model of the countercurrent distribution apparatus. The immiscible solvent pair was 90% aqueous ethanol and hexane-pentane.

Separation of oxidized material from unoxidized esters was again readily achieved as shown in Figure 5. Unoxidized esters present in tubes 11 through 23 amounted to 44.4% of the starting material. The remaining 55.6% was converted to the oxidation products found in tubes 0 through 10 and must average 2.02 moles oxygen per mole ester. By spectrophotometric analysis it was determined that the conjugated material in the first 11 tubes represented 49% of the methyl linolenate in these tubes. The ratio of diene to triene conjugation was 5.07. It is of interest to compare Figures 5 and 2. Despite the higher level and temperature of oxidation of experiment III there is a marked similarity of the two figures. The similarity is further emphasized in Table IV, where some of the important characteristics of the oxygenated fractions of experiments I, II, and III are summarized.

Summary

Methyl linolenate, oxidized to levels between 0.1 and 1.1 mol oxygen per mol ester, has been fractionated by countercurrent distribution techniques with aqueous ethanol and hexane as solvents.

The course of autoxidation of methyl linolenate differs significantly from that found for methyl oleate (reported in II of this series) and also from that reported for methyl linoleate.

Four major components are apparent from the weight distribution and ultraviolet absorption curves. They are, in order of decreasing polarity:

A. An alcohol-soluble component, approximately 40% of which is conjugated, with triene and diene conjugation present in about equal proportions.

B. An alcohol-soluble material, approximately 60% of which is diene conjugated, with triene conjugated substances being negligible.

C. A hexane-soluble oxidation product containing almost exclusively diene conjugation and occurring only at low levels of oxidation.

D. The unoxidized, hexane-soluble methyl linolenate.

It is concluded from the analytical studies that:

1. Less than half of the linolenate actually oxidized is converted to a conjugated form while more than half of the double bonds originally present are destroyed, probably through polymerization initiated by oxidative attack on the ethylenic bonds.
2. A partial separation of compounds containing conjugated triene (A) from diene (B) can be effected by countercurrent fractionation.
3. Compound C is apparently a precursor of components A and B.
4. Monomeric monohydroperoxide, if present, exists in very small quantity.
5. Dimers are formed during the oxidation process or immediately thereafter.

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Report of the Referee Board, 1950-1951

FOR the year ending May 31, 1951 33 Referee Chemists were appointed. All except two were renewals. Two were appointed for the first time. Twenty-four were given certificates on cottonseed oil cake and meal and fatty oils. Nine held restricted certificates either from choice of application or from discretion of the Board.

The chemists represent 11 states, 21 cities, and 25 different laboratories. To date no chemist has qualified for certification in the North Central States. While the Board feels that this is unfortunate, we also feel that our requirements are reasonable and a sustained and planned effort on the part of chemists in these areas to meet these requirements would result in certification.

The Referee Board was the official sponsor of a special set of four samples of refined cottonseed oil and four samples of refined soybean oil for collaborative tests by the new spectrophotometric color method. These samples were planned primarily as practice samples for the benefit of the Referee Chemists and prospective applicants for referee certification, but they were offered to and requested by nearly all collaborators on crude vegetable oils. This was essentially a service to the Referee Chemists and did not imply or constitute responsibility of the Board for this or any other specific A.O.C.S. method.

Considerable progress was made by A. S. Richardson in his final period as chairman of the Board to clarify and establish the policies and requirements of the Board.

The Board has strongly adhered to the policy of requiring that new applicants meet certain formal educational requiremens.

Many inquiries were received during the year relative to certification and handled in an expeditious manner.

The Board strongly urges any prospective applicants to participate in the Smalley check sample work even though an application for certification is not contemplated in the immediate future.

Applications for certification for 1951-52 already have been given some consideration, and they will be handled before May 31, 1951.

R. R. KING
J. P. HARRIS
J. R. MAYS, JR.
A. S. RICHARDSON
R. W. BATES, chairman