

TABLE I  
Melting Points of Mixtures

% Hexabromide	Original Powder	Remelt
100	180.0	176.1
90	174.7	170.5
80	170.1	168.0
70	167.0	165.6
60	165.3	164.4
50	160.7	160.7
40	159.6	157.0
30	154.0	152.2
20	149.3	142.2
15	136.0	135.0
10	129.4	124.4
9	129.8	112.3
8	126.0	112.7
7	122.2	112.9
6	119.3	113.1
5	115.5	113.6
4	114.0	113.6
3	114.3	114.0
2	115.0	114.6
0	115.6	115.3

ing identical remelts on a thoroughly reground powder and on a single chip.

Some evidence was obtained for a low melting form in the case of the 10 and 15% hexabromide mixtures. Upon immersion in the bath the samples appeared to melt almost completely at 10-20° below the final melting point but quickly recrystallized and then did not melt completely until the indicated temperature was reached.

Because of the thermal instability of the hexabromide, the data reported can only be considered rather

approximate, but at least accurate enough to define the general nature of the melting point diagram of this system. In their present form the data are not of value for estimating fatty acid composition although with further study an empirical method could possibly be devised.

### Summary

1. The melting points of binary mixtures of tetrabromostearic acid and hexabromostearic acid are reported.

2. The system shows a eutectic between 9 and 10% by weight of hexabromide and at a temperature of 112°C.

3. It has been shown that it is necessary to report heating time and rate as well as melting point for hexabromostearic acid because of its extreme sensitivity to heat.

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## Report of the Committee on Analysis of Commercial Fats and Oils

### Fall Meeting, November 1949

SUBCOMMITTEE ON F. A. C. COLOR STANDARDS, E. W. Blank, chairman: At the fall meeting of the subcommittee in New York (1948) R. C. Stillman offered to forward to the subcommittee data on the spectrophotometric analysis of the F. A. C. color standards. These data, obtained by the collaboration of R. C. Stillman and A. K. Presnell, have been received by the chairman of the subcommittee. They demonstrate in a striking manner that the standards are not uniform with respect either to chromaticity or to optical density. The conclusions to be drawn from these data will be discussed at the fall meeting in Chicago.

At the meeting of the subcommittee in New York (1948) the suggestion was made that possibly the F. A. C. standards could be read through colored filters and compared on a "gray" basis, ignoring chromaticity. On the basis of preliminary experience these suggestions do not appear feasible. Residual chromaticities appear through the filters. The only way to avoid this would be to rebuild the standards, which again is not a feasible project. Work will continue on this problem.

SUBCOMMITTEE ON ANALYSIS OF DRYING OILS, J. C. Konen, chairman: During the past year the subcommittee activities have included collaboration with A. S. T. M. on the determination of hydroxyl value and diene value, a collaborative study of the effect of temperatures on the determination of iodine value,

and a check for ash on a group of samples submitted for analysis by the Smalley Foundation. In addition correspondence and discussion were carried out on methods which have been written up and are now being edited for subcommittee approval before submission to the Fat Analysis Committee. These include ash, iodine value, saponification value, and viscosity. A method written up for flash and fire point is being held in abeyance pending results of the subcommittee on Determination of Closed Cup Flash Point.

Although no new methods are being offered this year, it is anticipated that four and possibly five new methods will be submitted to the Fat Analysis Committee during the coming year.

SUBCOMMITTEE ON ANALYSIS OF LECITHIN, H. T. Iveson, chairman: During the period since this committee has last reported, the work has been confined to two projects.

#### 1. *The Determination of the Acid Values of Lecithin.*

The original method which this committee submitted for the analysis for the acid value of lecithin was objected to by certain members because of the choice of the solvent used for the KOH, which was ethyl alcohol. Two other methods have been submitted for approval, one which uses water as the solvent and another which uses isopropyl alcohol as the solvent. The committee is still in the process of checking the

advantages and disadvantages of these methods and comparing them with the proposed method. Results from some of the collaborators have been received, and there still seems to be some disagreement so that further thought will be necessary.

## 2. *The Determination of Phosphorous on Lecithin.*

When the proposed method for the phosphorous determination on lecithin was submitted, there was some discussion as to the correct temperature at which the phosphorous should be precipitated. After the chairman had investigated the objections to this method, it was thought that a slight revision in the wording would be all that would be necessary in order to complete the method. The members of the committee from whom the chairman has heard concerning this suggestion are not in entire agreement so that further discussion and possible further experimental work will be necessary before this method can be submitted. As to future plans, the incomplete work mentioned above will keep subcommittee busy for some time. It is also hoped that the spectrophotometric method for the determination of color in oils may be applied to the determination of the color of lecithin.

**SUBCOMMITTEE ON DETERMINATION OF FLASH POINT,** D. S. Bolley, chairman: A round robin test has been conducted among the subcommittee members, using linseed oil containing varying percentages of hexane and mineral spirits. The oils were tested according to a modified A. O. C. S. method, using a Pensky-Martens closed cup flash point apparatus. The results obtained by the collaborators were correlated and distributed with comments by the subcommittee chairman. Re-

sults to date indicate the proposed modified method gave good results.

A second similar round robin is now being prepared employing degummed soybean oil. When this series of tests has been completed, the effects of small amounts of moisture and other variables will be considered.

**SUBCOMMITTEE ON DETERMINATION OF THIOCYANOGEN VALUES,** F. R. Earle, chairman: The subcommittee has nothing to report this year. However, during this coming year, I should like to get a better idea of the precision of the method. I believe it would be worthwhile to send out a sample of linseed oil (to give maximum variations) and ask that it be run in quadruplicate every two months, using at least two batches of lead thiocyanate. The thiocyanogen method is being used as a control method, but I believe that the variation in results obtained at different times is much greater than is realized. Duplicates ordinarily agree well, but the level may shift with different batches of reagent or under different conditions.

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## Refining Fatty Oils With Liquid Propane \*

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THE purpose of this paper is to present a resumé of a preliminary study of the Ewing (1) process of degumming fatty oils with propane. Gums, including phosphatides and other so-called minor constituents, are precipitated from liquefied normally gaseous hydrocarbons, of which propane is the most common example. This means that the process is carried out under pressure.

Liquid propane is an intriguing solvent which has been extensively employed as a selective solvent in the refining of petroleum oils. Large commercial installations for de-waxing and for de-asphalting these oils have been in operation for the past decade. Liquid propane is also one of the two immiscible solvents employed in the Duo-Sol process of refining hydrocarbon oils. The propane dissolves the paraffinic hydrocarbons, and cresylic acid, the other solvent, takes out the naphthenic hydrocarbons. Liquid propane is cheap and very fluid at low temperatures, has a low boiling point, and can supply refrigeration through evaporation. All these properties have attracted investigators to the study of industrial applications of it. An excellent account of the use of liquid propane in the refining of lubricating oils may be found in the article by Wilson, Keith, and Haylett (2).

The solvent properties of liquid propane are unique. Unlike most common solvents which dissolve more solute as the temperature is raised, liquid propane dissolves less solute as the temperature is increased from room temperature to higher temperatures. Such anomalous behavior has prompted investigators to refer to it as an anti-solvent. As the temperature is lowered to 0°F., and below, liquid propane exhibits "normal" solubility characteristics, that is to say, the solutes become more insoluble.

Liquid propane boils at -44°F., under one atmosphere pressure; at 70°F., the vapor pressure is 126 lb./sq. in.; at 100°F., it is 190 lb./sq. in.; and at 140°F., the pressure is about 310 lb./sq. in.

Hixson and Miller (3) proposed treating tall oil with liquid propane to fractionate the rosin acids from the fatty acids. Hixson and his associates (4) have made several valuable, fundamental contributions to the knowledge of the solubility of known mixtures of pure fatty acids, abietic acid, and refined or synthetic triglycerides in the region of the critical temperature of liquid propane. Drew and Hixson (5), continuing the researches along these lines, established the limitations of the use of liquid propane as a selective solvent.

Ewing (1) was the first to advocate the refining of fatty oils with liquid propane. His process en-

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