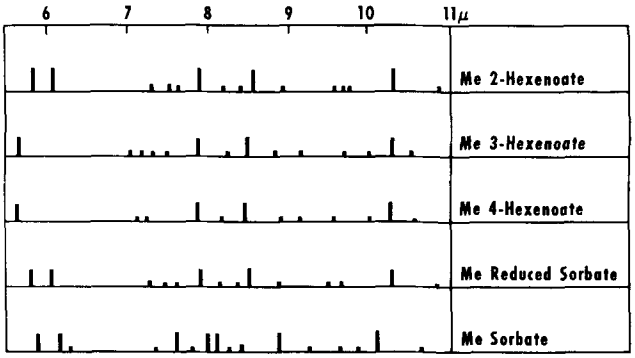

FIG. 9. Schematic presentation of absorption bands of methyl esters of 2-,3-, 4-hexenoic, reduced sorbic, and sorbic acids.

acid, the high degree of selectivity does encourage further study of homogeneous catalysis on higher fatty acids.

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Report of the Instrumental Techniques Committee, AOCS, 1962-1963

THE INSTRUMENTAL TECHNIQUES COMMITTEE held its inaugural meeting during the first International Convention of the Society in Toronto, Canada. The Committee Meeting was held in the Tudor Room of the Royal York Hotel as part of the 36th Fall Meeting, on October 1, 1962. Thirteen members and guests, including the Chairman and Chairmen of all Subcommittees, attended this first meeting.

The general organization of the New Instrumental Techniques Committee was reviewed by the Chairman. The Committee at present consists of three Subcommittees: 1) Gas Chromatography, a joint Subcommittee with the Association of Official Agricultural Chemists, consisting of twenty members with E. M. Sallee, Chairman; 2) Spectroscopy Subcommittee, with thirteen members and R. R. Allen, Chairman; and 3) Color Subcommittee with nineteen members and W. T. Coleman, Chairman. In addition, the Instrumental Techniques Committee has an active Special Task Group for the Preparation of Methyl Esters under J. R. Chipault, Chairman, consisting of nine members.

At this initial organizational meeting it was agreed that at each Fall Meeting of the Society a general meeting of the Instrumental Techniques Committee would be held. This meeting, to which all members of the Committee would be urged to attend and all members of the Society are invited, is to consist of more or less executive reports of the Subcommittee Chairmen as to progress made since the last Annual Meeting of the Society and their plans for the immediate future.

At each Annual meeting of the Society, each Subcommittee will conduct an individual meeting under the direction of its respective Subcommittee Chairman. At these sessions, details of the progress of the Subcommittee during the past year will be reviewed and plans and suggestions for future activities will be considered. These meetings will be followed by a meeting of the Instrumental Techniques Committee as a whole to be held later during the Annual Spring Meeting.

In accordance with the decisions at the inaugural meeting of the Instrumental Techniques Committee, each of the Subcommittees met during the 54th Annual Meeting of the Society at the Biltmore Hotel in Atlanta, April 22–24, 1963, and these individual Subcommittee meetings were followed by a meeting of the entire Committee.

Color Subcommittee

The Color Subcommittee, with Chairman W. T. Coleman presiding, met April 21, at 2:00 p.m. Only four members were in attendance. The Subcommittee has only one pending project, possible specifications and methods for surface color. Considerable subcommittee effort has been expended on a study of this problem. It was decided that the next step in this project is the poll of the industry for an indication of the needs and interest so that any ex-

TABLE I
Menhaden oil methyl esters
Sample 13

Lab	14:0	16:0	16:1	18:0	18:1	18:2	18:3 20:1	18:4 20:2	22:0	20:4 22:1	20:5	22:6
1	7.5	26.0	14.5	5.0	22.3	5.0	2.3	4.2	2.0	–	–	–
2	9.9	35.3	15.3	4.4	15.9	–	–	–	2.0	–	11.5	–
3	5.6	28.6	17.8	4.8	21.7	3.9	3.0	4.1	1.0	–	9.4	2.1
4	7.9	34.4	15.1	6.2	23.0	1.7	2.0	0.8	1.9	3.0	–	–
5	6.9	26.6	11.9	4.5	21.0	4.6	3.2	3.8	–	–	10.2	2.4
6	6.5	28.3	13.0	5.9	20.8	4.5	3.6	4.3	–	–	8.9	1.6
7	5.8	43.5	11.1	4.4	15.6	1.5	0.8	–	1.0	2.8	7.4	–
8	6.5	26.1	13.6	4.7	20.1	4.1	2.6	4.4	0.3	0.6	10.9	2.7
9	–	–	–	–	–	–	–	–	–	–	–	–
10	7.2	19.2	13.2	3.6	18.5	4.8	–	7.2	–	3.2	17.8	–
11	6.3	22.6	12.3	3.6	21.6	1.4	3.4	4.8	–	0.7	12.3	3.5
12	7.3	26.8	13.1	5.5	19.3	2.1	1.0	1.3	–	3.8	9.7	2.9
13	6.1	24.1	11.9	4.2	20.1	4.0	3.1	5.9	1.1	0.7	10.1	–

TABLE II
Castor oil methyl esters
Sample 14

Lab	16:0	18:0	18:1	18:2	18:3	18:1,OH
1	11.0	9.5	25.3	37.6	4.9	–
2	–	–	–	–	–	–
3	1.2	1.0	3.6	5.3	0.8	87.8
4	14.0	8.1	32.9	45.0	–	–
5	1.2	1.0	3.7	5.1	1.4	86.8
6	3.4	4.1	10.9	17.3	6.8	57.7
7	1.7	2.2	5.3	7.0	1.5	82.3
8	1.4	1.1	4.3	6.4	1.1	85.9
9	–	–	–	–	–	–
10	6.3	4.0	8.0	18.7	8.0	45.3
11	1.5	1.4	5.0	7.3	1.4	83.4
12	1.1	0.9	2.8	4.1	–	90.1
13	0.9	0.7	2.4	3.3	–	87.3

TABLE IV
Tall oil methyl esters
Sample 16

Lab	18:0	18:1	18:2	18:3 conj. 18:2 CT	20:1	conj. 18:2 TT
1	2.6	51.2	34.4	2.6	5.0	3.9
2	–	58.5	36.0	2.1	1.7	1.6
3	2.3	55.4	35.9	1.5	2.7	1.6
4	0.8	4.2	60.4	16.3	9.4	6.9
5	1.3	56.5	36.8	2.2	2.4	1.0
6	2.6	55.6	34.8	2.2	2.4	2.4
7	1.9	63.7	28.7	2.4	2.0	1.3
8	1.8	56.4	37.6	1.1	1.5	1.6
9	1.6	55.5	36.1	2.1	2.3	1.5
10	4.5	49.0	37.4	9.0	–	–
11	2.6	55.0	36.3	2.4	2.3	1.3
12	2.5	54.1	35.9	3.4	2.0	1.5
13	2.4	52.3	35.7	2.6	2.4	1.9

perimental outline developed can be kept on a practical basis. Further activity on this project will await the outcome of this poll (See JAOCS, *39*, 40, 1962).

Another possible pending problem discussed was the need for a revision of the photometric color method to improve its sensitivity. If the industry through the NCPA should vote to change its rules and adopt a bleached color basis for the settlement of the value of cottonseed oil, the present methods for color evaluation—either the subjective tintometer method (Cc 13b-45) or the objective spectrophotometric methods (C_6 13c-50)—would not, as written, be sufficiently sensitive for adequate measurements of the very light colored oils which would be involved in cottonseed oil transactions. It was decided, however, that any action required by the Color Subcommittee was contingent upon an adoption of a bleached color basis to replace the present refined color basis and that no action should be taken by the Subcommittee until such a change in rules had in fact been made by the NCPA.

Gas Chromatography Subcommittee

The Gas Chromatography Subcommittee met April 22. Twenty-two members and guests attended the meeting presided over by Subcommittee Chairman E. M. Sallee. Results of the collaborative analysis of samples of menhaden, castor, dehydrated castor, and tall oil methyl esters were discussed. Results obtained by the various collaborators are given in Tables I–IV. Despite the poor results from some laboratories unfamiliar with the types of esters analyzed, most laboratories did well. It is considered reasonable to assume that anyone analyzing a material in this work would have some idea of its composition. Future samples submitted for collaborative work will be accompanied by identification of the major components. If results reported include relative retention times, it will be easier to compile and evaluate results. After discussing the results, it was agreed that some changes in the method (AOCS Ce 1-62) were warranted.

Sample 13. Menhaden oil methyl esters. Menhaden oil is one of the most complex fish oils as well as the most important commercially. Peak identification was the greatest problem encountered by the collaborators and this problem was not resolved by those present at the meeting. Attempts will be made to get some help from the U. S. Bureau of Fisheries.

Samples 14 and 15. Castor oil and dehydrated castor oil methyl esters. The method appears applicable to these samples if the analyst recognizes that ricinoleic is present and waits for it to elute. It was pointed out that oxygenated compounds such as hydroxy acids show a reduced area response on polyester columns directly proportional to the length of the column or retention time. The possibility was

TABLE III
Dehydrated castor oil methyl esters
Sample 15

Lab	12:0	16:0	18:0	18:1	18:2	18:3 conj. 18:2 CT	20:1	conj. 18:2 TT	18:1,OH
1	0.9	2.1	5.1	44.4	1.5	19.8	–	–	–
2	–	–	–	–	–	–	–	–	–
3	0.5	1.1	1.1	4.3	48.6	22.4	6.4	12.0	3.8
4	0.6	1.4	1.1	4.9	52.9	16.9	10.9	11.3	–
5	–	1.3	1.2	4.1	51.1	22.4	6.8	12.6	tr.
6	–	1.3	1.2	4.5	50.1	22.5	3.5	15.0	1.5
7	0.5	1.3	1.5	4.6	51.5	19.8	8.6	12.2	–
8	0.5	1.0	1.1	4.2	48.7	22.8	3.7	17.9	–
9	0.5	1.1	3.0	7.5	52.0	20.1	5.1	10.8	5.0
10	–	2.7	3.3	8.2	33.0	19.7	11.5	16.5	–
11	0.4	1.3	1.2	4.3	48.6	24.0	7.1	13.1	–
12	0.3	1.2	1.0	3.9	44.3	20.5	9.0	13.5	4.7
13	0.5	1.3	1.1	4.1	49.5	21.4	6.7	13.0	–

mentioned that this might be an actual loss due to interesterification with the column packing.

Sample 16. Tall oil methyl esters. It was voted to delete the prohibition against tall oil from the scope of method Ce 1-62. Other changes approved were to extend the range of the permitted column lengths to 4–10 ft and to extend the fatty acids chain lengths included to 8–24 carbon atoms. It is recognized that a single sample with such a wide range can not be accurately analyzed using isothermal techniques but a single commercial fatty acid does not contain major components of the entire molecular weight range. These proposed changes were presented to the parent Instrumental Techniques Committee and to the Uniform Methods Committee.

The Gas Chromatography Subcommittee was organized as a joint AOCS-AOAC effort and the method is now being read into AOAC standards. It has also been copied on a letter ballot under ASTM Committee D-1 and seems likely to be accepted for fatty acid analysis by the ASTM. It thus appears that the goal of a uniform method in the various technical societies is being realized.

Future work of the Committee will include checking the applicability to other commercial fats and fatty acids as they are suggested. In the immediate future, collaborative analysis of peanut oil methyl esters to check the performance of individual collaborators with the analysis of a relatively simple sample, and re-examination of tall and menhaden oil methyl esters will be attempted. When the Special Task Group for the Preparation of Methyl Esters completes it work and makes specific recommendations, its suggested method will be tested in conjunction with the Gas Chromatographic method. The Fatty Nitrogen Derivatives Sub-Committee of the Industrial Oils And Derivatives Committee is working on a gas chromatographic procedure. When it is ready the Gas Chromatography Subcommittee will help in final cooperative testing.

Spectroscopy Subcommittee

The Spectroscopy Subcommittee met April 23, with fifteen members and guests present, presided over by Subcommittee Chairman, R. R. Allen. Since they had no pending projects, the meeting consisted of discussions of tasks which the Subcommittee should consider. It was decided that no purpose could be gained from further collaborative investigation of near IR methods for determination of hydroxyl groups as these methods are limited to the determination of a primary OH group in samples containing no secondary hydroxyl groups. (See Report of the Spectroscopy Committee 1961–62.) The group also agreed that no collaborative work should be undertaken on the near IR method for determination of isolated *cis* content as this value can be obtained by the official method for isolated *trans* (Cd 14-61) and iodine value, by difference. Similarly a proposal to investigate the determination of *cis, cis,* methylene interrupted dienes in fats by conjugation with lipoxidase was not accepted because of very little interest in this method.

A small absorption peak at 10.3 microns due to glyceride oils which contain no *trans* isomers but which give an apparent *trans* content of 1–3% was discussed and it was decided that an average correction should be worked out and considered for possible inclusion in the method for the determination of *trans* content of fats and derivatives. (Cd 14-61).

The subcommittee voted not to attempt any further collaborative work until a need for a specific method was presented.

Special Task Group for Preparation of Methyl Esters

This Task Group met with the Spectroscopy Subcommittee. J. R. Chipault presided at the portion of the meeting dealing with discussions of the activities of the Task Group.

This Task Group has obtained a triglyceride sample containing approximately 25% elaidate and 40% ricinoleate and prepared mixed fatty acids. Portions of the materials were sent to seven collaborating laboratories for preparation of methyl esters from the fatty acids by the methanol:sulfuric acid and the methanol:BF_3 methods and from triglycerides by the methanol:sulfuric acid procedure. Only three collaborators had reported by the time of the meeting. Despite the fact that only limited results were available and those reporting felt that the hydroxyl determination gave a poor analytical constant and that the determination of *trans* unsaturation in glycerides gave a value that was too high because of a small absorption at 10.33 microns by all triglycerides, the data available indicated that the esterification procedures used had little or no effect on the hydroxyl or the *trans* contents of the samples.

Plans for the coming year include the preparation of methyl esters by the H_2SO_4 and BF_3 methods from beef fat and beef fatty acids.

Instrumental Techniques Committee

The entire Instrumental Techniques Committee met April 24, following the individual meetings of the Subcommittees. Results of the Subcommittee meetings were reviewed and plans for future activities discussed. Recommendations of the Gas Chromatography Subcommittee regarding specific changes in method (Cc 1-62) were approved by the Instrumental Techniques Committee and moved on to the Uniform Methods Committee for final action.

E. M. Sallee
R. R. Allen
W. T. Coleman
J. R. Chipault
R. T. O'Connor, Chairman

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• *Erratum*

JAOCS 40, page 593, October, 1963. STOCKBURGER AND BRANDNER: THE REACTIONS OF ALKYLENE OXIDES WITH VARIOUS BUTYL AND OTHER ALCOHOLS. Equations F and H should be shown as:

$$ROCH_2CH_2O^- + CH_2CH_2 \xrightarrow{k^-_2} RO(CH_2CH_2O)^-_2 \qquad [F]$$

$$RO(CH_2CH_2O)^-_{x-1} + CH_2CH_2 \xrightarrow{k^-_x} RO(CH_2CH_2O)^-_x \qquad [H]$$