Although the phosphatides of linseed, corn, and soybean oil show many similarities, they also exhibit many differences. All can be separated into alcoholsoluble and alcohol-insoluble fractions, but the proportions are widely different. Seventeen per cent of the linseed phosphatides appear in Fraction I (alcohol-soluble) and 78% in Fraction IV. Corresponding values for these fractions of soy phosphatides (6) are 51% and 38%. Besides having the smallest alcohol-soluble fraction, linseed also has the lowest concentration of phosphorus (2.63%) in this fraction. It is interesting that the large alcohol-insoluble fraction of linseed also contains the highest concentration of phosphorus (3.97%). Amino nitrogen occurs in both Fractions I and IV; the concentration is the same in the two fractions of soy phosphatides, but widely different in linseed.

Countercurrent distribution between hexane and 90% methanol shows that the alcohol-soluble fraction of the phosphatides from soybean, corn, and linseed oils consists of a group of compounds of similar solubility, more soluble in alcohol than in hexane. Distribution of the alcohol-insoluble fractions of the phosphatides from the three sources shows two groups, one more soluble in alcohol and the other more soluble in hexane. In the soy fraction the two groups are about equal in amount; in corn, the hexane-soluble portion is somewhat larger; and in linseed, it is much larger.

The differences in composition are great enough to suggest that linseed phosphatides might be of outstanding value in applications where the desired effect is caused primarily by the alcohol-insoluble fraction.

Summary

Alcohol-soluble and alcohol-insoluble fractions of linseed phosphatides were subjected to countercurrent distribution in the Craig apparatus. The soluble portion, which was shown by analysis to contain essentially all of the choline and very little of the

inositol, gave little further fractionation on distribution between hexane and 90% methanol. Distribution of the alcohol-insoluble phosphatides between hexane and 90% ethanol showed two types of phosphoinositides to be present. Those concentrated in the alcohol phase had a phosphorus: nitrogen: inositol ratio of approximately 2:1:1 whereas those more soluble in hexane had a ratio of approximately 4:4:1.

Analysis for "ethanolamine" by the periodate method failed to show as much amino nitrogen as analysis by the Van Slyke method. This difference, considered with the shape of the ethanolamine nitrogen curves, prevents a satisfactory calculation of the amount of cephalin.

Sugar occurs also in all fractions, but its mode of combination, if any, was not demonstrated.

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Report of the Spectroscopy Committee, 1951

TN 1948 this committee recommended to the Society the adoption of a detailed ultraviolet spectrophotometric method for the determination of polyunsaturated acids. The method was essentially that of Beadle and Kraybill (1), Brice and Swain (2), Lemon (3), and others as modified for use in determining the polymerization index of soap used in synthetic rubber manufacture. The method as recommended and subsequently adopted by the Society was tested by the Spectroscopy Committee and reported upon in previous committee reports. Since the last report further cooperative work has been done to establish the validity of the method and to investigate suggested changes. This year's work reported herein was pointed toward:

a) The use of a new set of constants for calculating the tetraene, triene, and diene constituents of fats and oils. The new constants were reported on at the 1948 Fall Meeting in New York by Brice et al., following work done on natural acids prepared at the Eastern Regional Research Laboratory.

- b) The use of a 45-minute instead of a 25-minute isomeriza-
- Substitution of the equation $k_2 = k_{233} k_0$ for $k_2 = k_{233} k_0$ 0.029 - 0.052 P. In the calculation ko is 0.07 for esters and 0.03 for soaps and fatty acids. P was the estimated decimal fraction of oleic acid content of the sample being examined. Brice and Swain (2) showed that the absorption of methyl stearate and methyl oleate was identical and proposed in their report the use of the correction ko
- d) Simplification of the method by elimination of all measurements in the tetraenoic region when vegetable oils were being analyzed.

Four samples of vegetable oils, two cottonseed and two soybean, were submitted to six collaborating laboratories for analysis. Detailed instructions were given for analyzing the oils and for calculating the fatty acid contents. Five laboratories report the analysis of the samples by the prescribed spectrophotometric methods. Two laboratories also reported saturated acids by the Bertram oxidation procedure, two

TABLE I Cottonseed Oil No. 1

| | Lal | b. 1 | La | b. 2 | Lab. 3 | | | Lab 4 | | | 1 | Spread |
|--|--------|--------|-------|-------|--------|--------|--------|--------|--------|--------|-----------|--------|
| | A | В | A | В | A | A | В | C | D | E | E Average | |
| Method 1, AOCS Cd 7-48 | | | 1 | | | - | | | | | | |
| Conjugated Diene | 0.32 | | 0.33 | | 0.34 | 0.382 | | | | | 0.343 | 0.06 |
| Conjugated Triene | 0.007 | | 0.01 | | 0.007 | 0.006 | | | | i | 0.008 | 0.004 |
| Conjugated Tetraene | 0.000 | | 0.00 | | 0.0 | 0.00 | | | | i | 0.000 | 0.00 |
| Total % Conjugated | 0.327 | 0.327 | 0.34 | 0.34 | 0.347 | 0.388 | 0.388 | 0,388 | 0.388 | 0.388 | 0.351 | 0.061 |
| % Arachidonic | 0.00 | 0.00 | 0.00 | 0.00 | 0.002 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.00 | 0.002 |
| % Linolenic | 0.00 | 0.00 | 0.00 | 0.00 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.00 | 0.00 |
| % Linoleic | | 56.0 | 55.0 | 55.0 | 54.9 | 52.95 | 55.42 | 54.35 | 56.43 | 53.74 | 54.98 | 3.4 |
| % Oleic | 11.0 | 11.0 | 13.3 | 13.3 | 11.3 | 16.01 | 11.04 | 13.19 | 9.01 | 14.43 | 12.36 | 7.0 |
| % Saturated | 28.3 | 28.3 | 27.0 | 27.0 | 29.1 | 26.25 | 28.75 | 27.67 | 29.77 | 27.04 | 27.92 | 3.5 |
| Total % Acids | 95.627 | 95.627 | 95.64 | 95.64 | 95.649 | 95.598 | 95.598 | 95.598 | 95.598 | 95.598 | 95.611 | |
| Method 2, AOCS Cd 7-48 using | | | | | - 31 | | | | | | | |
| Revised Constants | | | | | | | | | | | | |
| Total % Conjugated | 0.327 | 0.327 | 0.34 | 0.34 | 0.347 | 0.388 | 0.388 | 0.388 | 0.388 | 0.388 | 0.351 | 0.061 |
| % Arachidonic | | 0.00 | 0.00 | 0.00 | 0.002 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.00 | 0.002 |
| % Linolenic | 0.00 | 0.00 | 0.00 | 0.00 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.00 | 0.00 |
| % Linoleic | | 52.4 | 51.5 | 51.5 | 51.4 | 49.58 | 51.89 | 50.88 | 52.83 | 50.31 | 51.47 | 3.2 |
| % Oleic | 18.4 | 18.4 | 20.3 | 20.3 | 18.5 | 22.79 | 18.14 | 20.18 | 16.25 | 21.32 | 19.46 | 6.5 |
| % Saturated | 24.5 | 24.5 | 23.5 | 23.5 | 25.4 | 22.84 | 25.18 | 24.15 | 26.13 | 23.58 | 24.33 | 3.3 |
| Total % Acids | 95.627 | 95.627 | 95.64 | 95.64 | 95.649 | 95.598 | 95.598 | 95.598 | 95.598 | 95.598 | 95.611 | |
| Method 3, AOCS Cd 7-48 using 45-Minute | | | | | | | | | | | | |
| Isomerization and Revised Constants | | | | | | | | | | | | |
| Total % Conjugated | 0.327 | 0.327 | 0.34 | 0.33 | 0.347 | 0.388 | 0.388 | 0.388 | 0.388 | 0.388 | 0.351 | 0.061 |
| % Arachidonic | 0.000 | 0.000 | 0.00 | 0.00 | 0.006 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.00 | 0.006 |
| % Linolenic | 0.00 | 0.00 | 0.00 | 0.00 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.000 | 0.000 |
| % Linoleic | | 53.3 | 51.8 | 51.7 | 51.5 | 52.03 | 52.13 | 50.33 | 50.92 | 51.35 | 51.84 | 3.0 |
| % Oleic | | 16.6 | 19.7 | 19.9 | 18.2 | 17.87 | 17.66 | 21.28 | 20.10 | 19.23 | 18.71 | 4.7 |
| % Saturated | | 25.4 | 23.8 | 23.7 | 25.6 | 25.31 | 25.42 | 23.60 | 24.19 | 24.63 | 24.71 | 2.0 |
| Total % Acids | 95.627 | 95.627 | 95.64 | 95.63 | 95.653 | 95.598 | 95.598 | 95.598 | 95.598 | 95.598 | 95.611 | •••• |

by a crystallization procedure and one by the Twitchell separation modified by thiocyanogen analysis on the solid acids separated. Two laboratories reported thiocyanogen analysis using AOCS method Cd 2-38. The data from one laboratory was too inconsistent, using any of the spectrophotometric procedures, to be considered as valid evidence and has therefore been excluded in its entirety.

The oils analyzed were identified as Cottonseed No. 1, Cottonseed No. 2, Soybean No. 3, and Soybean No. 4.

The spectrophotometric analyses are shown in Tables I, II, III, and IV. Only one analysis for preconjugated constituents is shown since none of the variations employed affected the amount of these acids originally present in the oil.

Method 1 is AOCS Tentative Method Cd 7-48 in exact detail and calculations.

Method 2 is AOCS Tentative Method Cd 7-48 in exact detail but the calculations are those of Brice et al. (4), which will be shown later in the report.

Method 3 follows the general outline of AOČS Tentative Method Cd 7-48 except that the sample was isomerized for 45 minutes instead of 25 minutes and Brice's et al. corrected constants for 45 minutes of isomerization time were used in the calculations. The calculations employed were,

$$\begin{array}{l} k'_2 = k'_{233} - k_{293} \\ k'_3 = 4.1 \left[k'_{298} - 1/2 \left(k'_{282} + k'_{274} \right) \right] - k_3 \\ k'_4 = 2.06 \left[k'_{315} - 1/2 \left(k'_{308} + k'_{322} \right) \right] - k_4 \\ X = 1.073 \, k'_2 - 1.271 \, k'_3 - 0.05 \, k'_4 \\ Y = 2.028 \, k'_3 - 3.91 \, k'_4 \\ Z = 4.33 \, k'_4 \end{array}$$

Table V shows the composition of the oils by the thiocyanogen method. Also shown are results using the Bertram, crystallization, and modified Twitchell procedures for saturated acids.

TABLE II Cottonseed Oil No. 2

| | La | b. 1 | La | b. 2 | Lab. 3 | | La | b. 4 | | | |
|--|--------|--------|-------|-------|--------|--------|--------|--------|--------|---------|-------------------|
| | A | В | A | В | A | A | В | C | D | Average | Spread |
| Method 1, AOCS Cd 7-48 | | | | | | - | | *** | | | |
| Conjugated Diene | 0.42 | | 0.41 | | 0.435 | 0.461 | | | | 0.432 | 0.051 |
| Conjugated Triene | 0.003 | | 0.00 | | 0.003 | 0.003 | | | | 0.003 | 0.003 |
| Conjugated Tetraene | 0.000 | | 0.00 | | 0.00 | 0.00 | | | | 0.000 | 0.00 |
| Total % Conjugated | 0.423 | 0.423 | 0.41 | 0.41 | 0.438 | 0.464 | 0.464 | 0.464 | 0.464 | 0.435 | 0.054 |
| % Arachidonic | 0.00 | 0.00 | 0.01 | 0.01 | 0.002 | 0.01 | 0.00 | 0.00 | 0.00 | 0.004 | 0.034 |
| % Linolenic | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.000 | 0.00 |
| % Linoleic | 54.6 | 54.3 | 53.6 | 53.2 | 52.2 | 53.74 | 54.42 | 52.59 | 52.21 | 53.43 | 2.4 |
| % Oleic | 11.7 | 12.3 | 14.1 | 14.8 | 15.8 | 12.25 | 10.89 | 14.59 | 15.35 | 13.53 | 4.5 |
| % Saturated | 28.9 | 28.6 | 27.5 | 27.1 | 27.2 | 29.14 | 29.82 | 27.96 | 27.58 | 28.20 | $\frac{4.5}{2.7}$ |
| Total % Acids | 95 623 | 95.623 | 95.62 | 95.55 | 95.640 | 95.604 | 95.594 | 95.604 | 95.604 | | 2.,1 |
| Method 2, AOCS Cd 7-48 using | 00.020 | 00.020 | 00.02 | 90.00 | 99.U4U | 33.004 | 93.094 | 93.004 | 90.004 | 95.599 | •••• |
| Revised Constants | | | i | | | | | | | | |
| Total % Conjugated | 0.423 | 0.423 | 0.41 | 0.41 | 0.438 | 0.464 | 0.464 | 0.464 | 0.464 | 0.405 | 0.054 |
| % Arachidonic | 0.00 | 0.00 | 0.41 | | 0.438 | | 0.464 | 0.464 | 0.464 | 0.435 | 0.054 |
| % Linolenic | 0.00 | 0.00 | 0.00 | 0.01 | | 0.00 | 0.00 | 0.00 | 0.00 | 0.003 | 0.009 |
| % Linoleic | F 3 1 | | | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.000 | 0.00 |
| % Oleic | 51.1 | 50.8 | 50.2 | 49.8 | 48.8 | 50.31 | 50.94 | 49.24 | 48.88 | 50.01 | 2.3 |
| 76 Oleto | 10.0 | 19.4 | 22.0 | 21.7 | 21.7 | 19.20 | 17.92 | 21.35 | 22.08 | 20.46 | 4.2 |
| % Saturated | | 25.0 | 23.0 | 23.7 | 24.7 | 25.63 | 26.28 | 24.55 | 24.18 | 24.70 | 3,3 |
| Total % Acids | 95,623 | 95,623 | 95.62 | 95.62 | 95.647 | 95.604 | 95.604 | 95.604 | 95.604 | 95.608 | **** |
| Method 3, AOCS Cd 7-48 using 45-Minute | | | | i | | 1 | | | | | |
| Isomerization and Revised Constants | | | | | | | | | | | |
| Total % Conjugated | 0.423 | 0.423 | 0.40 | 0.40 | 0.438 | 0.464 | 0.464 | 0.464 | 0.464 | 0.435 | 0.054 |
| % Arachidonic | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 | 0.00 | 0.00 | 0.00 | 0.00 | 0.001 | 0.010 |
| % Linolenic | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.000 | 0.000 |
| % Linoleic | 51.4 | 51.1 | 49.6 | 49.6 | 50.0 | 51.04 | 49.59 | 49.23 | 48.43 | 50.00 | 3.0 |
| % Oleic | 18.1 | 18.8 | 22.3 | 22.3 | 19.2 | 17.71 | 20.63 | 21.37 | 22.98 | 20.38 | 5.3 |
| % Saturated | 25.7 | 25.3 | 23.3 | 23.3 | 26.0 | 26.39 | 24,92 | 24.54 | 23.73 | 24.80 | 3,1 |
| Total % Acids | 95.623 | 95.623 | 95.60 | 95.60 | 95.648 | 95.604 | 95.604 | 95.604 | 95.604 | 95.616 | |

TABLE III Soybean Oil No. 3

| • | La | b. 1 | La | b. 2 | Lab. 3 | | _ Lai | Lab. 4 | | | | |
|--|--------|--------------|-------|-------|--------|---------|--------|--------|--------|---------|--------|--|
| _ | A | В | A | В | A | A | В | c | D | Average | Spread | |
| Method 1, AOCS Cd 7-48 | | | | | | | | | | | | |
| Conjugated Diene | 0.18 | | 0.18 | · | 0.181 | 0.190 | | | | 0.183 | 0.010 | |
| Conjugated Triene | 0.04 | | 0.04 | | 0.004 | 0.038 | | | 1 | 0.031 | 0.036 | |
| Conjugated Tetraene | 0.009 | | 0.01 | | 0.01 | 0.008 | | | 1 | 0.009 | 0.002 | |
| Total % Conjugated | 0.229 | 0.229 | 0.23 | 0.23 | 0.195 | 0.236 | 0.236 | | ı | 0.223 | 0.041 | |
| % Arachidonic | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.02 | 0.02 | | 1 | 0.009 | 0.020 | |
| % Linolenic | 6.94 | 7.01 | 7.24 | 7.24 | 6.68 | 7.43 | 7.59 | | 1 | 7.16 | 0.9 | |
| % Linoleic | 56.8 | 56.8 | 56.3 | 56.3 | 55.7 | 56.69 | 56.17 | | 1 | 56.39 | 1.1 | |
| % Oleic | 15.2 | 15.0 | 16.1 | 16.1 | 17.7 | 12.86 | 13.44 | | | 15.20 | 4.8 | |
| % Saturated | 16.5 | 16.6 | 15.8 | 15.8 | 15.3 | 18.36 | 18.15 | | | 16.64 | 3.1 | |
| Total % Acids | 95,669 | 95.639 | 95.67 | 95.67 | 95.595 | 95.596 | 95.606 | | | 95.622 | | |
| Method 2, AOCS Cd 7-48 using | 00.000 | 00.000 | 00.01 | 00.01 | 00.000 | 100.000 | 00.000 | | | 20.022 | •••• | |
| Revised Constants | | | ļ | | | | | | [| | | |
| Total % Conjugated | 0.225 | 0.225 | 0.23 | 0.20 | 0.195 | 0.236 | 0.236 | | ł | 0.216 | 0.041 | |
| % Arachidonic | 0.03 | 0.03 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | | | 0.009 | 0.030 | |
| % Linolenic | 7.16 | 7.24 | 7.63 | 7.63 | 7.06 | 7.84 | 8.01 | | 1 | 7.51 | 0.9 | |
| % Linoleic | 53.0 | 52.9 | 52.4 | 52.4 | 51.9 | 52.75 | 52.27 | | 1 | 52.52 | 1.1 | |
| % Oleic | 22.1 | 22.0 | 22.7 | 22.8 | 24.1 | 19.64 | 20.09 | | 1 | 21.92 | 4.5 | |
| % Saturated | 13.1 | 13.2 | 12.6 | 12.6 | 12.3 | 15.14 | 15.00 | | 1 | 13.42 | 2.5 | |
| Total % Acids | 95 615 | 95.595 | 95.56 | 95.63 | 95.555 | 95.606 | 95.606 | | ŀ | 95.595 | | |
| Method 3, AOCS Cd 7-48 using 45-Minute | 00.010 | 33.333 | 33.30 | 33.03 | 30.000 | 33.000 | 33.000 | | ľ | 00.000 | **** | |
| Isomerization and Revised Constants | | | | 1 | | 1 | | | i | | | |
| Total % Conjugated | 0.225 | 0.225 | 0.23 | 0.23 | 0.195 | 0.236 | 0.236 | 0.236 | 0.236 | 0.223 | 0.041 | |
| % Arachidonic | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.003 | 0.020 | |
| % Linolenic | 7.42 | 7.52 | 7.44 | 7.40 | 7.09 | 7.77 | 7.73 | 7.54 | 7.51 | 7.49 | 0.7 | |
| % Linoleic | 53.5 | 53.3 | 52.7 | 52.7 | 52.9 | 57.10 | 52.54 | 53.14 | 52.47 | 53.37 | 4.6 | |
| % Oleic | | 20.6 | 22.7 | 22.8 | 22.0 | 11.07 | 20.37 | 19.77 | 21.21 | 20.10 | 11.7 | |
| % Saturated | | 20.6 14.0 | 12.5 | 12.5 | 13.4 | 19.42 | 14.72 | 14.92 | 14.18 | 14.42 | 6.9 | |
| Total % Acids | | 95.645 | | 95.63 | 95.585 | 95.596 | 95.596 | 95.606 | 59.606 | 95,606 | 0.5 | |

Table VI shows a comparison of the average spectrophotometric data and the average data obtained using the thiocyanogen and saturated methods.

Examination of the data shown in Tables I through IV indicates quite clearly the absence of arachidonic acid in any of the samples studied regardless of the method used. The absence of linolenic in the two cottonseed oil samples is also clear-cut. The use of the newly proposed constants increases slightly the amount of linolenic acid found in soybean samples. This is accompanied by a decrease in the amount of linoleic acid and a large increase in oleic acid. At the same time the total saturated acids decrease slightly.

Examination of the spread between individual analyses as carried out by the four different laboratories shows that there is little difference in the precision of the 25-minute and 45-minute isomerization times. Table III shows a considerably greater spread in the oleic acid determined by the 45-minute isomerization. However this can be traced almost entirely to one

determination by Laboratory No. 4 which accounts almost 100% for the increase in spread for this particular sample. The committee members are divided on the question of whether a 25-minute or 45-minute isomerization time should finally be recommended. On the basis of this year's cooperative work increasing the time from 25 to 45 minutes cannot be justified. Future work must have as one of its objectives the determination of the ideal isomerization time.

Table V indicates why there will always be some discrepancies in the spectrophotometric method, which cannot be blamed on the determination of linolenic and linoleic acids. Note, for example, that there is a difference of 2 in the determination of the iodine value by Laboratories No. 2 and No. 3.

Extremely good checks were obtained by the same two laboratories for the thiocyanogen value of these oils. Individual calculation of the composition of the fats using the iodine-thiocyanogen method is not shown since it was felt that the average iodine value

TABLE 1V Soybean Oil No. 4

| | Lal | o. 1 | La | b. 2 | Lab. 3 | | Lat | o. 4 | | Average | Spread |
|--|--------|--------|-------|---------------------|--------|--------|--------|--------|---------------|---------|--------------|
| _ | A | В | A | В | A | A | В | C | D | Average | Spreau |
| Method 1, AOCS Cd 7-48 | | | | | | | | | | | |
| Conjugated Diene | 0.16 | 0.17 | 0.17 | | 0.181 | 0.175 | | | 1 | 0.171 | 0.021 |
| Conjugated Triene | 0.04 | 0.04 | 0.04 | 1 | 0.004 | 0.038 | | | 1 | 0.032 | 0.036 |
| Conjugated Tetraene | 0.007 | 0.007 | 0.01 | | 0.009 | 0.009 | | | 1 | 0.008 | 0.003 |
| Total % Conjugated | 0.207 | 0.217 | 0.22 | 0.22 | 0.194 | 0.222 | 0.222 | | 1 | 0.211 | 0.028 |
| % Arachidonic | 0.02 | 0.03 | 0.01 | 0.01 | 0.02 | 0.00 | 0.02 | | | 0.013 | 0.03 |
| % Linolenic | 7.07 | 7.15 | 7.35 | 7.32 | 6.70 | 7.38 | 7.45 | | | 7.20 | 0.7 |
| % Linoleic | 56.4 | 56.7 | 56.2 | 56.3 | 55.6 | 56.79 | 56.14 | | - 1 | 56.30 | 1.2 |
| % Oleic | 15.5 | 14.7 | 15.8 | 15.8 | 17.6 | 13.24 | 14.24 | | | 15.27 | 4.4 |
| % Saturated | 16.4 | 16.8 | 16.0 | 16.0 | 15.5 | 17.96 | 17.52 | | 1 | 16.60 | 2.5 |
| Total % Acids | 95.597 | 95.597 | 95.58 | 95.65 | 95,614 | 95.592 | 95.592 | | | 95.594 | |
| Method 2, AOCS Cd 7-48 using | 50.551 | 30.001 | 00.00 | 55,05 | 33.014 | 00.002 | 30.002 | | | 00.00* | •••• |
| Revised Constants | | | | | | | | | | | |
| Total % Conjugated | 0.204 | 0.214 | 0.22 | 0.22 | 0.194 | 0.222 | 0.222 | | i | 0.210 | 0.028 |
| % Arachidonic | 0.04 | 0.214 | 0.00 | 0.00 | 0.005 | 0.00 | 0.01 | | | 0.016 | 0.06 |
| % Linolenic | 7.30 | 7.36 | 7.75 | 7.71 | 7.07 | 7.77 | 7.88 | | | 7.55 | 0.8 |
| % Linoleic | 52.5 | 52.8 | 52.3 | 52.4 | 51.8 | 52.86 | 52.25 | | ı | 52.42 | 1.1 |
| % Oleic | 22.7 | 21.7 | 22.4 | 22.5 | 24.1 | 19.98 | 20.88 | | | 22.04 | 4.1 |
| % Saturated. | 22.7 | | 12.9 | $\frac{22.5}{12.8}$ | | 14.77 | 14.37 | | | 13.38 | 2.4 |
| Total % Acids | 95.644 | 13.5 | 95.57 | 95.63 | 12.4 | 95.602 | 95.612 | | | 95.616 | |
| Method 3, AOCS Cd 7-48 using 45-Minute | 95.044 | 95.634 | 95.57 | 95.05 | 95,569 | 95.002 | 95.012 | | | 95.010 | •••• |
| Isomerization and Revised Constants | | | | | | | | | | | |
| | 0.004 | | 0.00 | 0.00 | 0.101 | 0.000 | 0.222 | 0.222 | 0.222 | 0.210 | 0.028 |
| Total % Conjugated | 0.204 | 0.214 | 0.22 | 0.22 | 0.194 | 0.222 | 0.222 | 0.00 | 0.00 | 0.210 | 0.04 |
| % Arachidonic | 0.03 | 0.04 | 0.00 | 0.00 | 0.002 | 0.00 | | | | | |
| % Linolenic | 7.49 | 7.41 | 7.48 | 7.23 | 7.16 | 7.85 | 7.70 | 7.67 | 7.74 52.58 | 7.53 | $0.7 \\ 1.2$ |
| % Linoleic | | 52.9 | 52.3 | 52.3 | 52.5 | 53.50 | 52.68 | 52.37 | | 52.64 | |
| % Oleic | 21.9 | 21.5 | 23.4 | 24.1 | 22.5 | 18.43 | 20.53 | 21.28 | 20.64 | 21.59 | 5.7 |
| % Saturated | | 13.5 | 12.2 | 11.8 | 13.2 | 15.59 | 14.46 | 14.06 | 14.42 | 13.63 | 3.8 |
| Total % Acids | 95.624 | 95.564 | 95.60 | 95.65 | 95.556 | 95.592 | 95.592 | 95.602 | 95.602 | 95.608 | **** |

TABLE V Chemical Analyses on Oils

| | | | | Chemical | Analyses | on Ons | | | | | | | | |
|---------------------------------------|----------------------------------|------------------------------|----------------------------------|-------------------------------------|------------------------------|----------------------------------|----------------------------------|----------------------------------|-------------------|------------------------------|------------------------------|------------------------------|------------------------------|--|
| | | | | Iodine | Value | | Thiocyanog | | | | | gen Value | | |
| V V V V V V V V V V V V V V V V V V V | Lab. 2 | | Lab. 3 | Lab. | 4 | Lab. 1 | Average | | Lab. 2 | | Lab. | 4 A | verage | |
| Cottonseed Oil No. 1 | 112.1 110.5 136.7 136.6 | | 110.2 108.6 135.4 135.2 | | .9 .3 .2 .4 | 111.9 110.1 136.0 136.0 | | 111.3 109.6 135.8 135.8 | | 68.5 68.7 85.1 85.0 | | 1 3 5 5 9 | 68.8 69.2 84.8 85.0 | |
| | | % Satura | ted Acids | (as % of ? | Potal Acids |) | Compos | ition of (| }lycer | ides l | y Satura | ted Acids a | nd T. C.V. | |
| | Bert | ram | Crysta | llization | Twitch- | Average | I.V. | T.V. | T.V. % I | | % Lino- lein | % Olein | % Sat. | |
| | Lab. 3 | Lab. 4 | Lab, 3 | Lab. 1 | Lab. 2 | | Ì | | 1 | | 10111 | | | |
| Cottonseed Oil No. 1 | 26.4 26.0 15.2 15.4 | 25.0 24.5 15.1 14.4 | 25.2 24.4 14.3 14.4 | 26.6 25.7 13.8 14.0 | 23.9 23.4 12.6 12.0 | 25.4 24.8 14.2 14.0 | 111.3 109.6 135.8 135.8 | 68.8 69.2 84.8 85.0 | 1.7 1.9 10. | 0 61 | 50.2 47.4 49.6 49.2 | 22.6 25.9 25.6 26.1 | 25.4 24.8 14.2 14.0 | |
| | C | ompositio | C.C.V. Only | lenin) Composition 95.6% Acid Basis | | | | | | | | | | |
| | 1.V. | T. V | | ino- enin | Lino- lein | Olein | Sat. | % Li len | | | Lino- eic | % Oleic | % Sat. | |
| Cottonseed Oil No. 1 | 111.3 109.6 135.8 135.8 | 68. 69. 84. 85. | 2 6 | 0.00 | 52.5 49.9 | 23.7 27.0 | 23.8 23.2 | 0. 0. 10. | 0 | 4 | 0.2 7.7 7.4 7.0 | 22.7 25.8 24.6 25.0 | 22.8 22.2 13.5 13.4 | |

^{*} Corrected by thio analyses on solid acids.

and average thiocyanogen value as determined by all of the laboratories reporting would give a better overall value for comparison with the spectrophotometric results. Note that the substitution of the values 25.4 and 24.8 for the saturated acids found in the two cottonseed oil samples, when substituted into the equation for calculating the composition of the fatty acids by the thiocyanogen method, results in a percentage of linolenic acid found of 1.79 and 1.90. If the thiocyanogen equation, where linolenic acid is assumed to be absent, is used the calculated apparent saturated acids are 23.8 and 23.2%.

Table VI shows that in the case of the soybean oils the thiocyanogen method gives higher results for linolenic acid than does the spectrophotometric method of analysis. This is probably due to the fact that the saturated acids as determined by the Bertram and crystallization procedures are on the high side. This however remains to be proven, but the indication is clear with regard to the cottonseed oil samples as discussed above. The revised constants, whether used on samples isomerized for 25 minutes or 45 minutes, give results in much closer agreement with the thiocyanogen results. The wide discrepancy often encountered between the two methods, especially in the determination of oleic acid, is largely eliminated.

The effect of using the equation $k_2 = k_{233} - k_0$ instead of $k_2 = k_{233} - 0.029 - 0.52$ P is not clear-cut

from the results obtained on the 4 cooperative samples. The only effect is to reduce the amount of conjugated diene by about 0.3%. This is of course of no significance on samples where the conjugated diene content does not exceed 0.5%. Since the equation $k_2 = k_{233} - k_0$ is theoretically right and since very little change is introduced in the analysis of the common vegetable oils, the committee believes that the use of the revised equation is justifiable and advisable.

The maximum amount of conjugated or non-conjugated arachidonic acid in any of the 4 oils examined was .03%. Most of the collaborators found no arachidonic acid either conjugated or non-conjugated. In the light of these findings and the generally accepted knowledge that vegetable oils have no arachidonic acid present, the committee feels that measurements in the tetraenoic region $(k_{310} \text{ to } k_{322})$ are unnecessary and undesirable.

The committee feels that the use of background corrections in the calculation of substantial amounts of arachidonic, linolenic, and linoleic acids is generally unnecessary. In determinations where only small amounts of the acids are involved, background corrections are essential. It is hoped that future committee work will enable us to determine those cases in which background corrections may safely be elimi-

TABLE VI Comparison of Spectrophotometric and Other Analyses

| · | Arachi- donic Acid | | Total Tri | ene Acids | | Total Diene Acids | | | | | |
|----------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|--|--|
| | | T.C.V. | Sp | ectrophotome | trie | T. C.V. | Sp | ectrophotome | trie | | |
| | | T.C.V. | Method 1 | Method 2 | Method 3 | T. C. V. | Method 1 | Method 2 | Method 3 | | |
| Cottonseed Oil No. 1 | None None None None | 0.00 0.00 10.1 10.2 | 0.0 0.0 7.2 7.2 | 0.0 0.0 7.5 7.6 | 0.0 0.0 7.5 7.5 | 50.2 47.7 47.4 47.0 | 55.3 53.9 56.6 56.5 | 51.8 50.4 52.7 52.6 | 52.2 50.4 53.6 52.8 | | |
| | | | Total Ol | eic Acids | | | Total Saturated Acids | | | | |
| | | m 0 T | Sp | ectrophotomet | rie | TO CAY | Spectrophotometric | | | | |
| | | T.C.V. | Method 1 | Method 2 | Method 3 | T.C.V. | Method 1 | Method 2 | Method 3 | | |
| Cottonseed Oil No. 1 | | 22.7 25.8 24.6 25.0 | 12.4 13.5 15.2 15.3 | 19.5 20.5 21.9 22.0 | 18.7 20.4 20.1 21.6 | 22.8 22.2 13.5 13.4 | 27.9 28.2 16.6 16.6 | 24.3 24.7 13.4 13.4 | 24.7 24.8 14.4 13.6 | | |

nated. In some instances the use of a background correction may lead to erroneous results.

As a result of the work herein reported the Spectroscopy Committee recommends specific changes in AOCS Tentative Method Cd 7-48, to wit:

a) Change, on page 2, (1)-b under section A, from Rotate the sensitivity knob about 3 counter clockwise turns and keep in this approximate position for all measurements. Use the slit width adjustment for balancing the instrument and the sensitivity knob for final adjustment.

In making absorption measurements, the sensitivity control is usually set at 3 counter clockwise turns from its clockwise limit; the slit width control is usually used as a coarse adjustment for balancing the instrument and the sensitivity control for final adjustment. In general, slit widths are critical in this method only for absorption measurements at 262, 268, and 274 mµ. When making absorption measurements on isomerized samples in this region, slit widths at the final balancing adjustment must be 0.8 to 0.9 millimeters.

b) Change, on page 6, (a)-4 under section D, from Take spectral density readings at 322, 316, 310, 274, 268, 262, and 233 m μ .

Take spectral density readings at 322, 315, 308, 274, 268, 262, and 233 m μ . .

- c) Change, on page 6, (b)-4 under section D,

 Insert at the end of the first sentence, "The temperature should be checked by an accurate thermometer standardized at frequent intervals.'
- d) Change, on page 7, (a)-1 under section E, from Calculate the specific extinction coefficient k for each wavelength recorded in D(a), 5, using subscripts 322, 316, 310, etc., to designate each individual k.

Calculate the specific extinction coefficient k for each wavelength recorded in D(a), 5, using subscripts 322, 315, 308, etc., to designate each individual k.

e) Change, on page 7, (a)-2 under section E, from Specific extinction coefficient at 233 mm corrected for absorption by COOR and C = C groups = $k_2 = k_{233} - 0.029 - 0.052 P$ P = estimated proportions of oleic acid (decimal fraction)

to Specific extinction coefficient at 233 mu corrected for absorption by acid or ester groups = $k_2 = k_{223} - k_0$ $k_o = 0.07$ for esters, 0.03 for soaps and fatty acids

f) Change, on page 8, (a)-4 under section E, from Specific extinction coefficient at 316 mµ corrected for background absorption = $k_4 = 2.5 [k_{316} - 1/2 (k_{310} +$ k₃₂₂)]

Specific extinction coefficient at 315 mm corrected for background absorption = $k_4 = 2.5 [k_{315} - 1/2 (k_{308} +$

- g) Change, on page 8, (b)-1 and (b)-3 under section E, from (1) Conjugated diene, $\% = C_2 = 0.87 \text{ k}_2$ and
 - (3) Conjugated tetraene, $\% = C_4 = 0.49 \text{ k}_4$
 - (1) Conjugated diene, $\% = C_2 = 0.84 \text{ k}_2$ and (3) Conjugated tetraene, $\% = C_* = 0.45 \text{ k}_*$
- h) Change, on page 8, (c) 2 under section E, from $k'_2 = k'_{233} - k_{233}$ to

 $k'_2 = k'_{233} - k_2 - 0.03$

i) Change, on page 8, (c)-3 under section E, from $k'_{3} = 4.1 [k'_{268} - 1/2 (k'_{262} + k'_{274})] - k_{3}$

 $k'_3 = 4.03 [k'_{268} - 1/2 (k'_{262} + k'_{274})] - k_3$ j) Change, on page 8, (c)-4 under section E, from $k'_4 = 2.5 \left[k'_{316} - 1/2 \left(k'_{310} + k'_{322}\right)\right] - k_4$

 $k'_{4} = 2.06 \left[k'_{315} - 1/2 \left(k'_{308} + k'_{322}\right)\right] - k_{4}$

- k) Change, on page 8, (d)-1, 2 & 3 under section E, from 1. Linoleic acid, % = X = 1.16 k'_2 1.33 k'_3 +
 - 2. Linolenic acid, $\% = Y = 1.88 \text{ k}_3 4.43 \text{ k}_4$
 - 3. Arachidonic acid, $\% = Z = 4.43 \text{ k}'_4$
 - to1. Linoleic acid, $\% = X = 1.086 \, k'_2 - 1.324 \, k'_3 +$ 0.40 k'₄
 - 2. Linolenic acid, $\% = Y = 1.980 \text{ k}'_3 4.92 \text{ k}'_4$
 - 3. Arachidonic acid, % = Z = 4.69 k'
- 1) Insert, on page 8, at the end of the calculation immediately following (e)-5 under Section E, the following:
 - 6. In the analysis of normal vegetable oils all measurements in the tetraenoic region k₃₀₈ to k₃₂₂ may be omitted and in the calculations k_4 and $k'_4 = 0$. For such oils, determination of conjugated constituents may be omitted if desired. In this case procedure (a) under section D, and calculations (a) under section E would be omitted; also on page 8, section E it would be assumed that $k'_2 = k'_{23}$ under (c)-2, and that k_3 and k_4 = 0 under (c)-3 and (c)-4.

The committee expects to continue next year the investigation of the composition of additional oils both of a vegetable nature and those containing arachidonic acid. It hopes to receive from the Uniform Methods Committee and from members of the Society at large suggestions as to the future direction of its activities. In the event that such suggestions are not forthcoming the committee will proceed with such ideas as originate within itself, or will disband if the Society so orders.

B. A. BRICE F. R. SENTI R. T. O'CONNOR R. C. STILLMAN, chairman A. L. LINGARD

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3. Lemon, H. W., Can. J. Res., F., 22, 191 (1944).
4. Presented at the AOCS Meeting in New York City, November 1948. Furnished to the committee by B. A. Brice.

Evaluation of Safflowerseed Oil in Edible Fat Products¹

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CAFFLOWER has been known as an oilseed crop in the Middle East and Northern Africa since ancient times. It has generally been grown only on a relatively small scale for local use and the oil seldom enters world trade.

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² Trainee sponsored by Danish Government and the Dansk Sojakagefabrik A/S., Copenhagen.

³ One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

Experimental tests with this crop in the United States have been conducted during the past 20 years (7), and in recent years new varieties of safflower have been developed which produce seed of higher oil content and a higher yield of seed per acre than heretofore known varieties (7, 8). Some of these improved varieties are now being grown and processed for oil in Nebraska, Colorado, North Dakota, Montana, and California.