

TABLE 3

Relationship Between Solid Glycerides Content of Hydrogenated Cottonseed Oil and Micropenetrations of the Oil

Solid glycerides, percent	Micropenetration, mm./10
44.0	20
37.5	30
33.0	40
30.5	50
28.5	60
27.0	70
26.0	80
24.0	100
21.5	125
20.0	150
17.5	200
16.0	250
15.0	300
14.0	350

TABLE 4

Estimated Solid Glycerides Content of Commercial Plastic Fat Products at Different Temperatures.

Fat	Percent solid glycerides at indicated temperatures, deg. C.				
	15°	20°	25°	30°	35°
Shortening (Spry).....	40.5	32.5	24.5	18.0	14.5
Shortening (Crisco).....	41.0	32.0	24.0	17.0	14.5
Shortening (Dexo).....	40.5	32.5	24.0	17.5	14.5
Shortening (Soco).....	45.0	39.0	28.0	21.0	16.0
Margarine oil (Parkay)...	46.0	35.0	25.5	16.0
Margarine oil (Nucoa)...	45.0	33.0	22.5	14.0

or iso-oleic acids is quite understandable, in view of the rapidity with which the consistency changes with variation in the proportion of glycerides in the solid state.

Effect of Hydrogenation on the Solid Glycerides Content of Cottonseed Oil

From the present data and previously recorded micropenetration measurements (1) on samples of a cottonseed oil hydrogenated to different degrees, it is possible to show the typical effect of hydrogenation on the content of solids in hydrogenated oils at ordinary temperatures. The data for hydrogenated cottonseed oil at 25°, 30° and 35° C. are shown in Figure 2. It will be seen that within the range of temperatures and iodine values at which the oil is plastic, reduction of the iodine value by 1 unit causes the content of solids to increase approximately 1 percent.

Summary

1. The relative amounts of solid and liquid glycerides in cottonseed oil and a hydrogenated cottonseed oil have been estimated calorimetrically at different temperatures over the melting ranges of the oils.

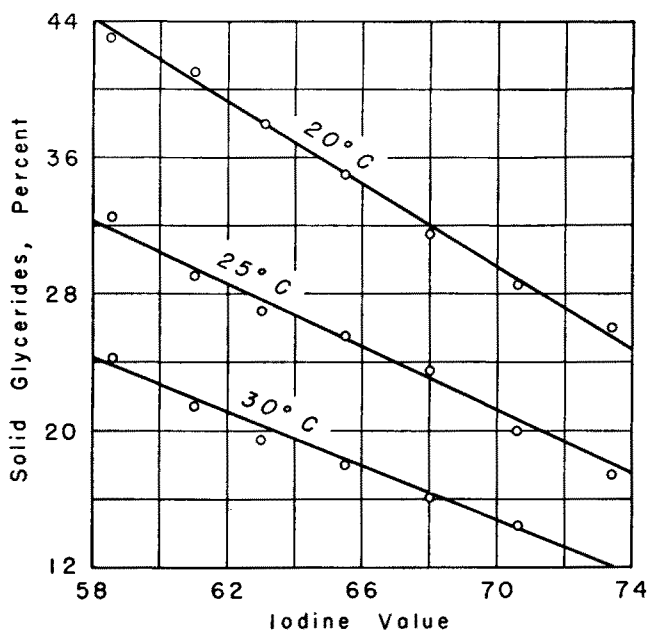


Fig. 2. Effect of hydrogenation on the solid glycerides content of cottonseed oil at different temperatures.

2. A correlation has been established between the content of solids in hydrogenated cottonseed oil and the consistency of the oil, as measured by micropenetrations.

3. From previous micropenetration data on commercial fat products, it is established that such fats are plastic and easily worked only at solids contents between about 15 and 35 percent. Differences in the content of solids of as little as 1 percent are sufficient to cause noticeable differences in the consistency of plastic fats.

4. Within the ranges of temperature and iodine value at which hydrogenated cottonseed oil is plastic, it is estimated that each decrease in iodine value of 1 unit causes the solid glycerides content of the oil to increase approximately 1 percent.

LITERATURE CITED

1. Feuge, R. O., and Bailey, A. E., *Oil & Soap* 21, 78-84 (1944).
2. Hilditch, T. P., and Jones, E. C., *J. Chem. Soc.* 1932, 805-20.
3. Jack E. L., and Brunner, J. R., *J. Dairy Sci.* 26, 169-77 (1943).
4. Oliver, G. D., Singleton, W. S., Todd, S. S., and Bailey, A. E., *Oil & Soap* 21, 297-300, Part II (1944).
5. Straub, J., and Malotau, R. N. M. A., *Rec. trav. chim.* 52, 275-88 (1933); 57, 789-94 (1938).

Abstract of Color Committee Report, 1943-44

THE work of the Color committee during the year has been confined to that done by a subcommittee consisting of Dr. R. T. Milner, R. C. Stillman, and Procter Thomson, with the exception of some color readings which were made on some special samples of oil sent out by Dr. E. B. Freyer to the whole committee.

The subcommittee has submitted a report, which, in somewhat condensed form, has been turned over to the Uniform Methods and Planning committee. As the work of the committee has not been completed and is therefore a report of progress and contains nothing that needs to be acted upon by the Society

at this time, Mr. Stillman, who prepared the report, has suggested the following abstract of the work that has been done:

"The Color Committee, during the past year, has confined itself to (1) a study of the reproducibility of the Lovibond color of a large number of oils and (2) a study of the color characteristics of 19 picked oils as is and made up into shortenings. These studies show that the Lovibond system of reading colors is certainly unreliable for measuring the color of oils widely varying in hue, nor is it improved to any great extent by the use of illuminant C as a light source. Preliminary results indicate that lu-

minous transmission correlates better with oil and shortening appearance than does Lovibond red readings. The committee believes that this work should be continued with the idea in mind that ultimately a simple photometric device could be evolved for the grading of oils."

The chairman of the committee would like to add, however, that the subcommittee has done a very large amount of valuable work which is a real contribution to the colorimetry of vegetable oils and takes this opportunity of thanking them for it.

G. WORTHEN AGEE, *Chairman*.

Melting Points of Binary Fatty Acid Mixtures C₆ to C₁₂ and Their Application in the Determination of Purity

B. J. GRONDAL and D. A. ROGERS

Lever Brothers Company, Cambridge, Massachusetts

TECHNOLOGICAL progress is creating a demand for individual fatty acids in high purity.

The literature contains considerable information on the determination of the purity of acids C₁₂ or higher, but there is a lack of data of this type for the naturally occurring acids C₆ to C₁₂. It is the purpose of this paper to present data by which the purity of these lower acids may be readily established.

Most of the physical or chemical constants of fatty acids, such as saponification value or refractive index, are valuable in themselves only in the estimation of average molecular weights or the composition of binary mixtures. In the case of binary mixtures having a high concentration of one acid, the determination of this acid by such methods lacks precision. This is truer of ternary mixtures of this type, in which case the sample must be resolved into binary mixtures as in fractional distillation.

Twitchell (1) and Wenzel (2) have demonstrated that the melting point is a valuable tool in determining the fatty acid composition of stocks of the tallow class. Schuette and Vogel (3) reported the solidification points of binary acid mixtures C₁₀ to C₂₄. Both constants are useful in assaying purity since they afford a means of determining total impurities with accuracy when present in amounts up to 20%. By either method, purity may be determined precisely by difference.

For use in analysis and control, the melting point has the advantage of faster examination of multiple samples in standard equipment, using familiar techniques. In the case of the lower acids, melting point depression is linear over a longer range of solute concentration and the determination is made at higher and more easily controlled temperatures, since mixtures of the lower acids supercool more than higher members of the series.

Data in this study were obtained by preparing samples of pure caproic, caprylic, capric and lauric acids and determining the melting points of binary mixtures of adjacent acids.

Preparation of Pure Fatty Acids

COCONUT oil fatty acids was the source from which a low-boiling fraction containing the acids C₆ to C₁₂ was separated by continuous rectification under vacuum. This fraction was separated by batch rectification into four cuts, each rich in one of the

acids. Each cut was further purified by successive vacuum distillations, using a Vigreux column until a product was obtained having the proper boiling point and a melting point not more than 0.2°C. lower than the best reported value.

Final purification was obtained by successive recrystallizations from acetone until no increase in melting point was observed. Each acid crystallized well from this solvent in white leaves or needles at -55°C. for caproic acid, -30°C. for caprylic acid and 5°C. for capric and lauric acids. Last traces of solvent were removed by heating gently under high vacuum.

The following melting points were obtained on the pure acids:

Acid	Observed M.P.°C.	Reported M.P.°C.	Refer- ence
Caproic.....	-3.4	-3.9	(4)
Caprylic.....	16.7	16.5	(5)
Capric.....	31.6	31.6	(3)
Lauric.....	44.2	44.2	(3)

The melting point of these acids is in agreement with best reported figures. It is true that Beilstein, quoting Fittig (6), gives -1.5°C. as the melting point of *n*-caproic acid, but this has not been confirmed in recent literature, and it is probable that the acid examined by Fittig is an isomeric acid, 2-3-dimethyl butanoic acid which has a melting point of -1.5°C., determined by Hommelen (4).

Melting Point Determination

THE equipment used is essentially that described by Wenzel (2). It can be assembled on short notice in any well-equipped laboratory. It consists of a one-liter Pyrex beaker three-quarters full of water or alcohol heating fluid, kept in rapid circulation by a motor-driven glass agitator. Above 0°C., water is used and the temperature is increased at the desired rate by a micro bunsen burner. For below zero temperatures, alcohol chilled with dry ice is employed and the beaker is insulated except for an observation space. Heat is absorbed from the atmosphere augmented by the micro burner. A microscope lamp provides illumination.

The thermometer has a range of -36° to 54°C., graduated in 0.2° units. Readings to less than 0.1°C. are readily made by the use of a magnifying attachment. The thermometer is calibrated in the range 0