The data obtained here extend the information on the solubilities of the fatty acids and derivatives that were studied and may be used as a guide for separation of mixtures by crystallization procedures. However, with mixtures of fatty acids in solution, the solubility of any given fatty acid is affected by the solubilizing effect of other fatty acids in the mixture, and the nature and extent of this solubilizing effect is also dependent on the nature of the solvent. Because of a paucity of data on the solubility characteristics of mixtures, it would be desirable to follow these measurements of pure individual compounds with studies of model mixtures. Nevertheless the solubility curves for the pure compounds are useful, even when fractional crystallization of mixtures is contemplated, and can be used in selecting solvents, solute concentrations, and crystallization temperatures.

Summary

An improved "synthetic" method of determining solubilities has been described which combines simplicity with accuracy.

Saturated fatty acids with even numbers of carbons from C_6 to C_{18} and oleic, linoleic, and linolenic acids, their methyl esters, their simple triglycerides, and their corresponding alcohols have been prepared in purified form. The solubilities in acetone of their most stable forms have been determined from ordinary room temperatures down to about −70°C. or to temperatures where they are only slightly soluble.

The precipitation of unstable polymorphs from solutions was observed in the case of palmityl alcohol.

Acknowledgment

The authors are indebted to Frederick J. Pusch for technical assistance in preparing some of the compounds.

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Report of the Uniform Methods Committee, 1957-58

AT A MEETING of the Uniform Methods Committee in the Peabody Hotel on April 21, 1958, the following matters were discussed and the indicated decisions were made. The meeting was attended by all members of the Uniform Methods Committee. L. H. Hodges, J. R. Mays Jr., and H. T. Spannuth were present during part of the discussion.

1. *Seed and Meal Analysis Committee, T. H. Hopper, chairman*

   Subcommittee on Sampling Bulk Meal, L. H. Hodges, chairman

   a) The committee recommends the adoption of additions to the Official Method for Sampling, Ba 1-38, to provide for the continuous sampling of bulk shipments of meal at point of origin. The automatic sampler is designed to take continuously a representative small portion of the entire cross-section of the meal at a point where the flow of meal is free-falling.

   This method is urgently needed. The subcommittee has spent several years in its development and has demonstrated its reliability. The Uniform Methods Committee concurs with their recommendation for its addition to Ba 1-38, provided more information is included on the design and source of the automatic sampler. Under "Apparatus—A 7 (a)" the following sentence should be added: "Any automatic sampler, equal or equivalent in performance, to those offered by Standard Geary-Jennings with Type C Modified Cutter, manufactured by The Galigher Company, 545 West 8th South street, Salt Lake City 4, Utah, and Automatic Sampler by Davidson-Kennedy Company, 1090 Jefferson street N. W., Box 97, Station D, Atlanta, Ga., will be satisfactory." Furthermore a drawing, showing construction of a suitable sampler, will be included in the printed method. Adopted.

   b) The Seed and Meal Analysis Committee further recommends advancement of the Tentative Method Ba 7-55, for "Free Gossypol," in cottonseed slab and sized cakes and meal, to Official status. The Uniform Methods Committee approves this proposed action. Adopted.

2. *Fat Analysis Committee, V. C. Mehlenbacher, chairman*

   Subcommittee on Analysis of Drying Oils, J. C. Konen, chairman.

   a) The Fat Analysis Committee recommends replacement of present Tentative Method, Ka 2-55 for "Acid Value," by a complete revision. This subcommittee is a joint committee with A.S.T.M., and the proposed revision contains three references to A.S.T.M. Designations for synthetic methyl alcohol, 99% isopropyl alcohol, and industrial toluol. These specifications will be placed by our editor of Methods in Section H of A.O.C.S. Methods, with the assistance and approval of the chairman of the F.A.C., and the specifications for these solvents will be referred to in the method by their appropriate number in Section H. With these changes the Uniform Methods Committee approves replacement of Ka 2-55 by this new Tentative Method. Adopted.

   b) The Fat Analysis Committee further recommends replacement of present Tentative Method, Ka 3-47, for "Color by Gardner (1933) Standard Colors," by a complete revision employing Gardner (1953) Standard Colors. Unfortunately this method also contains one reference to an A.S.T.M. Designation, which cannot be avoided without unwarranted duplication in our Methods. The caption "Composition" should be added to cover all four last columns of "Table 1. Reference Standard Color Solutions." With this slight addition the Uniform Methods Committee approves adoption of this revision of Tentative Method Ka 3-47. Adopted.

   c) At the Fall Meeting in Cincinnati, October 2, 1957, the Uniform Methods Committee approved, and the Society adopted, a "Continuous Flow Method for Sampling Tanks or Tank Cars During Loading or Unloading," as a replacement for C 1-47, D, (a), the present "Petcock Method." This was done with the understanding that certain criticisms made by the U.M.C. would be recognized and corrections made. This has been done, and no further action by the Society is necessary at this time. It is obvious however that a few changes are required in the drawing to show the following.

      1. By enlarged insert more detail on the 45° bevel end of the bleeder line, its orientation, and a somewhat more definite angle with the horizontal for the bleeder line than "a slight downward slope" should be shown.
      2. The drawing shows arrangement for sampling only while loading. To provide for sampling during unloading the drawing should be marked in appropriate places: "To Tank Car or Storage" (two places) and "From Storage or Tank Car" (one place).
      3. If, as we believe, this method is applicable to tank

trucks as well as tank cars, the title and text should so indicate.

All of these minor changes will be made by the editor of Methods with the assistance and approval of the chairman of the F.A.C.

Depending upon the length of the pipe line, through which oil is being pumped, variable pressure will develop which will affect the volume of the sample taken so that, under certain conditions, more than a 50-gal. sample may result. The Uniform Methods Committee requests the Fat Analysis Committee to investigate the following matters and recommend appropriate action to be taken in revising this method, if possible, before the next Fall Meeting.

1. Specify, within reasonable limits, the total volume of sample to be drawn, *e.g.*, 25 to 45 gal.
2. Devise some method for controlling the flow of oil through the bleeder line so as to assure a volume of oil in the drum within the limits specified. The U.M.C. suggests possible use of a number of reducing nozzles, each with a fixed outlet orifice, to be screwed on the discharge end of the bleeder line. These devices will apply only to reducing the volume of oil drawn during a normal pumping.

3. *Glycerine Analysis Committee, W. D. Pohle, chairman*

a) The Glycerine Analysis Committee recommends advancement from Tentative to Official status of the following methods:

1. Total and Organic Residue at 175°C., Ea 3-56.
2. Moisture by Karl Fischer Method, Ea 8-56.

These methods have been shown to be reliable, and the Uniform Methods Committee approves their advancement from Tentative to Official status. Adopted.

b) The Glycerine Analysis Committee has recommended additions to the following methods to show the degree of precision which can be expected in their use:

Ea 3-56; Ea 6-51; Ea 7-50; Ea 8-56; Ca 14-56, and Da 23-56.

The Fat Analysis Committee likewise recommends similar additions, for the same purpose, to Method Cd 11-57. The Uniform Methods Committee approves in principle these valuable contributions to the usefulness of these methods but, in the interests of uniformity and the best possible presentation of this information, requests that before the Fall Meeting these data be reviewed by and with our Statistical Committee and that a standard format for such presentation be formulated. In the course of this study a decision should be made on whether to use such terms as "inter" for "between," and "intra" for "in the same" laboratories. In general, data upon which conclusions are based should be submitted with the recommendation, *e.g.*, number of samples, number of laboratories, number of analysts, number of analyses, and when performed.

The Uniform Methods Committee is requesting the Statistical Committee to assist our technical committees by prescribing a standard format for expressing precision to be expected. Other A.O.C.S. Methods in which this information is presently shown should be scrutinized and, if possible, brought within the standard format. The Statistical Committee furthermore is requested to prepare a new section for A.O.C.S. Methods on a uniform method for determining the precision of an analytical method and a standard format for its expression. If possible, this project should be completed during the coming year.

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Quantitative Determination of Traces of Free Gossypol in Fats, Oils, and Fatty Acids by Paper Chromatography[1]

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COTTONSEED and cottonseed products contain the pigment gossypol. Trace amounts of this pigment, when fed to hens in rations, cause olive egg yolk discoloration. This discoloration is associated with free or labile-bound gossypol. Analytical methods which determine free or loosely bound gossypol in any such cottonseed product are of major importance.

The investigations by Pons and co-workers (8, 9) have led to the adoption of a tentative method for the analysis of gossypol in oils by the American Oil Chemists' Society (12). In this method the oil is dissolved in (4:6) hexane-isopropyl alcohol solution and reacted with p-anisidine to develop a colored complex, which is measured quantitatively by spectrophotometry. The lower limit of detection by this method is about 100 p.p.m. Since other pigments and aldehydes give similar color reactions, this method does not differentiate between gossypol and these other substances. Therefore the determined values are frequently higher than the actual gossypol content.

Smaller amounts of free gossypol (50 p.p.m.) are detected when phloroglucinol in acid solution is used for the color development (10). Since phloroglucinol is not a specific reagent for gossypol, other compounds in the sample give similar color reactions. Therefore the method is not specific. In addition, these colorimetric methods cannot be used to measure small amounts of gossypol in highly colored samples because of background absorption.

Grau and co-workers (2) developed a biological method, which measures both free and labile forms of gossypol. In this method the sample is fed to hens in a ration over a period of days. The egg yolks from the hens are extracted with acetone and then with 3:1 hexane-acetone. The absorbance of the latter extract at 400 mμ is proportional to the gossypol fed. Although this method is very sensitive, compounds other than gossypol will cause egg yolk discoloration. Therefore the method is not specific In addition, the method suffers from being time-consuming.

Because these methods lack specificity and because they do not detect trace amounts of gossypol, an improved analytical method was required. In the present work a specific and sensitive method for the determination of as little as 10 p.p.m. free gossypol was developed. The method is based on the concentration of gossypol by extraction and quantitative paper chromatography of the extract.

To separate gossypol from the bulk of the sample, a preferential extraction is necessary. Dimethylformamide-water solution extracts gossypol quantitatively from fats and fatty acids and does not form emulsions

[1] Presented at the fall meeting of the American Oil Chemists' Society, Cincinnati, O., September 30 to October 2, 1957.