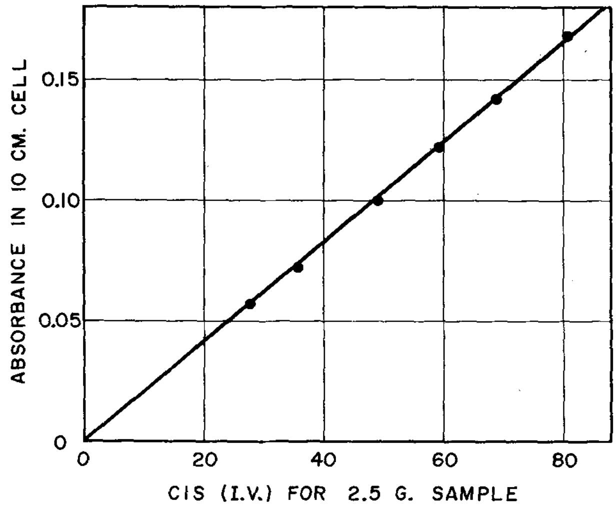

Fig. 3. Calibration curve prepared from hydrogenated cottonseed oils.

ration of a curve from hydrogenated oils is the more attractive because they more closely approximate the compositions of the unknowns to be analyzed.

Conclusion

An analytical method has been developed capable of giving reproducible and accurate determinations of the *cis* unsaturation present in refined oils, hydrogenated oils, and finished shortenings. Molar absorptivity measurements indicate that similar methods are applicable to the analyses of mixed esters or fatty acids.

The method depends upon the measurement of a band in the near infrared at 2.143 μ that is caused by *cis* unsaturation. The following pertinent conclusions are drawn from data obtained in this investigation.

a) The areas under the absorption bands for *cis* polyenes are integral multiples of the areas under the bands of *cis* monoenes.

b) The *cis* content of oils containing less than 10% *trans* triglycerides can be determined accurately by measuring the area under the absorption band.

c) Accurate results can be obtained for samples containing quantities of *trans* fatty acids in excess of 10% if a point baseline is used and the peak absorbance measured.

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Hydrogen Peroxide Variables in Increasing Epoxidation Efficiency[1]

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Prior studies in this laboratory (1) have shown that the conventional technique for *in situ* epoxidation of olefins, using hydrogen peroxide, acetic acid, and sulfuric acid catalyst, can be modified advantageously. The modifications—use of partially preformed peracetic acid prepared by premixing the hydrogen peroxide, and the acetic and sulfuric acids, and control of agitation—result in reduced epoxide cleavage and give a product of higher epoxide content.

Previously reported work with the partially preformed peracetic acid epoxidation method involved use of 50% hydrogen peroxide and was based on employment of a 15 mole % excess of hydrogen peroxide over the reacting olefin.

The present paper extends study of the partially preformed peracetic acid epoxidation method to include variation of both the quantity and concentration of hydrogen peroxide used in the reaction. Quantity variations include use of hydrogen peroxide/olefin mole ratios equivalent to less than a 15 mole % excess of hydrogen peroxide, and the concentration variation includes use of 70% hydrogen peroxide. Soybean oil is used as a model olefin.

Part I. Hydrogen Peroxide/Olefin Mole Ratio

The object of varying the hydrogen peroxide/olefin mole ratio was twofold: a) to determine the minimum amount of hydrogen peroxide required to produce epoxidized soybean oil of high oxirane oxygen content and low iodine number for stabilizer-plasticizer use, and b) to determine how hydrogen peroxide could be best utilized to prepare epoxidized soybean oil for new polymer uses in which low iodine numbers may not be required.

Experimental

A series of soybean oil epoxidations with 50% hydrogen peroxide was carried out by using the partially preformed peracetic acid method (1). Each epoxidation was made with 400 g. of soybean oil (Iodine No. 133.2), using mole ratios of hydrogen peroxide to olefin in the range of 0.25 to 1.15. All the epoxidations were run at 57°C., using an acetic acid to hydrogen peroxide mole ratio of 0.5, a sulfuric acid concentration of 1.8% by weight of acetic acid and 50% hydrogen peroxide, and the previously reported optimum stirring rate of 140 r.p.m. Time for addition of the preformed peracetic acid solution was two hours except in the 0.25 and 0.50 hydrogen

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[1] Presented at the 32nd annual meeting, American Oil Chemists' Society, Chicago, Ill., October 20–22, 1958.

peroxide/olefin runs, where only one hour was taken. Upon completion of the addition and periodically thereafter, samples were taken and analyzed for oxirane oxygen (2) and iodine number (Wijs). The preparation of these samples for analysis has been previously described (1).

Results and Discussion

Epoxidized Soybean Oil for Stabilizer-Plasticizer Use. Current commercial requirements for epoxidized soybean oil as a stabilizer-plasticizer suggest that it have an iodine number of 4 or less. In addition, most commercial epoxidized soybean oil stabilizer-plasticizers have oxirane oxygen contents of at least 6.3%.

Table I, which summarizes the data for a series of epoxidation runs, shows that a satisfactory epoxidized soybean oil for stabilizer-plasticizer use can be obtained by the partially preformed peracetic acid method with only a 5% excess of hydrogen peroxide. Use of a stoichiometric amount of hydrogen peroxide gives a product presumably with too high an iodine number for this application.

TABLE I

Hydrogen Peroxide Requirements for Preparation of Epoxidized Soybean Oil Stabilizer-Plasticizer, Using 50% Hydrogen Peroxide

H_2O_2/olefin mole ratio	Product composition			
	Oxirane oxygen (%)	Iodine number	Mole % epoxide[a]	Reaction time (hr.)[b]
1.00	6.42	7.2	84.7	15
1.05	6.50	4.0	86.9	14
1.10	6.66	4.0	88.0	10
1.15	6.75	4.0	88.7	9

[a] Calculated by using equations based on the assumption that the only cleavage product is the hydroxyacetoxy derivative.

[b] Includes 2-hr. addition period.

Epoxidized Soybean Oil for Polymer Use. New uses for epoxidized soybean oil have been recently proposed in the very large fields of surface-coating alkyds, polyesters, and epoxy resins (3, 4, 5, 6). These uses involve partial replacement of glycols with epoxidized soybean oil in alkyds and unsaturated polyesters, neutralization of residual acid in alkyds, and modification of epoxy resins. In the applications, epoxidized soybean oil becomes an integral part of the polymer molecule, usually through reaction of epoxy groups with carboxyl groups.

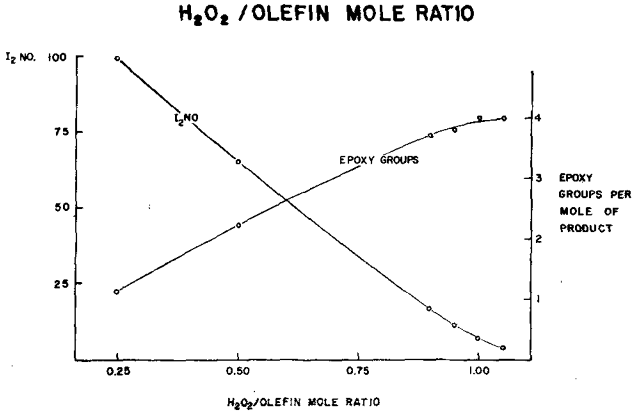


FIG. 1. Product composition with varying H_2O_2/olefin mole ratio.

For such uses epoxidized soybean oil of low iodine number and high epoxy oxygen content is not necessarily required. Instead it would appear that a minimum of two epoxy groups per glyceride molecule would satisfy essential requirements for chain extension. With these requirements in mind, a study was made of the partially preformed peracetic acid epoxidation method, using deficiencies of hydrogen peroxide.

Figure 1 illustrates some of the characteristics of the products obtained when using various deficiencies of hydrogen peroxide in soybean oil epoxidation. Since use of this type of product as a monomer would require at least two epoxy groups per molecule, it is interesting to note that an average of 2 epoxy groups per molecule can be obtained with a hydrogen peroxide/olefin mole ratio of only 0.50. This has important implications on the cost of epoxidized soybean oil prepared for these new polymer uses.

Table II shows that more than 95% utilization of hydrogen peroxide can be obtained when using less than a 1/1 mole ratio of hydrogen peroxide/olefin. The percentage of hydrogen peroxide utilization is defined as moles of double bond oxidized per mole of hydrogen peroxide charged.

Cleavage of the epoxy ring decreases with decreasing hydrogen peroxide/olefin mole ratio. Cleavage is less than 3 mole % of product at a mole ratio of 0.5 and increases to about 8% at a mole ratio of 1.0.

Part II. Epoxidation with 70% Hydrogen Peroxide

Although 70% hydrogen peroxide has been commercially available for some time, its use in epoxidation has not been reported thus far.

In adapting the partially preformed peracetic acid method for use with 70% H_2O_2, some modifications were introduced to insure against formation of potentially detonable mixtures of acetic acid, hydrogen peroxide, and water (7). Thus in contrast to 50% H_2O_2, where mixing the required amounts of acetic

TABLE II

Effect of Deficiencies of 50% Hydrogen Peroxide in Soybean Oil Epoxidation

H_2O_2/olefin mole ratio	0.25	0.50	0.90	0.95	1.00	1.05
Product composition at maximum oxirane oxygen						
Oxirane oxygen, %	1.91	3.74	6.13	6.29	6.44	6.61
Iodine No.	99.5	65.2	16.9	12.9	8.3	6.7
Oxirane, mole %	23.2	46.6	79.7	82.0	84.7	86.9
Unreacted, mole %	76.4	51.2	13.8	10.6	6.9	5.6
Cleavage, mole %	0.4	2.2	6.5	7.4	8.4	7.5
Stirring after addition, hr.	11	13	13	10	10	8
Product composition at termination of reaction						
Oxirane oxygen, %	1.91	3.74	6.12	6.22	6.42	6.50
Iodine No.	99.5	65.2	16.7	11.4	7.2	4.0
H_2O_2 loss, %	1.1	0.7	2.1	1.5	3.3	3.0
H_2O_2 unreacted, %	3.7	1.7	2.1	3.0	2.9	5.0
H_2O_2 utilization, %	95.2	97.6	95.8	95.5	93.8	92.0
Total reaction time, hr.	14	14	16	16	15	14

acid and hydrogen peroxide will yield peracetic acid solutions in the nondetonable range, similar use with 70% H_2O_2 can lead to potentially detonable mixtures.

To avoid these hazardous conditions a two-step partially preformed peracetic acid epoxidation method was developed for use with 70% H_2O_2.

Experimental

Two-Step Partially Preformed Peracetic Acid Epoxidation. Partially preformed peracetic solutions are prepared as with 50% H_2O_2 except that a sufficient portion of the 70% H_2O_2 and sulfuric acid is withheld in order to maintain the composition of the peracetic acid solution outside the detonable region. As a result, the hydrogen peroxide concentration of these solutions is below 30% and outside the detonable region. Addition to the reaction system is then carried out in two steps, as follows: addition of the hydrogen peroxide deficient partially preformed peracetic acid solution to the olefin and addition of the withheld 70% H_2O_2 and H_2SO_4.

The over-all rate of addition is uniform with respect to hydrogen peroxide. Agitation and temperature control are maintained throughout the course of the reaction.

The following epoxidation of soybean oil illustrates this procedure.

Preparation of Partially Preformed Peracetic Acid. To 36.5 g. of acetic acid (0.61 mole) were added 19.4 g. of 70.5% H_2O_2 (0.40 mole) and 1.94 g. of 51.5% H_2SO_4 (equivalent to 1.8% H_2SO_4 by weight of the acetic acid and 70% H_2O_2). After mixing, the solution was allowed to stand 20 hrs. at room temperature to reach equilibrium. Analysis (8) then showed the solution to contain 8.0% hydrogen peroxide and 34.2% peracetic acid (representing a conversion of approximately 64% of the H_2O_2).

Addition: Step 1. The partially preformed peracetic acid solution, representing 16.5% of the total hydrogen peroxide to be utilized, was uniformly added over a 20-min. period to 400 g. of soybean oil (Iodine No. 134.2, equivalent to 2.11 moles of double bond) contained in a 1-liter, three-necked flask fitted with a mechanical stirrer, reflux condenser, thermometer, and addition tube. (In this and other epoxidations, half-moon shaped borosilicate glass stirring blades 75 mm. long were used and were located 20 mm. from the bottom of the 1-liter, three-necked flask. Agitation rates were periodically checked with a tachometer.) During and after the addition period the contents of the reaction flask were continuously stirred at a constant rate while the temperature was maintained at 57°C.

Step 2. A mixture of 97.9 g. of 70.5% H_2O_2 (2.03 moles) and 3.42 g. of 51.5% H_2SO_4 was then uniformly added over a 100-min. period, making a total addition time of 2 hrs. Upon completion of the adtion and periodically thereafter, samples were taken and analyzed for oxirane oxygen (2) and iodine number (Wijs). The preparation of these samples for analysis has been previously described (1).

Results and Discussion

The following variables were studied to obtain high oxirane contents at low iodine numbers (less than 4) along with a minimum of cleavage.

Acetic Acid Concentration. Two-step partially preformed peracetic acid epoxidations were carried out, in which the molar ratio of acetic acid to hydrogen peroxide varied from 0.15 to 0.33.

Table III summarizes the results obtained. The optimum AcOH/H_2O_2 mole ratio range of 0.15–0.25 found is in marked contrast to that obtained with 50% H_2O_2 (1), where 0.40–0.50 mole ratios were found to be optimum for obtaining maximum oxirane oxygen values. Thus the use of 70% H_2O_2 permits a 50% reduction in acetic acid consumption as compared with the use of 50% H_2O_2.

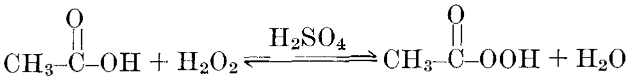
TABLE III
Effect of Acetic Acid Concentration in Epoxidation with 70% Hydrogen Peroxide[a]

AcOH/H_2O_2 mole ratio	0.15	0.20	0.25	0.33
Product composition at maximum oxirane oxygen				
Oxirane oxygen, %	6.83	6.86	6.79	6.57
Iodine No	5.6	3.5	2.9	7.0
Oxirane, mole %	88.9	89.7	89.1	86.0
Unreacted, mole %	4.6	2.9	2.4	5.8
Cleavage, mole %	6.5	7.4	8.5	8.2
Stirring after addition, hr	10	10	8	4
Product composition at Iodine No. 4				
Oxirane oxygen, %	6.83	6.85	6.79	6.55
Cleavage, mole %	7.5	7.2	7.8	10.4
Stirring after addition, hr	12	9	7	5½

[a] Iodine No. of soya oil, 134.2 (Wijs); 15 mole % excess of H_2O_2; H_2SO_4 concentration, 1.8% (by wt. of acetic acid and 70% H_2O_2); stirring rate, 140 r.p.m.; temperature, 57°C.; addition time, 2 hrs.

The variation in reaction time is typical. The higher acetic acid concentrations yield shorter reaction times. Therefore, in the optimum 0.15–0.25 range, the higher mole ratios should be used where short reaction times are desired. Lower mole ratios should be used when low acetic acid usages are wanted.

The lower actic acid requirement for epoxidations carried out with 70% H_2O_2 is readily understandable upon consideration of the peracetic acid equilibrium.



$$CH_3-\overset{\overset{O}{\|}}{C}-OH + H_2O_2 \underset{}{\overset{H_2SO_4}{\rightleftharpoons}} CH_3-\overset{\overset{O}{\|}}{C}-OOH + H_2O$$

The lower water content of 70% H_2O_2 moves the equilibrium further to the right as compared with 50% H_2O_2. Thus the acetic acid concentration can be reduced when using 70% H_2O_2, while still obtaining the same peracetic acid equilibrium as with 50% H_2O_2. This follows since the shift of the equilibrium to the left caused by a reduction in acetic acid concentration would be offset by a shift to the

TABLE IV
Effect of Sulfuric Acid Concentration in Epoxidation with 70% Hydrogen Peroxide[a]

H_2SO_4 conc. (% by wt. of acetic acid and 70% H_2O_4)	1.0	1.4	1.8	2.2
Product composition at maximum oxirane oxygen				
Oxirane oxygen, %	6.83	6.83	6.86	6.80
Iodine No	4.0	3.8	3.5	2.1
Oxirane, mole %	89.2	89.3	89.7	89.3
Unreacted, mole %	3.3	3.1	2.9	1.7
Cleavage, mole %	7.5	7.6	7.4	9.0
Stirring after addition, hr	14	12	10	10
Product composition at Iodine No. 4				
Oxirane oxygen, %	6.83	6.83	6.85	6.77
Cleavage, mole %	7.5	7.6	7.2	7.8
Stirring after addition, hr	14	12	9	7½

[a] Iodine No. of soya oil, 134.2 (Wijs); 15 mole % excess of H_2O_2; AcOH/H_2O_2 mole ratio 0.20; stirring rate, 140 r.p.m.; temperature, 57°C.; addition time, 2 hrs.

right because of the lower water concentration in 70% H_2O_2.

Sulfuric Acid Concentration. Epoxidations were carried out at 57°C. with concentrations of H_2SO_4 varying from 1.0% to 2.2% by weight of acetic acid and 70% H_2O_2. The results summarized in Table IV indicate no significant difference with respect to oxirane oxygen contents. However higher reaction rates are obtained with the higher concentrations of sulfuric acid.

Temperature. Epoxidations carried out at 57°C. and 67°C. with a 0.20 mole ratio of $AcOH/H_2O_2$ showed no differences other than the expected shorter reaction time for the higher temperature reaction (Table VII).

Control of Agitation. Agitation rates were investigated to determine their effect on the reaction. The data shown in Table V indicate that, similar to epoxidations with 50% H_2O_2 (1), mild rates of agitation are essential for optimum results.

TABLE V

Effect of Agitation in Epoxidation with 70% Hydrogen Peroxide[a]

$AcOH/H_2O_2$ mole ratio	0.20		0.33	
stirring rate, r.p.m.	140	275	140	275
Product composition at maximum oxirane oxygen				
Oxirane oxygen, %	6.86	6.40	6.57	6.20
Iodine No	3.5	4.1	7.0	6.5
Oxirane, mole %	89.7	84.7	86.0	82.1
Unreacted, mole %	2.9	3.4	5.8	5.4
Cleavage, mole %	7.4	11.9	8.2	12.5
Stirring after addition, hr	10	9	4	4
Product composition at Iodine No. 4				
Oxirane oxygen, %	6.85	6.40	6.55	6.09
Cleavage, mole %	7.2	11.9	10.4	15.3
Stirring after addition, hr	9	9	5½	5½

[a] Iodine No. of soya oil, 134.2 (Wijs); 15 mole % excess of H_2O_2; H_2SO_4 concentration, 1.8% (by wt. of acetic acid and 70% H_2O_2); temperature, 57°C.; addition time, 2 hrs.

TABLE VI

Hydrogen Peroxide Requirements for Preparation of Epoxidized Soybean Oil Stabilizer-Plasticizer, Using 70% Hydrogen Peroxide[a]

H_2O_2/olefin mole ratio	1.05	1.10	1.15
Product composition at maximum oxirane oxygen			
Oxirane oxygen, %	6.68	6.76	6.79
Iodine No	6.3	3.6	2.9
Oxirane, mole %	87.2	88.7	89.1
Unreacted, mole %	5.2	3.0	2.4
Cleavage, mole %	7.6	8.3	8.5
Stirring after addition, hr	10	10	8
Product composition at Iodine No. 4			
Oxirane oxygen, %	6.66	6.76	6.79
Cleavage, mole %	9.1	8.0	7.8
Stirring after addition, hr	14	9½	7

[a] Iodine No. of soya oil, 134.2 (Wijs); $AcOH/H_2O_2$ mole ratio, 0.25; H_2SO_4 concentration, 1.8% (by wt. of acetic acid and 70% H_2O_2); stirring rate, 140 r.p.m.; temperature, 57°C.; addition time, 2 hrs.

Effect of Excess Hydrogen Peroxide. Table VI shows the results obtained from a series of experiments in which the hydrogen peroxide/olefin mole ratio was varied. Object of this work was to determine minimum quantities of 70% H_2O_2 required to obtain epoxidized soybean oil of high oxirane oxygen and low iodine number for stabilizer-plasticizer use.

The results show that a product having an iodine number of 4 and a satisfactory oxirane content (over 85 mole %) can be obtained by use of only a 5 mole % excess of 70% H_2O_2.

Comparison of Partially Preformed vs. in Situ Epoxidations. A series of parallel reactions were run, one series by the two-step partially preformed peracetic acid procedure and the other by a completely *in situ* method. The latter method differed only in that the sequence of reagent additions was changed: acetic acid and sulfuric acid were added to the soybean oil in the reaction flask, and then the 70% hydrogen peroxide was uniformly added over a 2-hr. period. Table VII summarizes the data obtained from experiments in which the two epoxidation methods were compared at different temperatures and acetic acid concentrations.

The equivalence of the two methods at a 0.20 mole ratio of $AcOH/H_2O_2$ can be explained by the reduced reaction rates resulting from the lower concentration of acetic acid. The lower acetic acid concentration permits the preforming of only about 9% of the total hydrogen peroxide to peracetic acid in the partially preformed process. (This is in contrast to the approximately 25% conversion to preformed peracetic acid obtained with 50% H_2O_2 at 0.5 mole ratio of $AcOH/H_2O_2$.) This amount of preformed peracetic acid is not large enough to affect significantly the course of the reaction, thus the advantages derived from the use of preformed peracetic acid as previously described (1) are minimized.

Upon increasing the acetic acid concentration however, the advantages of the partially preformed method reappear. Thus at 0.33 mole ratio of $AcOH/H_2O_2$ approximately 15% of the total hydrogen peroxide is converted to peracetic acid. This larger amount of peracetic acid, in addition to the reduced contact of acetic and sulfuric acids with the olefin, gives higher epoxide yields with the two-step partially preformed peracetic acid method.

TABLE VII

Partially Preformed *versus in Situ* Epoxidation[a]

Temperature °C.	57°				67°	
$AcOH/H_2O_2$ mole ratio	0.33		0.20		0.20	
Method	Partially preformed	*In situ*	Partially preformed	*In situ*	Partially preformed	*In situ*
Product composition at maximum oxirane oxygen						
Oxirane oxygen, %	6.57	6.16	6.86	6.84	6.80	6.68
Iodine No.	7.0	3.6	3.5	4.1	2.4	2.8
Oxirane, mole %	86.0	82.2	89.7	89.3	89.3	87.9
Unreacted, mole %	5.8	3.0	2.9	3.4	2.0	2.3
Cleavage, mole %	8.2	14.8	7.4	7.3	8.7	9.8
Stirring after addition, hr	4	6	10	10	5	6
Product composition at Iodine No. 4						
Oxirane oxygen, %	6.55	6.15	6.85	6.84	6.80	6.68
Cleavage, mole %	10.4	14.6	7.2	7.3	7.8	9.1
Stirring after addition, hr	5½	5½	9	10	4	4½

[a] Iodine No. of soya oil, 134.2 (Wijs); 15 mole % excess of H_2O_2; H_2SO_4 concentration, 1.8% (by wt. of acetic acid and 70% H_2O_2); stirring rate, 140 r.p.m.; addition time, 2 hrs.

Summary

Variations in quantity and concentration of hydrogen peroxide were studied in epoxidation of soybean oil by using the partially preformed peracetic acid epoxidation method.

Use of a hydrogen peroxide/olefin mole ratio as low as 1.05/1 yields epoxidized soybean oil that meets the low iodine number and high epoxide content characteristics required for stabilizer-plasticizer use.

Use of a hydrogen peroxide/olefin mole ratio as low as 0.50/1 results in more than 95% hydrogen peroxide utilization and yields epoxidized soybean oil containing more than two epoxide groups per molecule. Products of this type may be of interest for recently proposed applications in alkyd, polyester, and epoxy resins.

Increasing the hydrogen peroxide concentration to 70% in epoxidation permits reduction of acetic acid usage to half that required when 50% hydrogen peroxide is used. Agitation control is also necessary for optimum results. A two-step epoxidation method can be used to avoid formation of potentially detonable mixtures in epoxidation with 70% hydrogen peroxide by the partially preformed peracetic acid method.

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[Received April 20, 1959]

Report of the Spectroscopy Committee, 1958-59

THE MAJOR ACTIVITY of the Spectroscopy Committee of the American Oil Chemists' Society during the year ending with the 50th Annual Meeting of the Society in New Orleans has been a collaborative investigation of a proposed method for the determination of *trans* isomers by means of infrared absorption. A single meeting was held during the 50th meeting of the Society in New Orleans.

Determination of trans Acids by Means of Infrared Absorption

As a collaborative test of a proposed A.O.C.S. Tentative Method for Isolated *trans* Isomers by Means of Infrared Absorption Spectrophotometry, 11 samples, including three primary standards of very high purity and eight analytical samples, were furnished each committee member as follows:

No. 1. Elaidic acid, primary standard
No. 2. Methyl elaidate, primary standard
No. 3. Trielaidin, primary standard
No. 4. Hydrogenated methyl oleate, low *trans* content
No. 5. Hydrogenated methyl oleate, high *trans* content
No. 6. Vegetable oil
No. 7. Fatty acids
No. 8. Methyl esters
No. 9. Hydrogenated soybean oil, low *trans* content
No. 10. Hydrogenated soybean oil, high *trans* content
No. 11. Hydrogenated vegetable oil

Each committee member was asked to analyze each sample at least in duplicate on as many different infrared spectrophotometers as were available to him. Results, received from all 10 members of the Spectroscopy Committee, are given in the attached table.

Committee Meeting April 21, 1959

The committee met in the Mardi Gras room of the Roosevelt hotel on April 21, 1959, with nine of the 10 committee members present (or represented), and five guests: members—Robert R. Allen, Anderson Clayton and Company; J. R. Chipault, Hormel Institute; N. D. Fulton, Procter and Gamble Company; Samuel F. Herb, Eastern Utilization Research and Development Division; William E. Link, Archer-Daniels-Midland Company; Donald E. Reid (for Robert D. Mair), Hercules Powder Company; Donald H. Wheeler, General Mills Inc.; Hans Wolff, A. E. Staley Manufacturing Company; and Robert T. O'Connor, Southern Utilization Research and Development Division; and guests—A. S. Fenton, Procter and Gamble Company; David Firestone, Food and Drug Administration; Marvin Formo, Archer-Daniels-Midland Company; Donald F. Kuemmel, Procter and Gamble Company; and Elizabeth McCall, Southern Utilization Research and Development Division.

Discussion of Results of Collaborative Tests. The recently completed collaborative work to test a proposed method for isolated *trans* isomers by infrared absorption was discussed in detail, and the following conclusions were agreed upon.

a) Inasmuch as infrared absorption measurements can and will be made with several instruments there is no object in citing a specific procedure for any one (or more) particular instrument. The proposed tentative method should be sufficiently broad to include any spectrophotometer meeting minimum requirements.

b) Inasmuch as the results on the analysis of acids do not show satisfactory agreement, the proposed tentative method should not include within its scope the analysis of acids. Acids can, of course, be analyzed by first converting them to their methyl esters.

c) Further work should be initiated to devise a satisfactory method for the direct analysis of acids. The committee feels that this analysis should also be attempted by some "baseline" type of background correction rather than a correction based on a measured oleic acid standard. Further collaborative work is recommended.

d) As the proposed infrared method is to be a general one, for use on all infrared spectrophotometers, it is not possible to supply values for absorptivities to be used in calculation of percentage of isolated *trans* (as such values differ widely for different instruments and even for the same models unless measured with exact programming). Therefore the primary standards, methyl elaidate and trielaidin, will be required for calibration of instruments by all users of the proposed tentative method. However such primary standards will not usually be readily available. Therefore the committee believes that it