

It is noteworthy that these acids are constituents of the members of three families: *Compositae* (*Dimorphotheca*, *Artemisia*, *Cosmos*, and *Helianthus*), the *Leguminosae* (*Calliandra*), and the *Zygophyllaceae* (*Balanites*). This indicates that the distribution of these acids in nature may be widespread and that they may have some metabolic significance.

The tremendous potential of the method of thin-layer chromatography is demonstrated in this work since, without its aid, the detection and differentiation of two hydroxy constituents in five of the oils could not readily have been accomplished.

Our results confirm and amplify some of the findings of Smith *et al.* (4). It is considered that the method of detection of this type of acid by near infrared spectrophotometry, as described in this paper, is more specific and more sensitive than the chemical methods outlined by them. However the requirement of expensive specialized equipment may make it less desirable to some laboratories. The complementary use of GLC, thin-layer chromatography, and ultraviolet and infrared spectrophotometry provided a means for detecting and evaluating small amounts of the hydroxy substances which interfere in the determination of epoxy acids.

Summary

The interference of certain unsaturated hydroxy acids in the Durbetaki method of epoxide determination has been demonstrated. The concentrations of these constituents were determined concurrently with those of epoxy components by measurement of the near infrared spectra of samples before and after treatment with anhydrous ethereal hydrogen chloride. The individual hydroxy esters were separated and isolated from samples of mixed esters by thin-layer chromatography. GLC of these esters resulted in their alteration to conjugated trienoates and gave

proof of their conjugated diene hydroxyl structure. Thin-layer chromatographic and infrared studies verified the *trans-trans* diene unsaturation of the acid from *Dimorphotheca aurantiaca* oil and showed that the other hydroxy compounds examined have a *cis-trans* diene system. These data suggest that the seed oils of *Artemisia absinthium*, *Calliandra eriophylla*, *Balanites aegyptica*, *Cosmos bipinnatus*, and *Helianthus annuus* contain 9-hydroxy-*trans*-10-*cis*-12- and 13-hydroxy-*cis*-9-*trans*-11-octadecadienoic acids.

Acknowledgment

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We are also grateful to P. R. Edmondson and H. Dinsmore, of the Department of Medicine of this university, for performing infrared analyses, on the micro scale, of some of our products.

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Report of the Uniform Methods Committee, 1959-60

THE MEETING of the Uniform Methods Committee of the American Oil Chemists' Society was held at 2 p.m., April 5, 1960, in the Baker Hotel. The meeting was attended by six members of the committee. (S. E. Tierney had resigned.) E. W. Sallee, editor of *Methods*, was present as a member *ex officio*. Guests present were R. H. Dreyer, Edward Handschumaker, E. R. Hahn, J. C. Harris, R. A. Marmor, I. D. Metcalfe, and W. E. Link.

The following matters were discussed, and the indicated decisions were made:

1. Fat Analysis Committee, V. C. Mehlenbacher, chairman

- a) *Hydroxyl Value. Tentative Method Cd 13-60.* This new method was proposed for adoption as tentative. With some minor revisions and with the data on its precision to be calculated and given for each of the products studied in the collaborative work, the method is recommended for adoption as tentative.
- b) *Soap in Oil. Tentative Method Cc 15-60.* This new method was proposed for adoption as tentative. It was developed and checked by a subcommittee under its chairman, P. W. Morgan. Precision data have been obtained. It is recommended for adoption as tentative.

- c) *Nonvolatiles (Solids). Tentative Method Ka 14-60.* This new method was proposed for adoption as tentative. It is for the determination of nonvolatiles (solids) in solutions of natural and synthetic drying oils in organic solvents. It was developed by the drying oils subcommittee, K. E. Holt, chairman. Precision data have been obtained. This method is recommended for adoption as tentative.
- d) *Consistency-Penetration Method. Tentative Method Cc 16-60.* This new method was proposed for adoption as tentative. The procedure is an adaptation of one of the A.S.T.M. methods, made by the consistency subcommittee, N. W. Ziels, chairman. With the precision to be calculated as coefficients of variation, the method is recommended for adoption as tentative.
- e) *Solid Fat Index. Tentative Method Cd 10-57.* It was proposed that Note 5 be revised to read as follows: "Note 5—The liquid thermal expansion is basic for calculating the solid fat index. It must therefore be accurate. Repeated analyses have shown values of 0.83 to 0.85 ml./kg. to be normal for cottonseed oil, soybean oil, lard, and tallow. Lauric acid oils, such as coconut, have values of 0.85 to 0.87 ml./kg. If determined values are abnormal, the analyses should be repeated. Standard thermal expansions may be applied in routine determinations where results are used within an organization, provided they are checked periodically by actual measurement."

The Uniform Methods Committee approved the revision and recommends its adoption.

- f) *Congeal Test. Tentative Method Ce 14-59.* It was proposed that this method be amended by addition of precision data:

“(1) Two single determinations performed in one laboratory shall not differ by more than 1.52,” and

“(2) single determinations performed in two laboratories shall not differ by more than 2.38.”

The data were approved by the Uniform Methods Committee and are recommended for addition to the method.

- g) *Oxirane. Tentative Method Cd 9-57.* On the proposal of the Fat Analysis Committee it is recommended that a note be added to this method to indicate that it is not applicable to oils containing *alpha*- and *beta*-unsaturated ketones, cyclopropenes, conjugated dienols, and oxidized fats.

- h) *Advancement of Methods from Tentative to Official Status.* The Fat Analysis Committee has recommended advancing of Tentative Methods to Official Methods as follows:

| | |
|--|----------|
| (1) Neutral oil..... | Ca 9f-57 |
| (2) Flash point..... | Ce 9b-55 |
| (3) Peroxide value..... | Cd 8-53 |
| (4) Total, free, and combined glycerol..... | Ca 14-56 |
| (5) <i>alpha</i> -Monoglycerides..... | Cd 11-57 |
| (6) Moisture, commercial fatty acids..... | L 2b-57 |
| (7) Polyunsaturated fatty acids, commercial fatty acids..... | L 12a-55 |
| (8) Photometric index, commercial fatty acids..... | L 13a-57 |
| (9) Acid value, drying oils..... | Ka 2-58 |
| (10) Viscosity, drying oils..... | Ka 6-59 |
| (11) Iodine value, drying oils..... | Ka 9-51 |
| (12) Diene value, drying oils..... | Ka 12-55 |
| (13) Dienoic acid, drying oils, photometric method..... | Ka 13-56 |

The Uniform Methods Committee recommends that this action be taken.

2. Soap and Synthetic Detergent Analysis Committee, J. C. Harris, chairman

- i) *Sodium Alkylbenzene Sulfonate. Tentative Method Dd 3-60.* This new method was proposed for adoption as tentative. This method determines sodium alkylbenzene sulfonate by ultraviolet absorption measurements. With modification of the scope to indicate known interfering substances and addition of precision data with the assistance of the Statistical Committee, this method is recommended for adoption as tentative.
- j) *Neutral Oil in Alkylbenzene Sulfonates. Tentative Method Dd 4-60.* This new method was proposed for adoption as tentative. It is applicable to powders, paste, and liquid alkylbenzene sulfonates and determines the unsulfonated material remaining in the product. With a limit of its applicability in scope to sulfonates, addition of a figure to describe the syphon used, recalculation of the precision, and specification of the solvent as petroleum ether, H 2-41, this method is recommended for adoption as tentative.
- k) *Free Acid or Free Alkali. Official Method Da 4a-48.* It was proposed that the scope of this method be revised to read “applicable to soda soaps or soda soap products except those containing borax. For potash soaps see A.O.C.S. Official Method Da 5-44;” and to delete “borates” from Note 1.

The changes proposed have been approved by the Uniform Methods Committee and are recommended.

- l) The Soap and Synthetic Detergent Committee has proposed the addition of data on precision to the following methods:

| | |
|--|----------|
| (1) Copper..... | Da 31-58 |
| (2) Ester SO ₃ | De 4-59 |
| (3) Unsulfated material..... | De 8-59 |
| (4) Alcohol insoluble matter..... | De 3b-59 |
| (5) Alcohol soluble matter..... | De 3a-59 |
| (6) Moisture by distillation..... | De 2-59 |
| (7) Combined alcohols..... | De 5-59 |
| (8) Alkalinity (as NaHCO ₃)..... | De 6-59 |

The precision data presented were not in the form indicated by the procedure for determination of Precision and Accuracy of Test Methods, M 1-59. The precision data will be calculated to conform to the accepted format with the assistance of the Statistical Committee. It is recommended that the revised precision data be added to the eight methods listed above.

- m) *Sulfates. Official Method Da 22-48.* The Soap and Synthetic Detergent Committee has proposed that the Note to paragraph C, 4 be changed to read as follows:

“Note—If silicates and polyphosphates are both absent omit step 4.”

The change is recommended.

3. Additional Recommendations of the Committee

- a) that the Fat Analysis Committee hasten its organization of a subcommittee for the development of procedures for the analysis of nitrogen derivatives of fatty acids;
- b) that all technical subcommittees proceed as rapidly as possible to develop precision data on all methods and request advice and assistance of the Statistical Committee;
- c) that a committee be organized for review and revision of Section I, Physical and Chemical Characteristics of Oils, Fats, and Waxes. M. F. Lauro is a potential chairman and has an interest in the work;
- d) that the Fat Analysis Committee restudy the procedure for sampling of oil, C 1-47, Section D, to take into account static pressures resulting from long pipe-lines. For a line going “to tank car or storage” that is more than 100 feet, the back pressure built up at the sampling breeder line has been found to provide for filling a 50-gal. drum before a tank car is half-empty;
- e) that a new illustration of the refining apparatus be given in the refining loss method Ca 9a-52.

The technical committees are commended for their diligence and interest in their continued efforts for development of new and improvement of approved methods. Thanks are due all technical committee chairmen and the members of their committees who have made progress in methods research possible.

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Approved unanimously by the Governing Board, April 6, 1960

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