this diene would each react like isolated monoenes. Therefore the 9,13-diene concentration would increase during the hydrogenation. In support of this view some 9,13-diene was detected by the chromatographic analysis of the products formed during the hydrogenation at 120°.

Added evidence for this mechanism was obtained by a determination of the geometrical configuration of the positional isomers produced during the hydrogenation. A sample of methyl eleostearate which contained approximately equal amounts of the α and β isomers was hydrogenated to an iodine value of 60. The positional isomers and the *trans*-isomer content were determined. The *trans* monoenes were isolated from the mixture by acetone crystallization, and the positional isomers in the pure *trans* were determined. The results of the analyses are shown in Table II.

TABLE II

Monoenoic Fatty Acid Composition of Partially Hydrogenated Methyl Eleostearate [a]

Double bond position	% total unsaturated (72.5% *trans*)	% *trans* fraction	% total unsaturated in *trans* form	% positional isomer in *trans* form
9	21.3	14.1	10.2	48.0
10	20.6	20.8	15.1	73.2
11	20.0	21.2	15.4	77.0
12	18.1	19.8	14.3	79.0
13	20.0	24.1	17.5	87.4

[a] 48% methyl β-eleostearate, 43% methyl α-eleostearate.

The last column in the table, which shows the percentage of each positional isomer that is *trans*, indicates that practically all the 13-octadecenoate is *trans* while the *trans* content of the 11 is somewhat less and the *trans*-9 much less. These observations are believed to be related to the geometrical configuration of the original conjugated triene. If the configurations of the α- and β-eleostearic acids as proposed by Paschke (16) are considered, it is apparent that hydrogenation of the 9 and 11 bonds of eleostearate would produce only *trans*-13. However, since the 11-monoene may be produced not only by saturation of the original 9 and 13 bonds but also from the intermediate dienes, some *cis* is formed even though the original 11 bond in the triene was *trans*. The percentage of the 9-monoene that is *trans* supports the proposed *cis*-9, *trans*-11, *trans*-13 geometrical configuration of α-eleostearic acid (16) since the 9 double bond would not undergo geometrical isomerization during the hydrogenation. The observed 48% *trans*-9 monoene is due mainly to the fact that methyl β-eleostearate was present in the original sample.

Further investigation of this proof of structure involved addition of two moles of hydrogen to a sample of α-eleostearic acid prepared by the method of O'Connor (15). The *trans*-octadecenoic acids were separated by acetone crystallization, and the positional isomers were measured. Only a very small amount of *trans*-9-octadecenoic acid was found. This procedure offers a method by which the configuration of the double bonds of a conjugated system in an aliphatic compound may be determined.

Summary

The hydrogenation of methyl eleostearate with and without solvent has been studied. The data indicate the stepwise addition of two moles of hydrogen to the conjugated triene to produce equimolar amounts of 9-, 10-, 11-, 12-, and 13-octadecenoates. Additional evidence for the *cis*-9, *trans*-11, *trans*-13 structure of α-eleostearic acid was obtained.

Acknowledgment

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Report of the Uniform Methods Committee

Business Session, Philadelphia, October 12, 1955

BOTH OF THE RECOMMENDATIONS for changes in A.O.C.S. Methods were by the Color Committee, of which R. C. Stillman is chairman.

1. The Color Committee recommends the adoption, as tentative, of a new method for the "Determination of Chlorophyll in Parts per Million in Refined and Refined and Bleached Oils." This method will be tentative and, if adopted, will be coded Cc 13d-55. The detailed procedure was published in the Journal of the American Oil Chemists' Society for September, 1955 (vol. 32, pp. 503-505). This recommendation is supported by a considerable amount of excellent cooperative work by the Color Committee.

   Approved by U.M.C. Adopted.

2. The Color Committee recommends several changes in Official Method Cc 13b-45 for "Color of Fats and Oils—Wesson Method Using Lovibond Glasses." These changes, relating to preparation of oils for color determination, are as follows:

B. Procedure: Change paragraph 1 to read:

"Crude, raw, and refined oil samples must be treated with 0.5 g. of official diatomaceous earth per 300 g. of oil. Add the diatomaceous earth to the oil and agitate for 2.5 min. at 250 r.p.m. at room temperature, or at no more than 10° to 15°C. above the melting point of the fat, if necessary, and filter through an approved paper.

"Oils which have just been bleached in the laboratory, in accordance with A.O.C.S. methods Cc 8a-52, Cc 8b-52, or Cc 8d-48, normally are sufficiently clear for the color determination. Suspended material, even if of colloidal size, will cause light scattering. If the sample is not absolutely clear, treat with official diatomaceous earth, as outlined above, and filter before proceeding with the color determination.''

Paragraph 2, under ''B. Procedure,'' will remain unchanged. Section ''a,'' under paragraph 3, with subheading ''Crude Oils of the Coconut Type,'' is changed to delete the sentences, ''Melt the oil in a water bath at a temperature not exceeding 50°C. and filter through approved filter paper at a temperature not above 35°C. If not clear, refilter once.'' These sentences are unnecessary if the requirement for treatment of all crude oils with diatomaceous earth, as proposed in paragraph 1 of ''B. Procedure,'' is adopted.

Approved by U.M.C. Adopted.

No change in the Photometric Method, Cc 13c-50, is necessary since the method as written applies only to refined, or to refined and bleached, cottonseed, soybean, and peanut oils.

Business Session, Houston, April 25, 1956

Bleaching Methods Committee, T. C. Smith, chairman

1. *Bleaching Test, Soybean Oils, Cc 8b-52*

   The committee requests deletion of Section C—Notes 1 and 2, as ''ambiguous and superfluous.''

   Approved by U.M.C. Adopted.

Fat Analysis Committee, V. C. Mehlenbacher, chairman

1. *Gossypol in Oils*

   The committee recommends adoption, as tentative, of a method developed by the Gosspyol subcommittee (C. L. Hoffpauir, chairman) of the Seed and Meal Analysis Committee.

   Since a pure gossypol is required and no source is given, the U.M.C. requests that the following note, in an appropriate location, be added to this method:

   ''On request, to A.O.C.S. headquarters, information will be supplied concerning a source for this standard.''

   Approved by U.M.C. Adopted.

2. *Spectrophotometric Determination of Conjugated Dienoic Acid*

   Adoption of this method, as tentative, is recommended by the Fat Analysis Committee. It was developed by the Drying Oils subcommittee (J. C. Konen, chairman) and is applicable to dehydrated castor oils, dehydrated castor oil fatty acids, and their methyl or ethyl esters, *only.*

   Approved by U.M.C. Adopted.

3. *Iodine Value. Sample Size for Dehydrated Castor Oil Fatty Acids, Cd 1-25, F, Note 2*

   Recommended that a provision be added to read: ''0.11 to 0.13-g. samples should be used for dehydrated castor oils and dehydrated castor oil fatty acids.'' The same note should be added, in an appropriate location, to Methods Ka 9-51 and L 8a-55.

   Approved by U.M.C. Adopted.

4. Make the Lopes Method, for checking halogen ratio, a part of all A.O.C.S. Iodine Value Methods. These are: Cd 1-25; Da 15-48; Ka 9-51; and L 8a-55.

5. Place limits on I/Cl ratio of 1.10 ± 0.1.

6. For the present the Fat Analysis Committee recommends that carbon tetrachloride be retained as the only approved solvent.

7. The use of iodine monochloride, of commercially available reagent grade, is allowed in all A.O.C.S. Iodine Value Methods. Baker or Eastman ICl reagents have been tested and found satisfactory.

   Methods of preparation of Wijs Solution from reagent iodine monochloride, and for sodium thiosulfate solution are included in the report of the Iodine Value subcommittee (R. C. Stillman, chairman).

   The U.M.C. has approved all of these recommendations with the suggestion, to the Editor of Methods, that these additions be made to one method only, Cd 1-25, and that appropriate cross-references be made in Methods Da 15-48, Ka 9-51, and L 8a-55. Needless duplication thus is avoided. The U.M.C. further recommends that the Iodine Value subcommittee continue its investigation of solvents for another year, with special emphasis on the possible use of ethyl acetate which has shown particular promise.

   The Soapstock Analysis Committee (K. E. Holt, chairman) is requested to continue the investigation of iodine value on acidulated soapstocks.

   The Iodine Value subcommittee is commended for its excellent work and clear-cut conclusions.

   Approved by U.M.C. Adopted.

8. *Oxirane Oxygen*

   Adoption of this new method, as tentative, is recommended by the Fat Analysis Committee. It was developed by the Epoxy subcommittee (K. E. Holt, chairman) and is applicable to epoxidized fatty materials. Pyridine is employed as a reagent. This substance is irritating to the eyes and mucous membranes. The U.M.C. suggests that the following warning be inserted by the Editor of Methods in an appropriate location:

   ''Pyridine is irritating to the eyes and mucous membranes. Avoid inhalation of its fumes and handle it only in a well-ventilated area.''

   Approved by U.M.C. Adopted.

9. *Moisture in Lecithin, Tentative Method, Ka 6-55.*

   The end-point in this Toluene Distillation Method is not well defined. As a temporary measure it is suggested that the second sentence in Section D-3 be revised to read:

   ''When the greater part of the water has been distilled, increase the distillation rate to *ca.* 200 drops per minute and continue until the water level in the receiver increases no more than 0.1 ml. in 30 min., or until the distillation has proceeded for two hours, whichever is the first to occur.''

   Approved by U.M.C. Adopted.

   The U.M.C. requests the Lecithin subcommittee to investigate applications of the Karl Fischer titration method to moisture in lecithin, as there is some evidence that the toluene distillation method may not be entirely satisfactory for this purpose.

   The U.M.C. further suggests to all technical committees, having distillation moisture methods under their jurisdiction, that a protection tube, or trap, of a suitable desiccant, be attached to the top of the condenser to eliminate possible errors caused by inside condensation of atmospheric moisture during analyses on days of high humidity. A poll of each committee on this matter is requested.

10. *Viscosity of Drying Oils*

    The Fat Analysis Committee recommends revision of Section C-2 to read:

    ''Express the viscosity of extremely high viscosity samples in time of bubble travel in seconds, or in stokes according to the following formula:

    $$\text{Viscosity (stokes)} = \frac{0.73\,t - 0.5}{t^2}$$

    t = time of bubble travel in seconds.''

    This will bring the A.O.C.S. method into accord with A.S.T.M. D-555.

    Approved by U.M.C. Adopted.

Glycerine Analysis Committee, W. D. Pohle, chairman

1. *Sodium Periodate Oxidation Method, Ea 6-51*

   The Glycerine Analysis Committee makes the following recommendations:

   In Section B-1a change ''reagent grade'' to read ''A.C.S. Spec.''

   Approved by U.M.C. Adopted.

2. Omit Section B-1b entirely.

   This section describes a test for the quality of the periodate reagent for use in glycerine analysis. Though it may not be necessary, if A.C.S. Specs. are met, the U.M.C. believes it should be retained for the present.

   Not approved by U.M.C.

3. In Section B-13 change ''40.84 g.'' to read ''10.21 g.'' This will change the buffer solution from 0.2 molar potassium acid phthalate to 0.05 molar and yield a solution having a pH of exactly 4.0 at 20°C.

   Approved by U.M.C. Adopted.

   The U.M.C. recommends revision of Section A-5, second sentence to read:

   ''Adjust pH meter to read pH 4.0, at 20°C. when electrodes etc. . . .''

   Approved by U.M.C. Adopted.

4. *Total and Organic Residue at 160°C., Ea 3-38*
   Replace this Official Method with a new Tentative Method, Ea 3-56, for "Total and Organic Residue at 175°C."
   The proposed tentative method yields much more precise values. The time required is shortened. The U.M.C. suggests that the words "to obtain organic residue" be added to the end of the second sentence under "Principle."
   Approved by U.M.C. Adopted.
5. *Total, Free, and Combined Glycerol, Fats, Oils, and Related Products*
   Adoption of this new, tentative method is recommended by the Glycerine Analysis Committee. The U.M.C. suggests revising the second sentence in Section D-7 to read: "Prepare two blanks exactly as described above, but with sample omitted, and from each pipette 50 ml. into a beaker containing 50 ml. of periodic acid reagent." This is to compensate for any effect of the reagents used in preparation of the sample.
   The Editor of Methods will place this new method in "E. Sampling and Analysis of Glycerine." Its applicability to fats, oils, fatty acids, soaps, etc., will be covered in the "Product-Method" and "Method-Product" lists. Some editorial changes will be necessary to bring within the standard methods format.
   Approved by U.M.C. Adopted.

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

The Seed and Meal Analysis Committee has made the following recommendations:

1. *Residual Lint on Cottonseed, Aa 7-55*
   Advance this tentative method to make it official.
   Approved by U.M.C. Adopted.
2. *Oil in Castor Beans, Ae 3-52*
   Work by the subcommittee on Analysis of Castor Beans and Pomace (V. B. Shelburne, chairman) has justified revision of this tentative method. The essential change is that commercial hexane will be used, instead of carbon tetrachloride, as extraction solvent. Substances other than oil are extracted by carbon tetrachloride.
   Approved by U.M.C. Adopted.
3. *Oil in Castor Pomace, Bd 3-52*
   Revision of this tentative method by a change of solvent from carbon tetrachloride to hexane is recommended.
   Approved by U.M.C. Adopted.
4. *H—Specifications for Reagents, Supplies and Apparatus*
   Specifications for commercial hexane for use as a laboratory extraction solvent are made necessary by the revisions of Ae 3-52 and Bd 3-52, if adopted. Suitable specifications are recommended.
   Approved by U.M.C. Adopted.
5. *Moisture and Volatile Matter in Castor Beans, Ae 2-52*
   In Section B-1 delete the words:
   ". . . unfilled beans (usually referred to as 'poppers') . . .".
   Approved by U.M.C. Adopted.

Soapstock Analysis Committee, K. E. Holt, chairman

1. *pH of Acidulated Soapstocks*
   Adoption of this new method, as tentative, is recommended by the Soapstock Analysis Committee for controlling the mineral acid remaining in acidulated soapstocks.
   Approved by U.M.C. Adopted.

Spectroscopy Committee, R. T. O'Connor, chairman

1. *Polyunsaturated Acids, Cd 7-48*
   Based on extensive and thorough investigation, a revision of this tentative method, so complete as to amount practically to its rewriting, is recommended by the Spectroscopy Committee. The method is greatly simplified and its range extended to include pentaenoic acids. It will require some editorial alteration to make it conform to the standard format.
   Tentative Method L 12a-55, for Polyunsaturated Acids in Commercial Fatty Acids, is based upon Cd 7-48. The references in L 12a-55 will require editing in order to make them conform to revised method Cd 7-48.
   Approved by U.M.C. Adopted.

Never in A.O.C.S. history have our technical committees shown greater activity. Progress reports, submitted to the Uniform Methods Committee by the chairmen of the various technical committees, indicate that additions to, and revisions of, our Official and Tentative Methods will continue to increase rapidly.

The Uniform Methods Committee wishes to thank the chairmen and members of our technical committees for their efforts which are making this steady progress possible.

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J. J. GANUCHEAU
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[Revised May 17, 1956]

Structural Studies on Sucrose Monolaurate

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THE PREPARATION of sucrose monoesters of fatty acids by alcoholysis has recently been reported (2, 3). Present studies concerning the structure of sucrose monolaurate involve inversion, tosylation, and periodate oxidation. The only detailed structural study of a sucrose monoester reported in the literature is that of sucrose 2-phosphate (1). This ester was inverted, and glucose phosphate was characterized by periodate oxidation. Consumption of periodate corresponded to the structure assigned. Formic acid production was only 87% of theoretical.

Experimental and Results

Sucrose Monolaurate. Sucrose (200 g., 0.586 mole) was dissolved in 700 ml. of dimethylformamide (DMF) containing 62.7 g. (0.293 mole) of methyl laurate (saponification value, dry, 258, theoretical saponification value 262). Sodium methoxide (4 g.) was added, and the solution was heated to 90°C. for 15 hrs. with stirring in an open beaker. The solution was then concentrated to 300 ml., cooled, and extracted for 24 hrs. in a liquid-liquid continuous extractor with hexane to remove unreacted methyl laurate. The DMF solution was then diluted with 1.5 liters of acetone. The unreacted sucrose which precipitated was removed by filtration, and the filtrate was distilled to a syrup. This syrup was redissolved in 350 ml. of boiling acetone, and the small quantity of sucrose remaining undissolved was separated by a rapid hot filtration. The acetone solution was allowed to cool slowly to room temperature. A white precipitate formed. This was separated, and the acetone filtrate was chilled to 0° to yield a second crop of precipitate. These two fractions were combined and reprecipitated from 150 ml. of acetone to give 43 g. (28% yield) of sucrose laurate, softening point

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