is limited to temperatures above the freezing point of mercury (—38.9° C.). Some fats do not remain completely solid at temperatures sufficiently far above this point for accurate measurements of thermal expansion to be made. However, in the samples of trilaurin, trimyristin, tripalmitin, tristearin, and highly hydrogenated cottonseed oil in the highest melting Form I the solid range was sufficiently wide (*ca.* 18° C.) to permit what may be considered reasonably accurate determinations. The solid range of tristearin in Forms II or III was less, hence the expansibilities for these

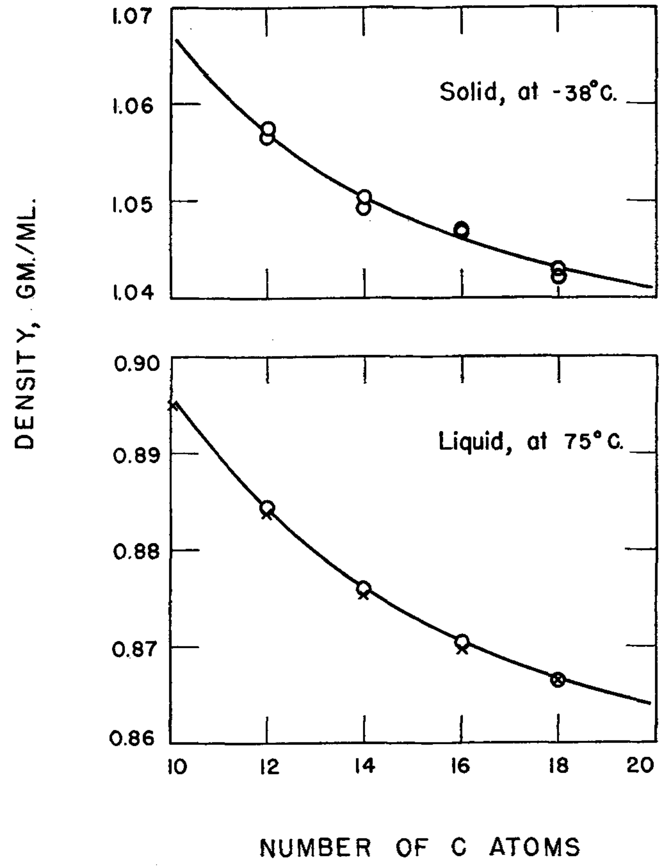

FIG. 3. *Density of simple saturated triglycerides in the liquid and in the most stable solid form, as a function of chain length of the fatty acids. Circles represent data obtained in this work; the crosses represent data of Joglekar and Watson (7).*

TABLE 2
Densities and Specific Volumes of the Fat Samples in the Liquid and Solid States

| Sample | Form | Liquid State | | | Solid State | | |
|---|---|---|---|---|---|---|---|
| | | Temp. | Density | Sp. Vol. | Temp. | Density | Sp. Vol. |
| Trilaurin | I | 55.0 | 0.8986 | 1.1128 | —38.6 | 1.057 | 0.946 |
| Trimyristin | I | 73.9 | 0.8767 | 1.1406 | —38.4 | 1.050 | 0.953 |
| Tripalmitin | I | 75.2 | 0.8706 | 1.1486 | —38.2 | 1.047 | 0.955 |
| Tristearin | I | 80.7 | 0.8625 | 1.1594 | —38.6 | 1.043 | 0.959 |
| Tristearin | II | ...... | ...... | ...... | —38.0 | 1.017 | 0.983 |
| Tristearin | III | ...... | ...... | ...... | —38.0 | 1.014 | 0.987 |
| Trielaidin | I | 55.0 | 0.8872 | 1.1271 | —38.0 | 1.017 | 0.983 |
| Triolein | I | 25.0 | 0.9078 | 1.1016 | —38.7 | 1.012 | 0.988 |
| Highly hydrogenated cottonseed oil | I | 73.9 | 0.8694 | 1.1502 | —38.2 | 1.022 | 0.978 |
| Partially hydrogenated cottonseed oil [a] | | 55.0 | 0.8867 | 1.1278 | —38.6 | 1.000 | 1.000 |
| Lard [a] | | 55.0 | 0.8885 | 1.1255 | —38.6 | 1.005 | 0.995 |

[a] Tempered at 29° C.

TABLE 3
Expansibility of the Different Fats in the Solid State

| Sample | Form | Temperature Range, °C. | Expansion, ml./g./°C. | Average Expansion |
|---|---|---|---|---|
| Trilaurin | I | —38 to —24<br>—38 to —23 | 0.000182<br>0.000193 | 0.00019 |
| Trimyristin | I | —38 to —22<br>—38 to —21 | 0.000210<br>0.000213 | 0.00021 |
| Tripalmitin | I | —38 to —28<br>—38 to —18 | 0.000223<br>0.000219 | 0.00022 |
| Tristearin | I | —38 to —28<br>—35 to —20 | 0.000227<br>0.000230 | 0.00023 |
| Tristearin | II | —38 to —33<br>—38 to —33[b] | 0.000298<br>0.000277 | 0.00029 |
| Tristearin | III | —38 to —32<br>—38 to —33 | 0.000311<br>0.000324 | 0.00032 |
| Trielaidin | I | —33 to —22 | 0.00018 | 0.00018 |
| Triolein | I | —38 to —33<br>—38 to —33 | <0.00038<br><0.00038 | <0.00038 |
| Highly hydrogenated cottonseed oil | I | —38 to —20<br>—38 to —20 | 0.000282<br>0.000300 | 0.00029 |
| Partially hydrogenated cottonseed oil [a] |  | —38 to —33<br>—38 to —33 | <0.00039<br><0.00038 | <0.00039 |
| Lard [a] |  | —38 to —33 | <0.00038 | <0.00038 |

[a] Sample tempered at 25° C.

materials are less certain. Previous calorimetric examination of the partially hydrogenated cottonseed oil (12) revealed that melting began at about —38° C., hence only a maximum value could be obtained for this fat. The rate of dilation of the lard and of the triolein immediately above —38° C. was almost exactly equal to that of the partially hydrogenated cottonseed oil. It appears probable, therefore, that the dilation recorded in this range is somewhat greater than that representing true thermal expansion in the solid state but that each of these fats is entirely or almost entirely solid at —38° C.

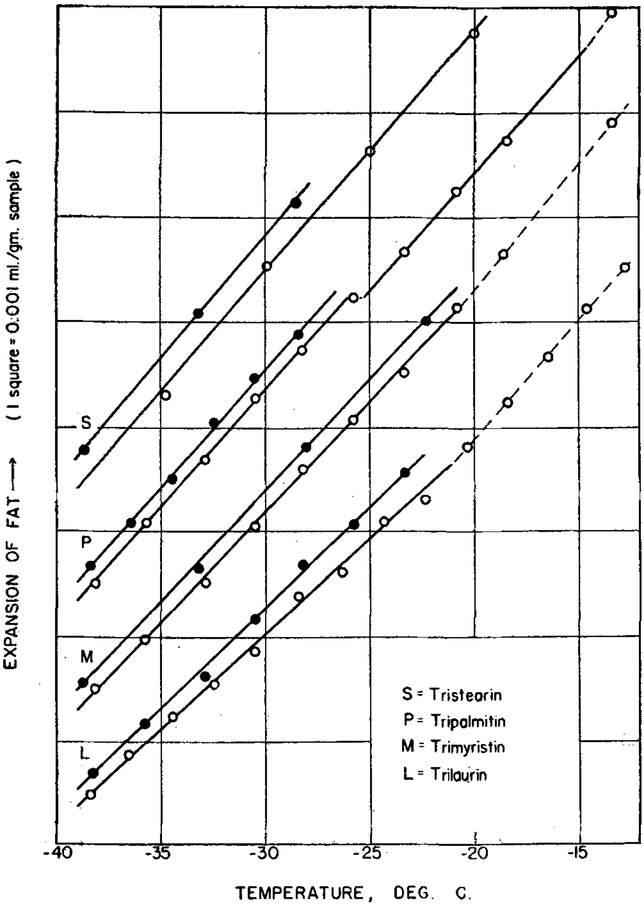

FIG. 4. Expansibility of simple saturated triglycerides in the most stable solid form. (Duplicate runs.)

It was not practicable to make measurements of dilations below —38° C. by using a liquid other than mercury in a volumetric dilatometer, since the solid expansibility measurements require the detection of volume changes of the order of 0.0001 ml. per gram of sample, and this is beyond the sensitivity of a volumetric instrument.

The data on expansibility of the solid materials are summarized in Table 3. Detailed data obtained in duplicate experiments on the series of simple saturated triglycerides are shown in Figure 4.

### Expansibility in the Liquid State

The results of duplicate measurements of the expansibility of the different samples in the liquid state are given in Table 4.

TABLE 4
Expansibility of the Different Fats in the Liquid State

| Sample | Temperature Range, °C. | Expansion ml./g./°C. | Average Expansion |
|---|---|---|---|
| Trilaurin | 47 to 60<br>47 to 64 | 0.000903<br>0.000890 | 0.00090 |
| Trimyristin | 57 to 66<br>58 to 67 | 0.000920<br>0.000905 | 0.00091 |
| Tripalmitin | 66 to 76<br>66 to 80 | 0.000909<br>0.000928 | 0.00092 |
| Tristearin | 73 to 80<br>73 to 78 | 0.000925<br>0.000913 | 0.00092 |
| Trielaidin | 55 to 62<br>44 to 52 | 0.000877<br>0.000872 | 0.00087 |
| Triolein | 18 to 27<br>7 to 25 | 0.000812<br>0.000823 | 0.00082 |
| Highly hydrogenated cottonseed oil | 74 to 78<br>74 to 78 | 0.000914<br>0.000907 | 0.00091 |
| Partially hydrogenated *cottonseed oil* | 45 to 59<br>45 to 59 | 0.000866<br>0.000872 | 0.00087 |
| Lard | 45 to 59 | 0.000870 | 0.00087 |

### Melting Dilations

The dilatometric data between *ca.* —38° C. and temperatures above the melting points of the different fats, together with the calculated melting dilations, are given in Table 5. A value of 0.00030 ml./g./°C. for expansibility of the solids was taken arbitrarily for calculations of the approximate melting dilation of triolein, lard, and partially hydrogenated cottonseed oil.

The melting dilations of tristearin in Forms II and III were calculated at 64.5° and 54.5° C., respectively, since these melting points were found for the respective forms by rapid heating of the samples in capillary tubes (1).

In Figure 5 are shown melting dilations for the series of simple saturated triglycerides as a function of the chain length of the component fatty acids.

### Polymorphic Transformations in Tristearin

The upper portions of comparative dilatometric curves for tristearin in the three different polymorphic forms are reproduced in Figure 6. These curves were constructed from closely agreeing measurements made on duplicate samples of material.

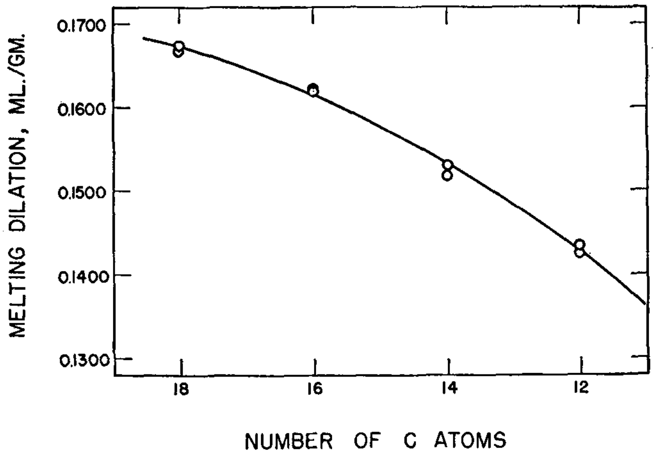

FIG. 5. Melting dilation of simple saturated triglycerides in the most stable solid form, as a function of chain length of the fatty acids.

In addition to the melting dilations for the different crystal forms which are recorded in Table 5, the following volume changes (contractions) were calculated for transitions from one form to another:

Form III to Form II at 54.5° C.........................0.0061 ml./g.
Form III to Form I at 54.5° C.........................0.0354 ml./g.
Form II to Form I at 64.5° C.........................0.0299 ml./g.

The dilatometric curves show that transformation of Form III began at a temperature considerably short of the melting point of this form, with rapid transformation occurring at about 48° C. Surprisingly, rapid transformation of Form II began at the even lower temperature of about 44° C. In neither Form II nor Form III did transformation take place uniformly; there are two well-defined plateaus in the portion of the Form III curve preceding rapid transformation and one similar plateau in the Form II curve. There is also a slight irregularity in each curve in the range 52°-62°C.

The course of transformation of Forms II and III was followed by taking x-ray diffraction photographs (1) of the original samples of Forms II and III and of portions of the samples taken at intervals along the dilatometric curves. The x-ray photographs revealed that at points representing incomplete transformation the material consisted in each case of the original form mixed with Form I. Form II was not detected in incompletely transformed Form III. It appears, therefore, to be impossible to prepare tristearin in Form II by heat treatment of Form III, as such treatment causes the material to pass directly from Form III to Form I.

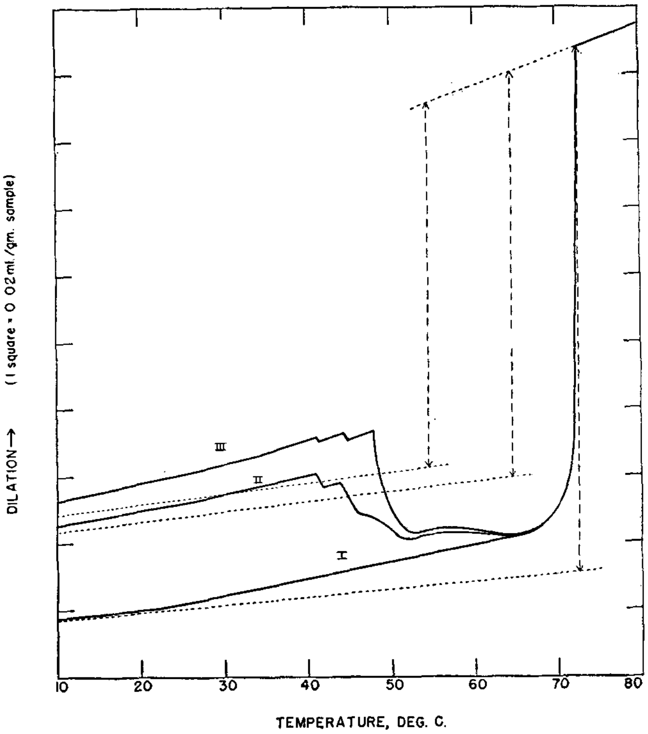

FIG. 6. Dilatometric curves showing the melting dilation of tristearin in different polymorphic forms.

## Discussion of Results

While the present data are not complete with respect to all types of triglycerides and triglyceride

TABLE 5

Over-All Volume Change Between *ca.* —38° C. and a Temperature Above the Melting Point, and Melting Dilation of the Different Fats

| Sample | Form | Range, °C. | Dilation | | | |
|---|---|---|---|---|---|---|
| | | | Total, ml./g. | Melting | Average Melting | Temp. °C.[a] |
| Trilaurin | I | —38.6 to 46.5<br>—38.6 to 46.5 | 0.1585<br>0.1595 | 0.1423<br>0.1433 | 0.1428 | 46.5 |
| Trimyristin | I | —38.6 to 64.0<br>—38.2 to 63.0 | 0.1796<br>0.1774 | 0.1529<br>0.1517 | 0.1523 | 56.6 |
| Tripalmitin | I | —38.2 to 74.0<br>—38.2 to 74.0 | 0.1924<br>0.1923 | 0.1619<br>0.1618 | 0.1619 | 65.5 |
| Tristearin | I | —38.6 to 77.6<br>—38.2 to 78.0 | 0.1976<br>0.1973 | 0.1674<br>0.1668 | 0.1671 | 72.5 |
| Tristearin | II | —38.0 to 74.5<br>—38.0 to 74.5 | 0.1709<br>0.1701 | 0.1320<br>0.1312 | 0.1316 | 64.5 |
| Tristearin | III | —38.0 to 74.5<br>—38.0 to 74.5 | 0.1670<br>0.1674 | 0.1190<br>0.1194 | 0.1192 | 54.5 |
| Trielaidin | I | —38.0 to 42.0 | 0.1326 | 0.1182 | 0.118 | 42.0 |
| Triolein | I | —38.7 to 5.0<br>—38.0 to 6.8 | 0.0974<br>0.0967 | 0.0840<br>0.0815 | 0.083 | 4.6 |
| Highly hydrogenated cottonseed oil | I | —38.2 to 74.0<br>—38.2 to 74.0 | 0.1716<br>0.1721 | 0.1319<br>0.1324 | 0.1322 | 62.5 |
| Partially hydrogenated cottonseed oil[b] | | —38.6 to 59.0 | 0.1314 | 0.0760 | 0.076 | 13.8 |
| Lard[b] | | —38.6 to 59.0 | 0.1340 | 0.0732 | 0.073 | 3.5 |

[a] Temperature at which melting dilation was calculated—point of half-fusion for lard and partially hydrogenated cottonseed oil; point of final melting for all other samples.

[b] Sample was tempered at 29° C.

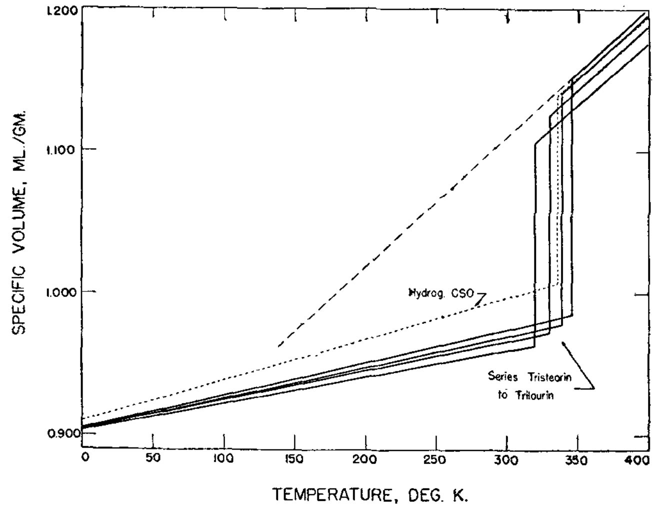

FIG. 7. Calculated dilatometric curves of pure simple saturated triglycerides and highly hydrogenated cottonseed oil showing expansibility and melting dilation in relation to absolute temperature.

mixtures, they nevertheless suggest certain generalizations applicable to this class of materials.

The calculated dilatometric curves of pure compounds in the homologous series represented by trilaurin, trimyristin, tripalmitin, and tristearin, in terms of specific volume *vs.* absolute temperature, are represented in Figure 7. It may be seen that the specific volume decreases as the molecular weight decreases, in both solid and liquid states, but that the expansibility of the fats in the solid state increases with increased molecular weight so that the curves projected linearly to absolute zero tend to converge at a specific volume of *ca.* 0.905 ml. per gram. A similar tendency in the dilatometric curves of the even-numbered *n*-paraffin hydrocarbons has been mentioned by Seyer, Patterson, and Keays (13). It is to be noted, however, that the hydrocarbons differ from triglycerides in having an inverse relationship between specific volume and chain length so that their expansibility decreases as the molecular weight increases.

The highly hydrogenated sample of cottonseed oil, consisting essentially of mixed glycerides of stearic and palmitic acids, had a melting dilation less than that of either tristearin or palmitin, but a higher expansibility in the solid state so that its solid line intersects the specific volume axis near the same point as the solid lines of tristearin, tripalmitin, etc. Other triglycerides, including the two unstable forms of tristearin, appear to fall into the pattern indicated in Figure 6, hence it appears probable that this pattern defines in a general way interrelationships amongst the various properties of melting point, density, expansibility of the solid, and melting dilation. Obviously the melting dilation will generally be lower, the lower the melting point of the material.

It will be seen from Figure 7 that the liquid line of any fat projected linearly according to the slope at the melting point does not intersect the solid line similarly projected at absolute zero, in accordance with the rule of Tammann (14), but at about 100° K. Therefore, calculations of the melting dilation from this rule and incomplete expansibility and specific volume data can be expected to be somewhat in error. Block (4) has previously reported that, while Tammann's rule predicts the melting dilation of many materials with accuracy, it gives a value about 20% too high for stearic acid.

The data on the lard and on the two hydrogenated cottonseed oil samples indicate that the melting dilation of mixed glycerides is not additive on the basis of the component fatty acids, but is less than that calculated from data on simple triglycerides containing the same fatty acids.

Within the limited range covered there is no great variation in expansibility in the liquid state according to chain length of the glycerides. Similarly, Seyer, *et al.* (13) found the expansibility of liquid *n*-paraffin hydrocarbons to vary little with chain length. The expansibility of liquid glycerides decreases markedly with increase in unsaturation, however, and the density increases.

A point of particular interest is the high degree of correspondence between the present volume and density data and thermal data obtained on some of the same fats in a series of calorimetric investigations (1, 3, 10, 11, 12) carried out in this laboratory.

There is a striking similarity of form between the dilatometric curves of fats, which show the variation of volume with change in temperature, and calorimetric curves, in which the heat content is similarly represented as a function of temperature. In such curves the melting dilation is comparable to the heat of fusion, whereas coefficients of expansion correspond to specific heats. Actually, in all of the fats examined so far by both methods there has been a general correspondence between expansibilities and specific heats and a remarkably close correspondence, as indicated in Figure 8, between the heats of fusion and melting dilations. This is, of course, not surprising when it is recalled that dilation of a material occurs through its molecules becoming separated to an increased degree and that the specific heat and the heat of fusion are in part measures of the energy required to effect this separation.

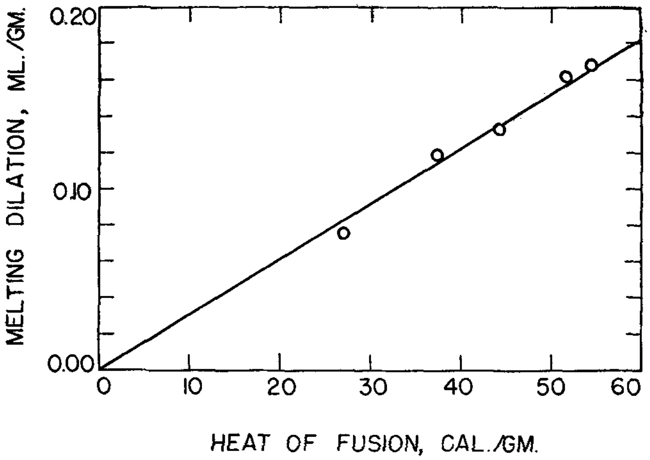

FIG. 8. Melting dilation *vs.* heat of fusion of triglycerides.

The fundamental thermodynamic equations which might be expected to shed light upon the relationships discussed above are complicated by factors which are unknown for triglycerides, or indeed, for any series of organic compounds. However, it is to be noted that in the Clapeyron equation concerning equilibrium between phases a direct relationship is expressed between latent heat and change of volume with change of state and that the rule of Gruneisen expresses a similar relationship between thermal expansion and specific heat.

### Summary

1. Dilatometric curves between *ca.* —38° C. and temperatures representing the liquid state have been determined for a number of pure triglycerides and some commercial fats.

2. From the dilatometric data and density data on the liquid samples, determinations have been made of the density in both the liquid and the solid states, the expansibility of each state with increase in temperature, and the dilation accompanying melting of the samples. From similar comparative data on the three polymorphic forms of tristearin determinations have been made of the volume changes accompanying transformation from one form to another.

3. Certain relationships are pointed out among the various properties of melting point, density, expansibility, melting dilation, specific heat and heat of fusion, and the relation of these properties to chain length and degree of unsaturation in triglycerides is discussed.

### Acknowledgments

The authors wish to express their appreciation of the kind assistance of S. T. Bauer and Maizie Caravella, who prepared the pure triglycerides used in this investigation; of Florence B. Kreeger, who made the x-ray examinations; of Frank C. Magne, who determined the densities of the samples in the liquid state; of Grace H. Charbonnet, who assisted in making some of the dilatometric measurements; and of R. O. Feuge, who helped in designing and building the low-temperature bath.

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# Tenderness of Pastries Made With Different Soy Flours

CLARA GABEL and GERTRUDE SUNDERLIN

A STUDY to determine the shortening value of fat in different types of soy flour furnished evidence that the fat in the flour is effective as a shortening agent in pastries made with soy flour.

Three series of pastry mixes consisting of 12 variants were set up to show variations in the tenderness in wafers when three types of soy flour containing different percentages of fat were used. In the first series the plastic fat used in the pastry formulas was constant, but the total fat in the pastries varied because of the different amounts of fat in the five flours used. In the second series the total fat in the pastries was constant because the plastic fat added was reduced by the amount of fat present in the different flours. In the third series defatted flour was used, and the total fat in the pastries was constant. In three of the mixes in this series soybean oil was substituted for part of the plastic fat in the formula. The amounts of oil replacing the fat in the formula were equivalent to the amounts present in soybean flours containing 5%, 15%, and 22% fat.

The basic formula used was flour 100, fat 40, and water 60. Mixing, rolling, cutting, and baking conditions were controlled as closely as possible. One hundred wafers from 10 batches of each of 12 variants were broken on the Bailey shortometer, and statistical measures of the breaking strength data were used. These data are given in Table 1 and in Figure 1.

TABLE 1

The Mean Breaking Strengths of the Soy Flour Pastries and Statistical Measures.

| | Mean breaking strength | Standard deviation | Standard error of mean | Range for 100 wafers |
|---|---|---|---|---|
| Series I. Added fat constant; total fat varied | *ounces* | | | *ounces* |
| A. Defatted flour | 14.6 | 3.8 | 1.2 | 7.5-34.0 |
| B. Extracted flour, 5% fat | 11.6 | 2.2 | 0.7 | 5.0-20.0 |
| C. Expeller flour, 5% fat | 2.9 | 0.35 | 0.1 | 1.5- 5.5 |
| D. Extracted flour, 15% fat | 6.4 | 0.69 | 0.2 | 4.0-11.5 |
| E. Full fat flour, 22% fat | 6.6 | 1.07 | 0.3 | 3.5-11.0 |
| Series 11. Total fat constant; added fat varied | | | | |
| A. Defatted flour | 14.6 | 3.8 | 1.2 | 7.5-34.0 |
| F. Extracted flour, 5% fat | 15.4 | 3.6 | 1.1 | 7.0-36.0 |
| G. Expeller flour, 5% fat | 4.4 | 0.64 | 0.2 | 2.5- 8.0 |
| H. Extracted flour, 15% fat | 13.7 | 3.6 | 1.1 | 6.0-29.0 |
| I. Full fat flour, 22% fat | 19.0 | 3.5 | 1.1 | 9.5-33.0 |
| Series III. Total fat constant; proportion of oil varied | | | | |
| A. Plastic fat only | 14.6 | 3.8 | 1.2 | 7.5-34.0 |
| J. Oil as in flour with 5% fat | 15.2 | 2.9 | 0.9 | 7.5-32.0 |
| K. Oil as in flour with 15% fat | 14.3 | 3.3 | 1.0 | 7.0-28.0 |
| L. Oil as in flour with 22% fat | 12.2 | 2.9 | 0.9 | 7.0-20.5 |

When like amounts of fat were added in making pastries using extracted soy flours containing different percentages of fat, the mean breaking strength of the pastries was greatest with the defatted flour, less with extracted flour containing 5% fat, and still less with extracted flour containing 15% fat. These differences were "significant" ones. The fat in the flour seemed to be responsible for the decreased breaking strength or increased tenderness of the pastries made of extracted flours containing 5% and 15% fat.

When varying amounts of plastic fat were used in the formulas for pastries with extracted soy flours of different amounts of fat (0, 5%, 15%), adjusting the fat increments so that the total fat in the