vestigated to evaluate the possibility of removing the solvent by adding water to the extract. After mixing and separating the components into two layers, each layer was analyzed for DMSO, water and acids.

The fatty acids were considered as a whole to be a single component for convenience, since practically all the acids separated in one layer, which also contained some DMSO and water. The other layer consisted mostly of DMSO and water, with only a small amt of acids.

In actual continuous countercurrent extraction, the water in the solvent should be removed before the solvent is recycled because the presence of water will greatly reduce the solubility of fatty acids. It is recommended that the water remaining in the solvent should not be more than 2%.

In determining the separation factors of linoleic acid, all experiments were carried out at room temp, since temp appeared to have little effect on the separation factor in this particular system studied.

Analytical Methods. The fatty acids were analyzed directly by gas-liquid chromatography on an F&M chromatograph. The chromatographic column was packed with LAC-3R-728 (diethylene glycol succinate polymer) treated with phosphoric acid (6). It seemed that the solvents present did not interfere with the determination. Therefore, the samples were analyzed without solvent removal.

For the system Unitol ACD-DMSO-water, the acids were analyzed by titration with alcoholic potassium hydroxide and the water by the Karl-Fischer method. The water contents in both layers were added up. The results agreed with the total amt of water originally present, within experimental error. The DMSO was, therefore, obtained by difference.

Experimental Results. Separation factors of linoleic acid were correlated as the function of the concn of acids and the results were shown in Figure 1. The equilibrium distribution curve and the tie lines for the system Unitol ACD-DMSO-water were given in Figure 2.

TABLE I

Analysis of Unitol ACD

Fatty acids, %	98.8
Rosin acids, %	0.6
Unsaponifiables, %	0.6
Acid no.	199
Saponification no.	200
Color	4
Saturated acids, %	2.4
Iodine no.	132
Titer, °C	0.4
GLC analysis[a]	
Oleic acid, %	56.6
Linoleic acid, %	39.0
Stearic acid, %	2.6
Others, %	1.8

[a] GLC analysis was done at the author's laboratory.

Discussion. The separation factor of linoleic acid decreases with increasing acid concn in the DMSO layer. In designing equipment for extraction an optimum condition should be chosen, considering both the selectivity and the capacity.

If operating conditions are chosen such that the average separation factor of linoleic acid is 1.7, a minimum of twelve theoretical plates will be required to obtain a 95% oleic acid fraction at one end and a 95% linoleic acid at the other.

Conclusions

The experimental data presented here indicate that oleic acid and linoleic acid can be separated by extraction with selective solvents. As extraction possesses many favorable engineering aspects as a unit operation, the process warrants further consideration from an economic standpoint.

REFERENCES

1. Cannon, J. A., K. T. Zilch and H. J. Dutton, Anal. Chem. *24*, 1530–1532 (1952).
2. Freeman, S. E., (Pittsburgh Plate Glass Co.) U.S. 2,200,391 (1940).
3. *Ibid.* U.S. 2,278,309 and 2,291,461 (1942).
4. *Ibid.* U.S. 2,313,636 and 2,316,512 (1943).
5. Gloyer, S. W., Ind. Eng. Chem. *40*, 228–236 (1948).
6. Metcalfe, L. D., Nature *188*, 142–143 (1960).
7. Passino, H. J., Ind. Eng. Chem. *41*, 280–287 (1949).
8. Will, F., III, Anal. Chem. *33*, 647–648 (1961).

[Received April 27, 1964—Accepted October 20, 1964]

Report of the Instrumental Techniques Committee, AOCS, 1963-1964[1]

AT ITS INAUGURAL meeting, the Instrumental Techniques Committee agreed that each of its Subcommittees would meet during each Annual Convention of the Society to discuss progress and to consider plans for the coming year. These independent meetings of the Subcommittees are to be followed by a meeting of the entire Committee where objectives of each Subcommittee would be reviewed, and any activities involving more than one Subcommittee would be discussed. At each Fall Convention of the Society only a single meeting of the entire Instrumental Techniques Committee, to consider progress and plans of each Subcommittee, is to be scheduled.

Accordingly, during the past year the Instrumental Techniques Committee held two meetings. The first of these was in the Marquette Suite A of the Radisson Hotel in Minneapolis, Minn., on Tuesday, Oct. 1, 1963, during the 37th Fall Meeting of the Society. The second meeting was held, following earlier meetings of the Subcommittees, on Wednesday, April 22, 1964, in the Red Oak Room of the Roosevelt Hotel, during the 55th Annual Meeting of the Society.

[1] Report of collaborative work of the USDA, ARS, S. Utiliz. Res. Dev. Div.; E. Utiliza. Res. Devel. Div.; Dep. of Health, Education, and Welfare, FDA; Hormel Institute; and the following companies: Anderson, Clayton & Company; Archer-Daniels-Midland Company; Arizona Chemical Company; Carnation Company; Colgate-Palmolive Company; Darling & Company; Procter & Gamble Company; Provincial Traders Pty., Limited; and A. E. Staley Mfg. Company.

Color Subcommittee

As described in the last report (*1*), the Color Subcommittee had been considering two problems: (1) specifications and methods for surface color by reflectance techniques; and (2) possible revisions of present methods for color evaluation either by the subjective tintometer method (Cc 13b–45) or the objective spectrophotometric method (Cc 13c-50) to provide for the measurement of very light-colored oils if trading rules are modified. At a meeting of the Color Subcommittee held in Atlanta, Georgia on April 22, 1963, during the 54th Annual Meeting of the Society (1), it was decided that before any further experimental effort was devoted to the project on surface color by reflectance

techniques, a poll of the industry for an indication of the needs and interest would be made (2). As there was no response to this published request for expressions of specific needs or of desires of AOCS members with respect to a project for investigating uniform surface color determination, at the recommendation of the Subcommittee Chairman, this project was discontinued.

At the Fall Meeting of the Instrumental Techniques Committee, the Color Subcommittee Chairman reported that, since the National Cottonseed Products Association did not vote favorably to change its trading rules, to require colors of bleached oils for settlement purposes, there did not appear to be any further action required by the Subcommittee at this time, as methods Cc 13b-45 and Cc 13c–50 are satisfactory for present requirements. He recommended that this project be closed. As both projects submitted to the Color Subcommittee were, therefore, terminated, and no unsolved problems remained for its consideration, the Chairman further recommended that this Subcommittee be inactivated until such time as problems upon which it could work profitably were referred to the Instrumental Techniques Committee. This proposal was favorably voted by the entire Committee, and the Color Subcommittee has been inactivated until such time as its services are required by the Instrumental Techniques Committee and by the Society.

TABLE I

Study of Gas Chromatographic Analyses
Sample GC 17
Tall Oil Fatty Acid Methyl Esters

Laboratory	16	18	18:1	18:2	18:2 iso	18:2 t,t conj.	18:2 c,t conj.
1	1.4	2.1	53.2	37.0	1.5	2.6	1.5
2	1.8	2.4	47.4	35.9	3.1	2.9	3.4
3	1.5	2.0	49.6	39.1	2.3	2.6	1.8
4	2.0	1.9	48.9	36.6	2.4	1.9	3.3
5	1.9	2.1	43.4	37.3	4.9	3.7	4.5
6	1.7	2.0	48.2	36.0	3.0	1.7	2.7
7	2.2	4.7	44.7	35.0	5.5	....	1.6
8	1.3	1.1	50.1	39.0	2.5	2.8	2.2
9	2.0	2.3	53.2	34.9	1.9	1.2	0.1
10	1.7	1.4	54.1	39.4	1.1	1.4	.....
11	1.9	2.1	50.3	37.1	2.3	2.4	0.6
12	1.8	2.1	49.2	37.4	2.2	2.8	3.2
13	2.6	4.0	44.7	32.4	3.8	5.5	4.1
14	1.5	1.9	52.8	36.6	1.9	2.5	2.2
Average	1.8	2.3	49.3	36.7	2.7	2.4	2.2
Standard deviation	.33	.94	1.68	1.86	.32	.30	.33

Sample GC 18
Peanut Oil Methyl Esters

Laboratory	16	18	18:1	18:2	20	20:1	22	24
1	13.3	2.9	39.4	37.6	1.7	1.4	3.6	.....
2	12.8	2.9	39.3	37.0	1.6	1.4	3.1	1.4
3	12.0	4.4	38.0	37.4	1.4	1.4	3.5	1.7
4	13.1	2.8	39.3	36.9	1.2	.....	3.6	2.0
5	13.0	2.6	40.0	37.8	2.6	....	2.0	.....
6	13.1	2.6	39.6	37.2	1.9	1.6	2.0	1.2
7	14.2	5.7	33.3	30.9	3.6	.....	5.9	2.4
8	13.3	0.9	38.7	41.1	1.2	1.2	2.8	0.7
9	17.7	3.5	49.3	21.6	1.6	1.3	4.0	1.0
10	13.8	2.2	42.0	38.7	0.7	0.4	2.3	.....
11	13.0	2.7	38.4	37.9	1.3	1.4	3.5	1.8
12	13.3	2.8	38.2	38.4	.....	1.7	3.8	1.6
13	12.7	4.0	34.9	34.9	3.5	3.1	6.2	.....
14	13.6	2.3	38.4	39.0	0.9	1.0	3.3	1.4
Average	13.5	3.0	39.2	36.2	1.7	1.1	3.5	1.1
Standard deviation	.91	.92	1.5	1.95	.88	.82	.94	.86

Sample GC 19
Menhaden Oil Methyl Esters

Laboratory	14	16	16:1	18	18:1	18:2	18:3	18:4	20:5	22:6
1	8.1	21.2	13.6	4.9	14.3	1.5	2.0	5.0	18.6	.....
2	5.6	18.2	11.4	4.4	13.4	1.8	3.2	4.6	14.2	14.9
3	5.2	17.1	9.5	4.0	12.2	2.7	3.0	4.0	14.3	17.6
4	4.6	18.3	10.3	5.3	14.9	3.8	3.1	5.0	13.4	14.5
5	8.0	17.6	14.6	4.5	14.8	7.4	3.5	5.0	11.5	11.2
6	5.5	18.8	10.9	4.6	12.5	2.4	1.9	4.5	13.0	11.0
7	5.6	11.1	9.7	6.2	11.1	5.6	5.6	7.0	15.2	20.7
8	6.3	17.5	12.2	4.1	13.4	3.0	3.0	4.5	14.3	15.8
9	8.0	27.6	14.5	5.0	16.8	2.9	4.0	3.3	tr	0.9
10	9.8	31.4	15.9	5.2	17.6	1.7	2.1	.....	13.0	.....
11	5.6	19.4	10.5	3.2	12.6	2.3	3.1	4.2	14.8	18.3
12	6.4	16.5	12.7	4.9	14.3	3.4	3.5	3.3	14.7	14.7
13	7.4	21.0	12.8	7.2	15.7	4.3	1.8	8.8	....	
14	6.0	19.4	11.0	4.1	12.6	2.8	3.2	4.5	13.4	17.6
Average (omitting 10 and 13)	6.2	18.6	11.7	4.6	13.6	3.3	3.3	4.6	13.1	13.1
Standard deviation	1.0	1.58	1.27	0.78	1.15	1.13	0.78	0.79	1.65	2.31

Gas Chromatography Subcommittee

When analyzing the results of previous collaborative tests, the Gas Chromatography Subcommittee decided that some of the disagreements among collaborators arose from the fact that laboratories were being asked to analyze types of esters totally unfamiliar to them. It was considered reasonable to assume that anyone analyzing a material in his work would have some idea of its composition, and the Subcommittee agreed that future samples submitted for collaborative study should be accompanied by identification of the major components (1). Accordingly, early in the past year three samples were submitted for collaborative study with information regarding major components and relative retention times. Fourteen laboratories participated in the collaborative tests. Results are shown in Table I. These results confirm precision statements in the published AOCS Tentative Method Ce 1–62. They also substantiate the Committee action in 1963 in removing tall oil from the samples prohibited by the method. Considerable dismay has been voiced by several gas chromatographists at the lack of precision, particularly in the analysis of peanut oil methyl esters, sample GC 18, which should be about as simple a test as can be devised; but where the results for stearic acid (for example) range from 0.9 to 5.7%, several collaborators insist that they are able to take a specific published method, as the AOCS Tentative Method Ce 1–62, and obtain reasonably good to good reproducibility (defined as repeating the same analysis in the same laboratory by the same analyst using the same equipment). However, attempts to have the identical analyses performed in collaborative study have resulted in somewhat unsatisfactory precision (defined as repeating analyses on the same sample in different laboratories, by different analysts, using different equipment).

This problem of nonprecision in collaborative results gave rise to two schools of thought. One group

TABLE II

% Trans in Secondary Standards

Laboratory	Sample 1	2	3	4	5	6
1	52.4	24.7	41.5	11.3	61.9	30.2
	52.1	24.5	41.9	11.6	61.7	31.0
2	52.2	24.2	42.1	10.6	61.5	30.9
	52.2	23.8	42.1	11.1	61.1	30.4
3	53.4	23.9	41.1	9.4	64.0	30.1
	53.1	23.9	40.7	9.2	64.4	30.3
4	44.3	24.3	40.1	11.2	63.6	31.9
	49.5	23.3	39.7	10.9	61.6	29.6
5	53.0	25.3	40.6	10.6	62.4	31.6
	53.2	24.8	41.9	10.4	64.8	32.0
6	49.3	23.6	39.6	10.7	63.2	30.8 PE-221
	50.9	24.2	40.5	10.4	60.8	31.0 PE-21
7	48.2	24.0	42.1	11.4	59.8	30.6
	48.8	23.8	42.3	11.4	60.2	30.6
8	49.4	22.8	41.6	11.4	60.8	32.6
	49.2	23.1	42.3	11.3	59.5	31.9
9	49.5	23.7	40.5	10.4	60.6	30.9 IR5A
	50.2	23.9	40.1	10.8	58.4	30.3 PE-221
10	51.2	24.6	36.7	9.7	67.1	31.4
	49.0	23.5	37.7	10.0	66.5	31.9
Average	50.6	24.0	40.8	10.7	62.2	31.0

believed and recommended that the Gas Chromatography Subcommittee of the Instrumental Techniques Committee should disband or probably reorganize into a very small group of three to five members. This group did not believe that further collaborative testing would appreciably improve the precision of gas chromatographic methods. Future methods involving gas chromatography could arise in other technical committees of the Society, designed for a particular type of material. A small Gas Chromatography Subcommittee would act merely as an advisory group with no laboratory assignments. Its principal function would be to review the methods proposed by other Committees with a recommendation to the parent Instrumental Techniques Committee regarding adoption.

A second group believed that, by its very nature, precision must be attacked by a collaborative group studying the causes for poor precision among laboratories. They insisted that poor precision could not be solved by the individual analyst in a specific laboratory, who is obtaining very satisfactory reproducibility (as defined above). Both groups appear to have recognized the fact that greater precision can probably be achieved only by more rigorous specifications of conditions and procedures.

These two views were discussed throughout the past year, and especially at the two meetings of the Committee in Minneapolis, Minn., and in New Orleans, La. At the meeting of the Gas Chromatography Subcommittee preceding the meeting of the entire Committee, and at which 19 members were in attendance, the future objectives of the Gas Chromatography Subcommittee were discussed. It was decided that the Committee should continue collaborative testing, and the following tasks were suggested for future work:

1) Ionization detectors and programmed temperature operation permitted as optional alternates.
2) Precision should be improved.
3) Means for identifying sample components should be provided.
4) Standard samples for checking techniques should be provided or certified.

The Subcommittee Chairman has pointed out that Items 1 and 2 are mutually exclusive. From what little is known as to how to insure better precision, it appears to be more or less agreed that more rigid speci-

TABLE III

Studies of Preparation of Methyl Esters

Methods: Methanol-sulfuric acid; boron trifluoride

Samples: Fatty acids and triglycerides containing isolated *trans* unsaturation and hydroxyl groups.

A. Yield

(g of esters/g of starting materials)

Laboratory	From Acids		From Triglycerides	
	$MeOH-H_2SO_4$	BF_3	$MeOH-H_2SO_4$	BF_3
1	1.03	1.01	0.96	0.95
2	.....	.....	.....	.....
3	0.97	1.03	0.98	.....
4	.....	.....	.....	.....
5	0.95	0.97	0.97	.....
6	1.03	1.03	0.98	.....
7	0.99	1.00	0.98	.....

B. Hydroxyl Value

Laboratory	From Acids			From Triglycerides		
	Original	Methyl Esters by		Original	Methyl Esters by	
		H_2SO_4	BF_3		H_2SO_4	BF_3
1	84.4	78.9	77.4	79.7	78.4	.....
2	.....	.....	.....	.....	.....	.....
3	83.1	82.8	82.8	84.1	84.1	.....
4	.....	.....	.....	.....	.....	.....
5	77.0	70.5	74.3	79.7	77.8	.....
6	83.9	78.8	75.8	78.1	84.2	.....
7	84.3	.....	92.4	84.6	....	.....

C. *Trans* Content

Laboratory	From Acids			From Triglycerides			
	Original (% elaidic)	Methyl Esters by		Original (% Trielaidin)		Methyl Esters by	
		H_2SO_4	BF_3	As reported	Corrected [a]	H_2SO_4	BF_3
		(% Methyl elaidate)				(% me. elaidate)	
1	28.6	27.0	25.7	33.1	29.6	27.1	.....
2	.....	.....	.....	.....	.....	.....	.....
3	26.0	24.1	24.1	28.9	25.4	24.9	.....
4	.....	.....	.....	.....	.....	.....	.....
5	25.5	26.6	24.5	25.6	22.1	24.9	.....
6	25.6	24.8	25.3	25.7	22.2	25.2	.....
7	23.7	20.9	21.4	24.2	20.7	20.9	.....

[a] Corrected for absorption of triglycerides at 10.3μ by subtracting 3.5 from reported values. This correction represents the average *trans* content calculated for 9 natural fats, which otherwise showed no *trans* when analyzed as methyl esters. (Table 2, Firestone and Villadelmar, JAOCS *44*, 459–64 (1961).

D. Ricinoleic Acid Content

Laboratory	Acids					Triglycerides		
	Original (% acid) From OH value	Methyl Esters (% Methyl ricinoleate)				Original (% triricinolein) From OH value	Methyl Esters (% Methyl ricinoleate)	
		$MeOH: H_2SO_4$		BF_3			$MeOH: H_2SO_4$	
		From OH value	By GLC	From OH value	By GLC		From OH value	By GLC
1	44.9	43.9	.....	43.1	.....	44.1	43.6	.....
2	.....	.....	.....	.....	.....	.....	.....	.....
3	44.6	46.1	40.3	46.1	40.3	46.6	46.8	40.3
4	.....	.....	.....	.....	.....	.....	.....	.....
5	41.2	39.2	43.7	41.4	45.6	44.2	43.3	44.4
6	45.0	43.9	42	42.2	42	43.3	46.9	44
7	45.2	.....	41.4	51.4	41.5	46.9	.....	42.2

fications of instrument conditions and operating procedures will be required, thus further limiting freedom in choice of equipment and operating conditions.

The Subcommittee on Gas Chromatography has taken a leading part in establishing liaison with other organizations. The method published by the Subcommittee, AOCS Ce 1–62, has been reviewed by ASTM Committee E–19 on Gas Chromatography. It has appeared in expanded form on a letter ballot to ASTM Committee D–1 members. It has been adopted in essentially this same form by the AOAC. Copies of the final draft have been received for comment and were distributed to all Subcommittee members at about the time of the New Orleans meeting of the Subcommittee.

TABLE IV

Study of Preparation of Methyl Esters

Esterification Trial No. 3

Methanol Sulfuric Acid Procedure

A. Effect on Isolated *trans* Unsaturation

Sample	Glycerides G–3		Acids A–3	
	B–IR5A	PE–221	B–IR5A	PE–221
	% trans	% trans	% trans	% trans
Original Materials	25.5 27.7	23.6 24.5	22.8 24.1	21.9 23.0
Average	26.6	24.1	23.5	22.5
Methyl Esters				
Lab No. 1	23.0 25.3	23.7 21.7	23.7 25.5	23.2 23.5
Lab No. 3	23.4 19.5	22.2 22.5	23.0 25.3	22.2 21.5
Lab No. 5	26.5	25.0	25.8 27.8	24.2 23.2
Lab No. 7	23.4 28.5	24.5 22.5	23.4 28.5	23.0 26.3
Lab No. 8	22.0	22.7	23.2 28.5	21.7 23.2
Lab No. 9	23.2 25.0	22.2 20.2	23.0 25.5	22.2 21.2
Average	24.0	22.7	25.3	23.0
S.D.	2.5	1.4	2.1	1.4

B. Effect on Hydroxyl Content

Sample	Glycerides G–3		Acids A–3	
	OH Value	% Ricinoleate	OH Va ue	% Ricinoleate
Original Materials	51.6	28.7	60.5	32.2
Methyl Esters:				
Lab No. 1	59.5	33.2	83.0	46.3
Lab No. 3	68.1	38.0	40.3	22.5
Lab No. 5	186.7	104.2	60.1	33.5
Lab No. 7	60.1	33.5	19.5	10.8
Lab No. 8	60.1	33.5	29.1	16.2
Lab No. 9	73.2	40.8	57.4	32.0

C. Effect on Diene Conjugation

Sample	Diene Conjugation	
	Glycerides G–4	Acids A–4
	% 18:2 conj.	% 18:2 conj.
Original Material	26.5 27.2	27.9 28.1
Average	26.9	28.0
Methyl Esters:		
Lab No. 1	28.0 28.1	28.1 28.5
Lab No. 3	26.8 27.4	27.8 28.1
Lab No. 5	19.1[a] 19.3[a]	26.6 26.7
Lab No. 7	25.2 25.5	29.0 29.4
Lab No. 8	27.2 27.4	25.7 25.8
Lab No. 9	24.7 24.9	25.4 25.6
Average	26.5	27.2
S.D.	1.3	1.4

[a] Not included in average

Spectroscopy Subcommittee

At the first meeting of the Instrumental Techniques Committee during the past year, the Spectroscopy Subcommittee discussed the advisability of rechecking the secondary standards which are distributed for use with AOCS Method Cd 14–61 for the determination of isolated *trans* isomer content by means of IR spectroscopy. These secondary standards were established several years ago to enable oil chemists to use the IR procedure for quantitative determinations of isolated *trans* isomers without the necessity of preparing the labile primary standards otherwise necessary to calibrate the IR spectrophotometer. The secondary standards are based on very highly purified primary standards, elaidic acid, methyl elaidate and trielaidin. A complete set of secondary standards consists of six samples, fatty acids, methyl esters and triglycerides containing a "high" level of isolated *trans* content, 40–60%, and a "low" level, 10–30%, precisely determined by the entire Subcommittee. Over fifty sets of these standards have been distributed and, although tests indicate that they are very stable, it was decided that the extent of interest and the necessity for reliable secondary standards was of sufficient importance that the Subcommittee should reexamine the *trans* content of these secondary standards. Through the excellent cooperation of Anderson, Clayton & Company, and of the Hormel Institute, new primary standards of elaidic acid, methyl elaidate and trielaidin were obtained and carefully checked by each member of the Subcommittee. The set of six secondary standards were remeasured using these new primary standards for instrument calibration. Results are given in Table II. These results confirm the opinion that the secondary standards had not altered appreciably during the years they have been distributed. The small differences in the absolute values may indicate very slight changes in the secondary standards, or they may be indicative of slightly different values for the new primary standards as compared to those prepared and measured some years ago. While the changes in the values are very small, the Subcommittee at its meeting in New Orleans, in April, 1964, voted to use the newer values. These new values will be given to each analyst requesting the secondary standards.

At their meeting in New Orleans in April, where the above decisions were made, the Spectroscopy Subcommittee considered future objectives. The popularity of the IR method for determination of isolated *trans* isomers indicates that if readily available, a method for the determination of *trans-trans and cis-trans* conjugated dienes by IR spectroscopy would be very helpful to many oil chemists. Methods to accomplish these determinations are available in the literature, but unless the individual analyst can obtain the required primary standards, pure *trans-trans* and *cis-trans* conjugated acids, esters and triglycerides the quantitative aspects are not readily available. The Subcommittee voted to work on an AOCS procedure which would be based on stable secondary standards, which like the standards for isolated *trans* isomers, could be furnished to analysts interested in using the proposed procedure. Work on this method will continue during the next year. The Subcommittee also voted to investigate further problems in background corrections in the quantitative determination of small quantities of isolated *trans* isomers in triglycerides.

Special Task Group for Preparation of Methyl Esters

This Task Group was established to devise and recommend a method for the preparation of methyl esters from either the free acids or from the triglycerides, which would be rapid, reliable, simple and widely applicable. The method was to be designed especially for the preparation of methyl esters as required for analyses by either infrared spectrophotometry or by gas chromatography. The first collaborative investigations of this group with soybean oil fatty acids showed that the methanol:acid and the methanol:BF_3 procedures were preferred by a majority of the collaborators and were most promising (3). As their second collaborative effort, the Task Group obtained a triglyceride sample containing approximately 25% elaidate and 40% ricinoleate, and prepared mixed fatty acids. Portions of each of these materials were sent to collaborating laboratories for preparation of methyl esters from the fatty acids by the Methanol:sulfuric acid and by the methanol:BF_3 methods and from triglycerides by the methanol:sulfuric acid procedure. These collaborative tests, in which seven laboratories participated, were completed and analyzed early during the past year. Results are given in Table III. From a yield standpoint, both procedures give essentially complete recovery of methyl esters. The hydroxyl values and the content of ricinoleate calculated from them and from GLC analysis indicate little or no loss of hydroxyl groups during esterification of either acids or triglycerides. Similar conclusions can be derived from the results of *trans* determination. Both methods appear equally satisfactory, and can be used to prepare methyl esters from fatty acids containing hydroxyl groups and isolated *trans* unsaturation. However, the methanol:H_2SO_4 procedure can be used to prepare esters directly from triglycerides. Because of this wider applicability, and because the Uniform Methods Committee has requested that we limit our recommendation to a single method, the Task Group voted to devote a major portion of its future efforts to further study of this method.

A draft of a Tentative Method for the preparation of methyl esters by the methanol:H_2SO_4 procedures has been prepared. This draft was discussed at the Spectroscopy Subcommittee Meeting on Monday, April 20, in New Orleans, La. The draft will be sent to all members of the Task Group for their comments, changes, and corrections before it is submitted to the Instrumental Techniques Committee with recommendation for referral to the Uniform Methods Committee for inclusion as a Tentative Method of the Society.

A third collaborative effort of this Task Group consisted of a test of the methanol:sulfuric acid procedure when employed to prepare methyl esters from glycerides and fatty acids containing isolated *trans* unsaturation, conjugated diene and hydroxyl groups. Results of this collaborative test, in which six laboratories participated are given in Table IV. This method appears to have little, if any effect on *trans* unsaturation or on conjugated diene content. The hydroxyl determination of these samples were, however, very inconsistent, and further collaborative effort is being planned to ascertain the reason for this inconsistency.

ROBERT T. O'CONNOR, Chairman
E. M. SALLEE, Subcommittee Chairman
ROBERT R. ALLEN, Subcommittee Chairman
W. T. COLEMAN, Subcommittee Chairman
J. R. CHIPAULT, Subcommittee Chairman

ACKNOWLEDGMENT

Anderson, Clayton & Company, and the Hormel Institute, University of Minnesota, contributed to the very highly purified new primary standards.

REFERENCES

1. Sallee, E. M., Allen, R. R., Coleman, W. T., Chipault, J. R., and O'Connor, R. T., JAOCS *41*, 158–160 (1964).
2. See JAOCS *39*, No. 10 p. 40, Oct. 1962.
3. O'Connor, R. T., and J. R. Chipault, JAOCS *40* (No. 3) 14, 32–34 (1963).

• *Letter to the Editor*

Mean First Double Bond Distance in Natural Fat Fatty Acids and Its Influence on Azelaoglyceride Analysis

THE MEAN POSITION of the first double bond (mean first double bond distance, MFDB) in the unsaturated fatty acids in a natural fat determines the miscellar molecular weight (MMW) of the dibasic acids remaining in the azelaoglycerides when the fat is oxidized and is of essential importance in azelaoglyceride analysis. Prior to 1955 it was the custom to assume that the MFDB distance in the common vegetable and animal fats of palmitic-stearic-oleic-linoleic type was nine carbon atoms corresponding to azelaic acid and this assumption was made use of in the original gravimetric azelaoglyceride analysis technique (1).

The following evidence, however, rendered the above assumption untenable:

A. Examination by an improved lead salt procedure showed that the common vegetable and animal fats contained a minimum of 2–8% of solid iso-oleic acids, based upon the amount of unsaturated acids present (2). Since vegetable fats as well as lard did not show presence of *trans* isomers by IR spectrophotometry, the iso-oleic acids in these would be *cis*-positional isomers (2).

B. Various proportions of $\Delta^{8:9}$ *cis*-octadecenoic acids were detected in purified oleic acid from various sources (3–5); however, purification will usually tend to remove isomers occurring in smaller proportions.

C. More recently evidence obtained by recalculating earlier data on ripening niger seed (5) and on "after-ripening" linseed (7) has indicated that the more unsaturated acids in vegetable fats are produced by a process of desaturation of the less unsaturated and in the case of linseed this was further confirmed by the new technique of χ "extended after-ripening" (7). It is hence quite possible that polyethenoid acids in natural fats also may not contain the first double bond exclusively in the $\Delta^{9:10}$ position since they may possibly be derived from all the monoethenoid acids.

If the MFDB distance is different from the assumed nine carbon atoms then the original calculations of