

TABLE II

	Sample No. 1	% K ₂ O Sample No. 2	Sample No. 3
Made to contain.....	2.61	2.61	2.53
Laboratory A.....	2.62	2.61	2.54
Laboratory B.....	2.60	2.61	2.50
Laboratory C.....	2.60	2.58	2.54
Laboratory D.....	2.61	2.61	2.54

If both calcium and sulfates are present, results may be low as a result of the formation of a double potassium calcium sulfate. It is possible to separate potassium from as much as 70 times as much sodium. Free acids, other than hydrochloric, may be present, but large quantities produce a precipitate that filters only with difficulty. Perchloric acid will produce a precipitate of KClO₄ which will not be converted to periodate with 30 minutes of stirring.

The presence of large amounts of glycerine, ethanalamines, and similar organic compounds that may nitrate will interfere with the results. The samples, in such cases, should be prepared by ashing.

Summary

The method of determining potassium as KIO₄, as suggested by Willard and Boyle, has been modified and applied to the analysis of soap and caustic lye. The precipitated periodate is completely reduced, and the liberated iodine is titrated with standard thiosulfate. The determination is sensitive to 0.1 mg. of potassium and the analysis of large numbers of samples may be carried out simultaneously in a relatively short time. This, along with the low cost of the re-

agents, makes the method applicable to industrial control work. An extensive literature search was made and the complete list of references is included.

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Report of the Color Committee May, 1949

Introduction

THIS report covers work completed since the 1948 report printed in the *Journal of the American Oil Chemists' Society*, Volume XXVI, No. 2, 45-51, February, 1949. At the meeting of the Color Committee, held in New York in November, 1948, it was decided to continue the investigation of the reproducibility of the Coleman Junior spectrophotometer and to set up a method that could be recommended for adoption by the Uniform Methods Committee of the Society. The work reported herein is aimed at completing this program.

Work Completed

The Committee submitted to 20 collaborators four oil samples and a nickel sulfate solution, together with a proposed spectrophotometric color method. Each collaborator was requested to

- Follow the method as given, except no dilutions should be made
- Report both densities and transmissions
- Be sure the instrument was calibrated exactly
- Use Type B cuvettes
- Read oils at 525 and 550 m μ ., using CCl₄ as a blank
- Read nickel sulfate at 400, 470, 510, 525, 550, and 700 m μ ., using both water and CCl₄ as blanks
- Read Lovibond colors on the oil samples.

The data obtained are shown in Tables I, II, and III. Complete spectral information on the four oils is given in Table IV and Fig. 1. The Barrow-Agee laboratories obtained Lovibond red readings on a large number of oils in process and read spectrophotometric transmissions on the same oils at several wavelengths. These data are plotted in Figs. 2 and 3.

Discussion of the Data

A total of 20 sets of samples were sent out to the Color Committee collaborators. Sixteen reported results using the Coleman Junior spectrophotometer and one using the Coleman Universal Model 11. Agreement was generally good except for Laboratory 6, which was extremely erratic on the nickel sulfate solution. Instruments in Laboratories 11 and 19 appear to be out of wavelength calibration. The instrument in Laboratory 12 gave too high results at all wavelengths for some unexplained reason. Laboratory 13 was slightly high because a 20.5 mm. cell was used for all measurements. All of these results (Table II) are excluded, leaving 12 sets of results, which have been assembled in Table I.

While all of the instruments, except the one in Laboratory 15, were supposed to be calibrated against the instrument maker's didymium filter, instruments in 6, 11, 12, and 19 are out of calibration if the nickel

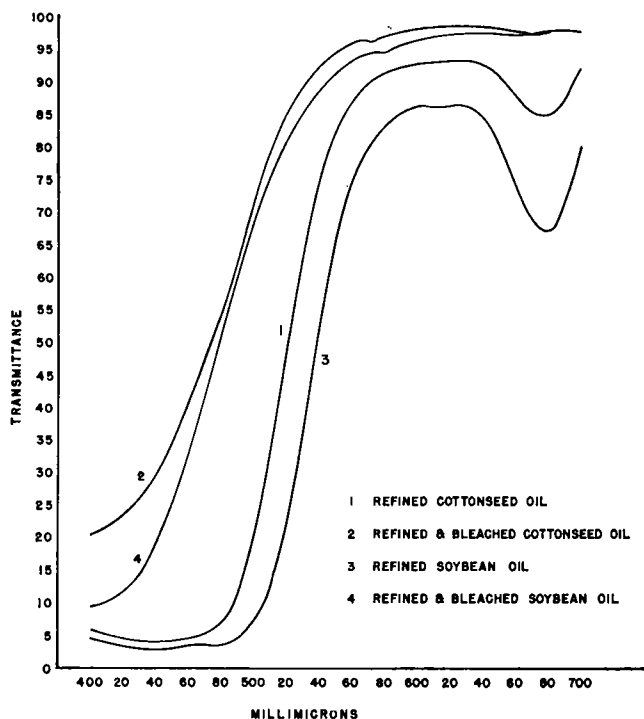


Fig. 1. 1949 A.O.C.S. Oils

sulfate solution is used as a criterion. The results from these laboratories are excluded if the limits, transmission at 470, 510, and 550 millimicrons plus or minus 2%, are used. These limits are

470 millimicrons.....	36.77 to 40.77
510 millimicrons.....	71.41 to 75.41
550 millimicrons.....	52.39 to 56.39

It is interesting to note that all of the excluded instruments are out at 470 millimicrons.

The nickel sulfate submitted for color readings was prepared as follows: 200 grams of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ was dissolved in water. Ten ml. of concentrated HCl were added, and the total volume was brought to exactly 1,000 ml. at room temperature. By analysis, the solution showed 3.96% nickel by weight. The density of the solution was 1.115 at 24.3°C.

The average readings obtained on the spectrophotometer were very close to those obtained and reported in the 1948 report.

Wavelength	Present Results	Previous Results
400	3.37	3.17
470	38.77	36.94
510	73.41	72.64
525	69.40	69.20
550	54.39	54.39
700	1.62	1.75

Table III shows density values on the nickel sulfate sample and on the oil samples obtained by the 12 accepted laboratories. Averages and standard deviations have been calculated. The standard deviations for spectrophotometric readings at 525 and 550 μm . have been converted to red readings for comparison with Lovibond standard deviations. The comparison is highly favorable for the spectrophotometric readings. Dividing the standard deviation by the actual Lovibond reading shows clearly that up to 10 red from a reproducibility standpoint makes little difference whether readings are obtained at 525 or 550 μm . and that either are better than Lovibond

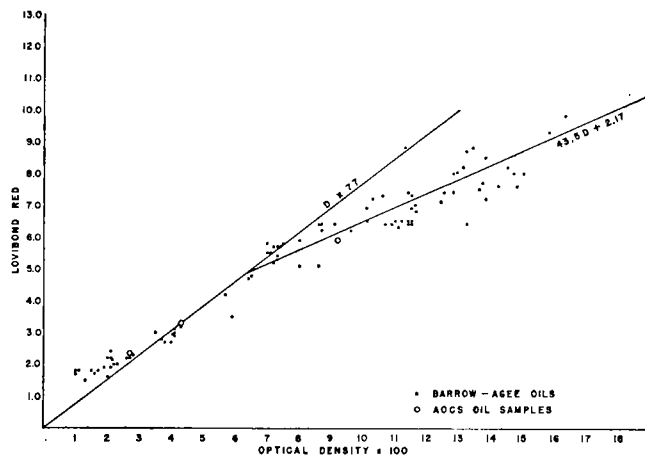


Fig. 2. Lovibond red readings vs. optical density at 550 μm .

red colors. Lovibond red readings are much less reliable at low color values.

In Fig. 2 have been plotted the data obtained in the Barrow-Agee Laboratories showing optical density measurements at 550 millimicrons versus Lovibond red readings. The relationship, Lovibond red = density at 550 μm . times 77, holds reasonably well for refined and bleached oils, but does not hold on refined. The refined oils probably contain a considerable amount of chlorophyll, as indicated in the refined cottonseed and refined soybean oils shown in Fig. 1.

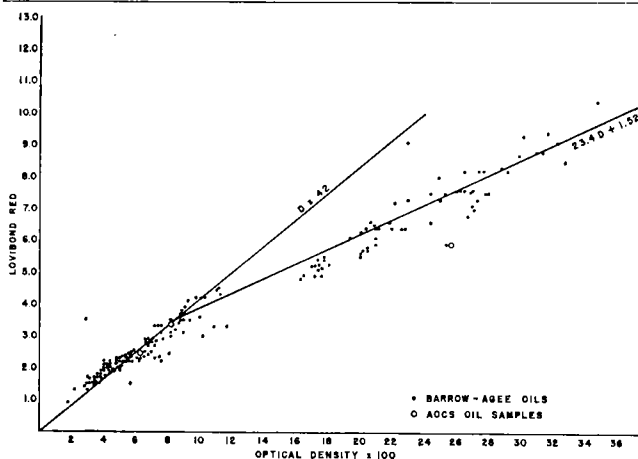


Fig. 3. Lovibond red readings vs. optical density at 525 μm .

If it is desirable to obtain values approximately equal to the Lovibond red values, the following equations may be used:

up to .065 density
 $\text{red} = \text{density} \times 77$

above .065 density
 $\text{red} = 43.5 \text{ density} + 2.17$

In Fig. 3 are shown similar data obtained in the Barrow-Agee Laboratories at 525 millimicrons. Here again, as would be expected, the refined oil colors break away from the relationship, Lovibond red = density at 525 μm . \times 42. The curves required to approximate Lovibond red values are:

up to .085 density
 $\text{red} = \text{density} \times 42$

above .085 density
 $\text{red} = 23.4 \text{ density} + 1.52$

TABLE I
A. O. C. S. Oils and Nickel Sulfate

	Transmissions														Average	Average report 1948	Lovibond red	
	1	2	3	4	5	7	8	14**	15†	16	17	18						
Nickel sulfate																		
400 mμ.....	3.8	3.6	3.3	3.3	2.8	4.0	3.2	3.5	1.7	4.0	3.2	4.0	3.37	3.17	
470 mμ.....	40.1	36.9	39.3	39.0	37.1	39.5	37.0	40.4	39.3	38.8	37.9	37.9	38.77	36.94	
510 mμ.....	74.8	71.5	74.4	73.0	73.0	73.6	75.0	74.0	73.8	71.4	73.8	72.6	73.41	72.64	
525 mμ.....	70.7	69.1	69.5	69.9	69.5	69.0	70.0	69.8	69.7	67.8	68.9	68.9	69.40	69.20	
550 mμ.....	55.4	54.2	54.2	55.0	54.4	55.2	54.7	54.0	55.0	53.7	52.9	54.0	54.39	54.39	
700 mμ.....	1.8	1.2	1.8	1.2	1.3	2.5	1.8	1.3	1.3	2.2	1.8	1.2	1.62	1.75	
Oil No. 1																		
525 mμ.....	54.5	54.7	55.1	55.0	54.0	55.9	55.5	55.0	55.7	58.4	56.5	53.5	55.32	55.32	7.54 ^a	7.54 ^a	7.54 ^a	
550 mμ.....	80.6	80.6	81.0	81.0	80.5	80.0	81.8	81.2	81.7	82.7	81.2	79.3	80.85	80.85	6.19 ^c	6.19 ^c	6.19 ^c	
Lovibond color.....	35/6.3	35/5.8	50/6.4	35/6.0	35/6.2	35/5.8	/5.4	35/6.0	55/5.5	35/5.8	35/6.3	5.88*	5.88*	5.88*	5.88*	5.88*	
Oil No. 2																		
525 mμ.....	86.7	86.8	85.9	86.3	86.1	87.0	87.5	86.9	87.0	86.7	87.0	86.72	86.72	2.65 ^b	2.65 ^b	2.65 ^b	
550 mμ.....	94.2	94.1	92.3	94.0	94.0	94.1	94.5	94.0	94.2	93.7	94.1	93.92	93.92	2.16 ^a	2.16 ^a	2.16 ^a	
Lovibond color.....	27/2.7	20/2.4	25/2.6	24/2.4	25/2.5	25/2.5	/1.7	24/2.4	30/2.7	25/2.5	2.38*	2.38*	2.38*	2.38*	2.38*	
Oil No. 3																		
525 mμ.....	28.9	29.9	30.1	29.1	28.5	31.7	29.2	29.5	30.8	32.6	31.1	27.8	29.93	29.93	13.77 ^a	13.77 ^a	13.77 ^a	
550 mμ.....	68.7	62.7	63.8	62.9	62.9	64.3	64.0	63.8	65.2	64.3	65.0	61.1	63.64	63.64	10.75 ^c	10.75 ^c	10.75 ^c	
Lovibond color.....	70/10.4	35/9.2	70/9.6	70/9.4	35/9.2	70/8.8	/9.6	70/10.0	95/9.8	70/9.0	70/10.1	9.51*	9.51*	9.51*	9.51*	9.51*	
Oil No. 4																		
525 mμ.....	83.1	82.0	82.5	83.0	82.3	83.7	83.5	83.0	83.0	83.7	83.1	81.5	82.87	82.87	3.43 ^b	3.43 ^b	3.43 ^b	
550 mμ.....	91.0	90.2	89.7	90.8	90.6	91.3	91.5	91.1	91.0	90.9	90.8	90.0	90.74	90.74	3.39 ^a	3.39 ^a	3.39 ^a	
Lovibond color.....	35/3.8	30/3.1	35/3.6	34/3.4	34/3.4	34/3.4	/2.6	35/3.5	35/3.3	34/3.4	35/4.0	3.33*	3.33*	3.33*	3.33*	3.33*	

** Type "A" cuvettes. † Instrument uncalibrated. * Average of all colors. ^a 23.4D + 1.52. ^b D × 42. ^c 43.5D + 2.17. ^d D × 77.

TABLE III
A. O. C. S. Oils and Nickel Sulfate

	Densities														Average	σ	σ (red units)	σ × 100 Lov. red
	1	2	3	4	5	7	8	14	15	16	17	18						
Nickel sulfate																		
400 mμ.....	1.420	1.500	1.500	1.875	1.500	1.800	1.5	393	1.77	1.37	1.49	1.573	
470 mμ.....	396	433	407	410	430	404	432	431	405	412	398	421	4118	
510 mμ.....	152	147	130	138	138	135	125	131	132	147	133	139	1352	
525 mμ.....	256	161	160	168	168	163	155	158	157	161	163	163	1599	
550 mμ.....	256	266	269	259	262	258	261	268	260	269	277	267	2643	
700 mμ.....	1.800	1.900	1.800	1.8+	1.80	1.8	1.9	1.70	1.77	1.819	
Oil No. 1																		
525 mμ.....	264	262	260	259	266	254	255	259	253	236	248	271	2573	
550 mμ.....	994	994	1000	991	993	998	987	989	988	983	990	1000	9923	
Lovibond red.....	5.86	
Oil No. 2																		
525 mμ.....	062	063	065	063	065	061	068	061	061	063	061	0630	
550 mμ.....	027	027	036*	028	028	027	024	028	026	028	027	0270	
Lovibond red.....	2.38	
Oil No. 3																		
525 mμ.....	538	525	522	533	542	502	532	530	512	487	505	555	5236	
550 mμ.....	198	204	197	202	201	193	193	197	186	194	188	213	1972	
Lovibond red.....	9.51	
Oil No. 4																		
525 mμ.....	081	086	085	081	083	078	078	081	081	077	080	088	0816	
550 mμ.....	042	045	049	043	043	042	038	041	041	043	043	047	0431	
Lovibond red.....	3.33	

* This value is rejected, probably .026.

TABLE II
A. O. C. S. Oils and Nickel Sulfate

	Transmissions										
	Average Table I	No. 6	No. 9	No. 10	No. 11	No. 12	No. 13	No. 19	No. 20		
Nickel sulfate											
400 m μ	3.37	1.75 ¹ ²	3.6	6.0	3.2	2.5 ²		
470 m μ	38.77	52.50	34.6	46.1	42.0	33.0		No. 6, results erratic, maximum at 525 m μ .
510 m μ	73.41	67.25	73.1	77.2	74.1	72.5		
525 m μ	69.40	72.00	70.3	74.3	69.7	70.0		
550 m μ	54.39	40.50	55.4	62.4	55.1	55.5		No. 11, 4% low at 470 m μ , oil results all low.
700 m μ	1.62	3.25	1.9	2.5	2.35	1.0		
Oil No. 1											
525 m μ	55.32	59.5	52.8	63.1	59.4	51.0		No. 12, all results too high, may be stray light.
550 m μ	80.85	83.25	79.8	86.0	82.2	79.3		
Lovibond color.....	5.88	35/5.8	45/5.1	35/6.1	60/5.7		
Oil No. 2											
525 m μ	86.72	87.5	85.4	89.1	87.6	85.0		No. 13, 20.5-mm. cell, model 11 inst. used.
550 m μ	93.92	94.75	93.6	95.0	94.1	94.0		
Lovibond color.....	2.38	25/2.7	25/1.8	24/2.4	38/2.0		No. 19, results generally low, 22.0-mm. cell.
Oil No. 3											
525 m μ	29.93	32.5	26.5	37.0	33.2	26.0		
550 m μ	63.64	65.25	62.1	71.0	67.1	60.7		Instruments in laboratories 11 and 19 appear to be out of wave length calibration toward the low wave lengths.
Lovibond color.....	9.51	70/9.4	60/8.8	70/10.1	80/9.2		
Oil No. 4											
525 m μ	82.87	84.25	81.2	86.8	83.9	81.2		
550 m μ	90.74	91.50	90.1	93.0	91.3	90.5		
Lovibond color.....	3.33	35/3.5	35/3.0	30/3.0	50/3.0		

¹ Run on Beckmann. ² No report.

Conclusions

From the data discussed above it can be concluded:

1. That a nickel sulfate standard (or other solutions or filters) are desirable for calibrating the Coleman Junior spectrophotometers. The manufacturer's didymium filter alone is insufficient.

2. That reproducibility obtainable on the spectrophotometers is approximately the same at 525 and 550 millimicrons and at either wavelength exceeds that obtainable by Lovibond red readings.

3. That by the use of proper conversion equations red values can be obtained from spectrophotometric readings at 525 or 550 millimicrons, which will closely approximate Lovibond red readings.

Recommendations

It is recommended that the Society, through the Uniform Methods Committee, adopt the following spectrophotometric method for determining the color of fats and oils.

Color Spectrophotometric Method

Definition: This method denotes the color of an oil by determining the optical density at a specified wavelength of light, using a spectrophotometer.

Scope: Applicable to fats and oils.

A. APPARATUS

1. Spectrophotometer—A spectrophotometer capable of adjustment to give the following readings on a standardizing nickel sulfate solution (3), after setting the zero point and after adjusting the 100% transmittance point (0 density) against CCl_4 in a cuvette having the specifications outlined in (2) below.

400 millimicrons	Less than 4% transmittance
470 millimicrons	38.8 ± 2
510 millimicrons	73.4 ± 2
525 millimicrons	69.4 ± 2
550 millimicrons	54.4 ± 2
700 millimicrons	Less than 2%

2. Matched glass cylindrical cuvettes, approximately 21.8 mm. inside diameter; outside diameter approximately 24.5 mm. All cuvettes should check CCl_4 and the nickel sulfate solu-

tion at 525 m μ . within $\pm 0.6\%$ transmittance. The cuvettes should be kept clean and free from scratches.

- Standardizing Nickel Sulfate Solution—Dissolve 200 grams $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ in distilled water. Add 10 ml. of concentrated HCl. Dilute to exactly 1,000 ml. in a volumetric flask. The temperature of the solution should be between 25 and 30°C. The density of the solution at 25°C. should be 1.115 and nickel content must fall between 3.95 and 4.00% nickel by analysis.
- Filter paper—Fine porosity such as E & D No. 192, Whatman No. 12, Reeve-Angel No. 871, or S & S No. 596.

B. REAGENTS

- Carbon tetrachloride—Redistilled if the transmittance differs from distilled water by 0.5% at 400 m μ .

TABLE IV
1949 A. O. C. S. OILS

Wave-length	Oil No. 1		Oil No. 2		Oil No. 3		Oil No. 4	
	Refined cottonseed		Refined and bleached cottonseed		Refined soybean		Refined and bleached soybean	
	Trans.	Density	Trans.	Density	Trans.	Density	Trans.	Density
400.....	5.9	1.23	20.2	.695	4.4	1.35	9.5	1.03
420.....	4.4	1.35	23.3	.630	3.3	1.47	11.4	.940
440.....	3.9	1.40	30.0	.523	3.0	1.52	19.2	.720
460.....	4.3	1.36	40.7	.390	3.3	1.50	33.3	.480
470.....	5.2	1.28	47.5	.324	3.5	1.50	42.3	.374
480.....	6.5	1.19	54.4	.265	3.5	1.45	51.0	.294
490.....	10.8	.970	62.1	.209	4.3	1.37	59.5	.226
500.....	20.0	.700	70.7	.152	6.5	1.18	68.1	.168
510.....	33.3	.480	78.5	.105	12.5	.910	75.0	.126
520.....	48.3	.318	84.3	.074	22.6	.645	80.2	.096
525.....	55.2	.258	86.8	.062	28.7	.543	82.3	.085
530.....	63.2	.200	89.4	.050	36.8	.435	85.3	.069
540.....	74.6	.128	92.4	.035	51.7	.288	88.6	.054
550.....	82.3	.085	94.5	.026	64.5	.192	91.2	.042
560.....	86.7	.062	96.0	.019	73.5	.134	93.2	.032
570.....	89.5	.049	96.5	.017	79.4	.100	94.4	.026
580.....	91.2	.041	97.0	.015	83.1	.081	94.8	.024
590.....	92.3	.036	97.6	.012	85.4	.069	95.9	.019
600.....	92.9	.033	97.9	.011	86.1	.066	96.5	.017
610.....	93.0	.032	98.2	.009	86.2	.066	96.9	.015
620.....	93.4	.031	98.3	.008	86.4	.064	97.3	.013
630.....	93.4	.031	98.3	.008	86.4	.064	97.5	.012
640.....	92.9	.033	98.3	.008	84.7	.072	97.3	.013
650.....	90.8	.042	98.3	.008	80.6	.095	97.4	.013
660.....	88.1	.057	98.0	.010	74.5	.128	97.3	.014
670.....	85.2	.070	97.5	.012	69.1	.162	97.3	.014
680.....	85.0	.071	97.8	.011	67.1	.174	97.7	.012
690.....	86.7	.062	97.9	.011	71.3	.147	97.9	.011
700.....	92.0	.046	97.8	.011	79.9	.097	97.8	.011

C. PROCEDURE

1. The sample must be absolutely clear. If not, filter through a specified paper at a temperature of at least 10°C. above the melting point of the fat. The sample should not be held melted longer than necessary since darkening may occur.
2. Turn on the spectrophotometer and allow at least a 20-minute warm-up period before standardizing or making any measurement.
3. Set the wavelength scale to the desired wavelength.
4. Recheck the zero reading of the instrument and, with a cuvette filled with CCl_4 in the instrument, set the 100% transmission point exactly.
5. Fill a cuvette with the standardizing nickel sulfate solution and read the transmittance at 400 μ . Repeat 3 and 4 at 470, 510, 525, 550, and 700 millimicrons. The readings must fall within the limits prescribed, or the instrument should be adjusted to give the correct response.
6. Fill a cuvette with the sample using a sufficient amount of oil to insure a full column in the light beam.
7. Place the filled tube in the instrument and read the optical density to the nearest 0.001 at 525 millimicrons.

REPORTING:

1. Report the optical density multiplied by 1,000.

(Red color = density \times factor)

Factors: up to .085 density

red = density \times 42

above .085 density

red = 23.4 density + 1.52

Special instrument scales for reading red colors directly may be used.

NOTE: The above report, which was prepared by a subcommittee, was approved by a majority vote of the Oil Color Committee and submitted to the Uniform Methods Committee for action. That committee recommended the spectrophotometric method for vegetable oil color measurement at the convention of the A.O.C.S. in New Orleans, and by unanimous vote of the Society it was adopted as a tentative method.

G. WORTHEN AGEE, chairman.

Nutrition in Relation to the Glyceride Oils*

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WHEN viewed in the light of their industrial and physiological importance, the oils and fats have received far less research consideration than they deserve. A scientist, who has worked with oils and fats, seldom has any difficulty in seeing opportunities for research on their nutritive properties that he would like to see explored. But an executive, who is responsible for managing an industrial enterprise, or a layman, who simply wants to make a contribution to the betterment of mankind, may have more difficulty in gaining a comparable degree of enthusiasm. There is constant need to focus convincing arguments upon specific problems that offer promise.

First of all, I would urge the basic principle that those who work toward the solution of practical problems in agriculture, in the food industry, and in other areas where fats and oils are used, could approach many of their respective tasks more efficiently if chemists could discover the reactions by which the various types of fats are formed. For example, no one knows how linolenic, oleic, ricinoleic, or eleostearic acids arise from simpler intermediates. In an empirical way it is known that carbohydrates are converted to fats in both plants and animals, but little is known of the intermediate steps. Economic gains from breeding farm crops with higher oil yields can be seen readily, as in the case of soybeans and corn. The geneticist could work more efficiently if the reactions he is seeking to accomplish were known.

The mechanism by which carbohydrates are converted to fats in the animal body is of frequent con-

cern to scientists who are interested in food. Those who are dealing with human health are especially interested in the changes because the key to several medical problems lies at the crucial point of carbohydrates-to-fat conversion in the human body. For example, there is much evidence to support the current concept of physicians and bio-chemists that in the diabetic patient most of the acute or chronic damage to the body is caused by the accumulation of intermediate fragments of fatty acids. At one time the injuries were thought to be associated almost solely with the high sugar content in the tissues of diabetic patients, but current evidence points chiefly toward the fats.

Additional illustrations of the importance of gaining further knowledge of fat metabolism come from observations on nutrients other than fats. Several acute and chronic diseases are characterized by an initial disturbance in fat deposits. The first evidence that something is wrong may be seen in the form of abnormal deposits of glyceride fats within the cells. Several vitamin deficiencies, for example, are characterized by such a change. The chemist who attempts to work with the physician or veterinarian in gaining a clue to the mechanisms that have become disturbed when the fat droplets appear in the kidneys and liver, scarcely has enough information about the origin or transport of fat to make a helpful suggestion.

ONE may well ask, "Why should a food scientist be concerned with this problem?" There are numerous answers, but perhaps one will suffice. Several years ago Wendell Griffith of St. Louis University,

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