4. Gordon, H., and Brock, J. F., So. African Med. J., *32*, 397 (1958).
5. Kritchevsky, David, and McCandless, R. F. J., Proc. Soc. Exper. Biol. Med., *95*, 152 (1957).
6. Cox, G. E., Taylor, C. B., Cox, L. G., and Counts, M. A., Arch. Pathol., *66*, 32 (1958).
7. Shull, K. H., Mann, G. V., Andrus, S. B., and Stare, F. J., Am. J. Physiol., *176*, 475 (1954).
8. Bargeton, D., Krumm-Heller, C., and Tricaud, M. E., Compt. rend. soc. biol., *148*, 63 (1954).
9. Anitschkow, N., and Chalatow, S., Centralblatt. Allg. Path. u. Path. Anat., *24*, 1 (1913).
10. Alfin-Slater, Roslyn B., Aftergood, L., Wells, A. F., and Deuel, H. J. Jr., Arch. Biochem. Biophys., *52*, 180 (1954).
11. Swell, L., Flick, D. F., Field, H. Jr., and Treadwell, C. R. Am. J. Physiol., *180*, 124 (1955).
12. Shapiro, S. L., and Freedman, L., *ibid.*, *181*, 441 (1955).
13. Jacobsen, N. L., Zaletel, J. H., and Allen, R. S., J. Dairy Sci., *36*, 832 (1953).
14. Kritchevsky, David, Moyer, A. W., and Tesar, W. C., Arch., Biochem. Biophys., *44*, 241 (1953).
15. Kritchevsky, David, Moyer, A. W., Tesar, W. C., Logan, J. B., Brown, R. A., Davies, M. C., and Cox, H. R., Am. J. Physiol., *178*, 30 (1954).
16. Kritchevsky, David, Moyer, A. W., Tesar, W. C., McCandless, R. F. J., Logan, J. B., Brown, R. A., and Englert, M. E., *ibid.*, *185*, 279 (1956).
17. Ahrens, E. H. Jr., Tsaltas, T. T., Hirsch, J., and Insull, W. Jr., J. Clin. Invest., *34*, 918 (1955).
18. Kinsell, L. W., and Michaels, G. D., Am. J. Clin. Nutrition, *3*, 247 (1955).
19. Kelsey, F. E., and Longenecker, H. E., J. Biol. Chem., *139*, 727 (1941).
20. Clement, G., Clement, J., and Louedec, A., Arch. sci. physiol., *8*, 233 (1955).
21. Swell, L., and Treadwell, C. R., J. Biol. Chem., *212*, 141 (1955).
22. Siperstein, M. D., Jayko, M. E., Chaikoff, I. L., and Dauben, W. G., Proc. Soc. Exper. Biol. Med., *81*, 720 (1952).
23. Hellman, L., Rosenfeld, R. S., Insull, W. Jr., and Ahrens, E. H. Jr., J. Clin. Invest., *36*, 898 (1957).
24. Gordon, H., Lewis, B., Eales, L., and Brock, J. F., Nature, *180*, 932 (1957).
25. Newburgh, L. H., and Clarkson, S., Arch. Int. Med., *31*, 653 (1923).
26. Meeker, D. R., and Kesten, D., Arch. Pathol., *31*, 147 (1941).
27. Freyberg, R. H., Arch. Int. Med., *59*, 660 (1937).
28. Nikkila, E. A., and Ollila, O., Acta Pathol. Microbiol. Scand., *40*, 177 (1957).
29. Kokatnur, M., Rand, N. T., Kummerow, F. A., and Scott, H. M., J. Nutrition, *64*, 177 (1958).
30. Leveille, G. A., and Fisher, H., Proc. Soc. Exper. Biol. Med., *98*, 630 (1958).
31. Stamler, J., Pick, R., and Katz, L. N., *Circulation* Res., *6*, 447 (1958).
32. Grant, W. C., and Fahrenbach, M. J., Fed. Proc., *16*, 50 (1957).
33. Moyer, A. W., Kritchevsky, David, Logan, J. B., and Cox, H. R., Proc. Soc. Exper. Biol. Med., *92*, 736 (1956).
34. Olson, R. E., Vester, J. W., Gursey, D., Davis, N., and Longman, D., Am. J. Clin. Nutrition, *6*, 310 (1958).
35. Mann, G. V., Andrus, S. B., McNally, A., and Stare, F. J., J. Exper. Med., *98*, 195 (1953).
36. Fillios, L. C., and Mann, G. V., Metabolism, *3*, 16 (1954).
37. Portman, O. W., and Mann, G. V., J. Biol. Chem., *213*, 733 (1955).
38. Portman, O. W., Lawry, E. Y., and Bruno, D., Proc. Soc. Exper. Biol. Med., *91*, 321 (1956).
39. Kritchevsky, David, Grant, W. C., Fahrenbach, M. J., Riccardi, B. A., and McCandless, R. F. J., Arch. Biochem. Biophys., *75*, 142 (1958).
40. Gustafsson, B. E., Bergstrom, S., Lindstedt, S., and Norman, A., Proc. Soc. Exper. Biol. Med., *94*, 467 (1957).
41. Lindstedt, S., and Norman, A., Acta Physiol. Scand., *38*, 129 (1956).
42. Nieman, C., Bacteriol. Rev., *18*, 147 (1954).
43. Camien, M. N., and Dunn, M. S., Arch. Biochem. Biophys., *70*, 327 (1957).
44. Rosenheim, O., and Webster, T. A., Biochem. J., *37*, 513 (1943).
45. Rosenfeld, R. S., Hellman, L., and Gallagher, T. F., J. Biol. Chem., *222*, 321 (1956).

[Received April 22, 1958]

Report of the Uniform Methods Committee

AT A MEETING of the Uniform Methods Committee, in the Sherman hotel, Chicago, on October 20, 1958, the following matters were discussed and the indicated decisions were made. The meeting was attended by six of the seven members of the Uniform Methods Committee. E. M. Sallee, who recently succeeded T. H. Hopper as editor of the methods, was present by invitation as a member *ex officio*. President J. C. Konen was present for part of the meeting.

1. **Color Committee—R. C. Stillman, chairman**

A.O.C.S. Official Method Cc 13b-45

This method for color of oils and fats by the "Wesson Method Using Lovibond Glasses," has been revised to require calibration of the glasses to conform to the National Bureau of Standards N" scale, or they may be compared against a standard set so calibrated. A complete technical explanation of this decision was published in J.A.O.C.S. for March, 1958 (Vol. *35*, pp. 134–135).

It was suggested that the correct addresses of the Electrical Testing Laboratories and the National Bureau of Standards, by whom the calibration may be performed, be retained in the revised method. Also a source and designation of a paint for the interior of the color booth, which will meet the required Munsell value of 4/, should be included in the method.

With these additions the U.M.C. recommends that the proposed revision be adopted. The method will retain its official status. *Adopted.*

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

a) *A.O.C.S. Tentative method Ca 2e-55*
Moisture—by Modified Karl Fischer Reagent

At the time of its adoption, several differences between this method, for moisture in oils and fats, and a similar method, Ea 8-56, for moisture in glycerine, were noted; and, in the interest of uniformity of reagents and apparatus, the appropriate committees were requested to explore the possibility of revisions to bring them into more perfect accord. C. L. Hoffpauir, chairman of the moisture subcommittee, has effected the desired revision in a commendable manner. After consultation with the chairman of the Fat Analysis Committee, the U.M.C. decided to include a magnetic or mechanical stirrer in "A-6 Apparatus" and require the use of such a stirrer in the procedures for calibration of iodine-methanol solution and for analysis of samples. Titration in a closed system already is prescribed in the method.

With this change the U.M.C. recommends adoption of this proposed revision. The method will remain "Tentative." *Adopted.*

b) *A.O.C.S. Official Method Cc 12-41, Titer Test*

This method has been revised to require the temperature of the air bath around the sample to be controlled by immersion in a liquid bath maintained at 15° to 20°C. below the expected titer point. An improved dry ice-ethylene glycol bath is employed for samples of titer below 35°C. The change in bath temperature, from the present 20° ± 1°C. for samples of titer 35°C. or higher, has been shown to be without effect on samples of fats of titer from 38°C. to 55°C., and even higher. The proposed revision has been delayed in order to enable these comparisons to be made.

The U.M.C. approves this proposed revision and recommends its adoption. The method will retain its official status. *Adopted.*

c) **Subcommittee on Analysis of Commercial Fatty Acids—J. L. Trauth, chairman**

1) *Rosin Acids in Fatty Acids, L 14a-58*

A new method, for determination of rosin acids in commercial fatty acids, is proposed. In principle it is similar to Da 12-48, but sulfuric acid is substituted for naphthalene-beta-sulfonic acid in the esterification with methanol.

A few minor additions have been made to amplify specification of apparatus and reagents. In calculation of the percentage of rosin acids, the subtraction of the correction "0.74" is made mandatory instead of implied, as indicated in "Note 1." In the standardization of alcoholic potassium hydroxide, a reference to A.O.C.S. Spec. H 12-52 is added.

With these few changes, which have been accepted by the committee, the U.M.C. approves adoption of this new method as "Tentative." *Adopted.*

2) *Heat Stability Test, L 15a-58*

The proposed method measures the stability of fatty acids under specified conditions. With the approval of the committee, certain additions have been made to the apparatus specifications, and a requirement is made that color be determined by the A.O.C.S. Tentative Method Ka 3-58, using 1953 Gardner Color Standards. This instruction is of a temporary nature until the committee can determine and advise their preferred method for color measurement. All development work on this method was performed by use of the Gardner Color Standards.

The Uniform Methods Committee approves and recommends adoption of this method as "Tentative." *Adopted.*

3) *Titer Test, L 6a-55*

Action upon a proposed revision of this method was deferred until the annual meeting next spring in order to permit more time for incorporation into it of certain items of apparatus which are dependent upon the adoption of the proposed revision of Cc 12-41, as recommended above. This deferment will not delay the issue of this method in printed form so it will be included with the 1949 revisions.

The Statistical Committee, W. E. Link, chairman, has been active in carrying out the requests made of it by the Uniform Methods Committee following the last annual meeting. By the end of this year we hope to have a standard method for evaluating the precision of any analytical method, and a standard format for expressing it. A number of committees have submitted such data, but their addition to the appropriate methods is being delayed temporarily in order to bring them within the desired uniformity of expression.

J. J. GANUCHEAU T. H. HOPPER
D. L. HENRY R. J. HOULE
K. E. HOLT T. C. SMITH
J. T. R. ANDREWS, chairman

Ricinelaidic Acid and Methyl Ricinelaidate. Their Preparation and Determination by Infrared Spectroscopy

MARIAN A. McCUTCHON, R. T. O'CONNOR, ELSIE F. DuPRE, L. A. GOLDBLATT, and W. G. BICKFORD, Southern Regional Research Laboratory,[1] New Orleans, Louisiana

IN CONNECTION with other work in progress at this laboratory, an analytical procedure for the determination of ricinelaidic acid (*trans*-12-hydroxy-9-octadecenoic acid) and its methyl ester in elaidinization mixtures was required, and, for this, authentic samples of these pure compounds were necessary. Infrared absorption procedures for the determination of *trans* isomers (8, 18, 20) have proved satisfactory for elaidic acid and methyl elaidate, but characteristics of the spectra of ricinelaidic acid and methyl ricinelaidate necessitated modifications of the equations.

The purpose of this paper is to present a simplified method for the satisfactory preparation of ricinelaidic acid and its methyl ester, a procedure for their determination by means of infrared absorption spectra and data concerning the composition of the equilibrium mixture of methyl ricinelaidate-methyl ricinoleate. Characteristic features of the infrared spectra as additional criteria of purity of the *trans* isomers and a mechanism for the elaidinization are discussed.

Experimental

Starting Materials

Castor Oil. The castor oil employed in this investigation had the following characteristics:

I.V., 85.3; sap. value, 180.1; acid value 10.4;% hydroxyl, 5.2; $[\alpha]^{23/D} + 4.46$; $n^{25/D}$ 1.4784.

Methyl Ricinoleate. This ester was prepared by alcoholysis of castor oil, followed by fractional distillation of the crude esters (19). The methyl ricinoleate obtained had the following characteristics:

Anal.: calcd. for $C_{19}H_{36}O_3$: C, 73.02; H, 11.61; % hydroxyl, 5.4; sap. equiv., 312.5; I.V., 81.2.
Found: C, 72.69; H, 11.65; % hydroxyl, 5.2; sap. equiv., 312.0; I.V., 81.8; $[\alpha]^{25/D} + 5.00$; $n^{25/D}$ 1.4609.

Ricinoleic Acid. Ricinoleic acid was obtained through saponification of methyl ricinoleate, followed by low-temperature crystallization of the recovered acid (4). The acid had the following characteristics:

Anal.: calcd. for $C_{18}H_{34}O_3$: C, 72.43; H, 11.48; % hydroxyl, 5.7; I.V., 85.0.
Found: C, 72.18; H, 11.39; % hydroxyl, 5.8; I.V., 84.8; $n^{25/D}$ 1.4710.

Preparation of Methyl Ricinelaidate

The *cis* to *trans* isomerization of the ethylenic bond of methyl ricinoleate was effected in the following manner. To 150 g. of methyl ricinoleate, maintained at 60°C., were added with rapid stirring 10 ml. of 2M sodium nitrite solution and 6.66 ml. of 6M nitric acid. The heating and stirring were continued for 3 hrs., after which time the reaction mixture was poured into 500 ml. of water contained in a separatory funnel. The elaidinized esters were extracted with ether, washed with distilled water until free of acid, and finally dried over sodium sulfate. The product recovered from the solvent weighed 142 g. and contained *circa* 76% methyl ricinelaidate as determined spectrophotometrically. The methyl esters were dissolved in 690 ml. of pentane-acetone (5:1) and allowed to crystallize at −20°C. over-night. After filtration, the crystals were washed with 100 ml. of cold pentane and dried *in vacuo*, yield 82.9 g. This material was dissolved in 500 ml. of acetone-water (9:1), treated with a decolorizing carbon, filtered, and allowed to crystallize at −20°C. over-night. The solution was filtered, and the crystals were washed with 50 ml. of cold acetone. After drying *in vacuo*, there were obtained 66.5 g. of methyl ricinelaidate, m.p. 28.9–29.8°C.

Anal.: calcd. for $C_{19}H_{36}O_3$: % hydroxyl, 5.4; sap. equiv., 312.5; I.V., 81.2.
Found: % hydroxyl, 5.7; sap. equiv., 312.8; I.V., 81.4 $[\alpha]^{23.4/D} + 4.35$ (35% solution in acetone); $n^{60/D}$ 1.4470; absorptivity, a = 0.459 at 10.28μ in CS_2.

---

[1] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.