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.
to
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 mμ . . . .
to
Take spectral density readings at 322, 315, 308, 274, 268, 262, and 233 mμ . . . .

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.
to
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 mμ 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 mμ corrected for absorption by acid or ester groups =
$k_2 = k_{233} - k_o$
$k_o = 0.07$ 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 = $k_4 = 2.5\ [k_{316} - 1/2\ (k_{310} + k_{322})]$
to
Specific extinction coefficient at 315 mμ corrected for background absorption = $k_4 = 2.5\ [k_{315} - 1/2\ (k_{308} + k_{322})]$

g) *Change,* on page 8, (b)-1 and (b)-3 under section E, from
(1) Conjugated diene, % = $C_2 = 0.87\ k_2$ and
(3) Conjugated tetraene, % = $C_4 = 0.49\ k_4$
to
(1) Conjugated diene, % = $C_2 = 0.84\ k_2$ and
(3) Conjugated tetraene, % = $C_4 = 0.45\ k_4$

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
$k'_3 = 4.1\ [k'_{268} - 1/2\ (k'_{262} + k'_{274})] - k_3$
to
$k'_3 = 4.03\ [k'_{268} - 1/2\ (k'_{262} + k'_{274})] - k_3$

j) *Change,* on page 8, (c)-4 under section E, from
$k'_4 = 2.5\ [k'_{316} - 1/2\ (k'_{310} + k'_{322})] - k_4$
to
$k'_4 = 2.06\ [k'_{315} - 1/2\ (k'_{308} + k'_{322})] - k_4$

k) *Change,* on page 8, (d)-1, 2 & 3 under section E, from
1. Linoleic acid, % = X = $1.16\ k'_2 - 1.33\ k'_3 + 0.09\ k'_4$
2. Linolenic acid, % = Y = $1.88\ k'_3 - 4.43\ k'_4$
3. Arachidonic acid, % = Z = $4.43\ k'_4$
to
1. Linoleic acid, % = X = $1.086\ k'_2 - 1.324\ k'_3 + 0.40\ k'_4$
2. Linolenic acid, % = Y = $1.980\ k'_3 - 4.92\ k'_4$
3. Arachidonic acid, % = Z = $4.69\ k'_4$

l) *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 k_{308} to k_{322} 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 $k'_2 = k'_{233}$ 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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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 Products[1]

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SAFFLOWER 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.

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.

[1] Presented at the 24th Fall Meeting of the American Oil Chemists' Society, San Francisco, Calif., Sept. 26-28, 1950.
[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.

Safflowerseed oil is classed as a drying oil and serves as a vehicle for paints, varnishes, and enamels, and in the production of oil-modified alkyd resins. Although the oil has, for many years, been used locally for edible purposes, especially in India, little information is found in the technical literature concerning the effect of processing on its utility as an edible product. Schenderowitsch (21) reported that safflowerseed oil which had been refined, bleached, and hydrogenated to a melting point of 34-36°C. was comparable to an inferior grade of hardened sunflowerseed oil for the production of margarine.

Various investigators have reported data for the characteristics (5, 6, 11, 12, 14, 17) and fatty acid composition (5a, 6, 11-15, 17, 23, 24, 26) of safflowerseed oils of different origins. The range of these values is included in the tables of similar data reported by the present authors.

Preparation and Extraction of the Seed

The seed used in the present investigation was grown in Nebraska and Colorado from varieties designated as Nebraska 852, Nebraska 55, Indian, and Nebraska 804-32. The seed of the three first-mentioned varieties was dehulled in a Bauer mill, and the hulls separated by screening on an 8-mesh screen. The meats containing some hulls which were not separated by screening were flaked, charged into a batch extractor (19), and extracted with commercial hexane. The extraction was carried out for about 8 hours at 38°C. with approximately 4 gallons of solvent per pound of flaked meats. The miscella was concentrated at 82°C. under partial vacuum in a current of carbon dioxide. The meal was first air-dried followed by drying in an oven at 50°C. Seed of Nebraska 804-32 was flaked without prior hulling and extracted as described for the other varieties.

The composition of the whole seed, hulls, and extracted meals are given in Table I.

On a moisture-free basis the oil content of the four varieties ranged from 28 to 35% and the protein (N × 6.25) from 12 to 21%. The extracted meals were low in oil content (0.11 to 0.36%) and contained 23 to 37% protein (N × 6.25) on a moisture-free basis. However considerable oil was lost as a result of the inability to separate cleanly the meats and hulls, a problem which has been noted and commented on by others (20).

TABLE I

Composition of Safflowerseed, Hulls, and Extracted Meal [a]

Constituent	Variety			
	Nebraska 852	Nebraska 55	Indian	Nebraska 804-32
Seed				
Moisture,[b] %	5.66	7.84	7.39	6.30
Oil, %	30.55	35.29	28.25	29.63
Nitrogen, %	3.07	1.97	2.85	3.40
Protein (N × 6.25), %	19.19	12.31	17.79	21.25
Ash, %	2.77	2.76	3.04	3.26
Potassium, %	0.60	0.62	0.56	0.65
Phosphorus, %	0.53	0.47	0.54	0.66
Calcium, %	0.19	0.17	0.16	0.22
Crude fiber, %	28.7	29.3	32.8	27.6
Total sugar (calcd. as invert.), %	1.70	1.14	1.52	1.47
Hulls				
Moisture,[b] %	8.46	6.56	7.86	.....
Oil, %	7.78	18.33	7.42	.....
Nitrogen, %	1.07	1.04	0.92	.....
Protein (N × 6.25), %	6.69	6.50	5.75	.....
Extracted meal				
Moisture,[b] %	8.26	7.62	7.15	8.72
Oil, %	0.36	0.22	0.19	0.11
Nitrogen, %	6.01	3.69	6.00	5.35
Protein (N × 6.25), %	37.53	23.06	37.50	33.43

[a] Data supplied by the Analytical and Physical Division. Percentage of moisture is reported on an as-received basis. All other analyses have been calculated to a moisture-free basis.
[b] Original moisture.

Characteristics and Composition of Oils

The official methods of the American Oil Chemists' Society (1) were used in determining the physical and chemical characteristics of the oils with the following exceptions. The percentage of unsaponifiable matter was determined by the method of the Society of Public Analysts (9), thiocyanogen values by the method described by Lambou and Dollear (16), and hydroxyl numbers by the method of West, Hoagland, and Curtis (25). Saturated fatty acids were determined by the Bertram oxidation method as modified by Pelikan and von Mikusch (18) except that sintered glass filter sticks were used for filtering the magnesium soaps. The results of these determinations are given in Table II together with the range of values previously reported in the literature for the same characteristics.

TABLE II

Characteristics of Safflowerseed Oils

Characteristics	Variety				Literature values
	Nebraska 852	Nebraska 55	Indian	Nebraska 804-32	
Specific gravity, 25°/25°	0.9206	0.9215	0.9211	0.9211	0.9184-0.9243
Refractive index, n_D^{40}	1.4690	1.4692	1.4690	1.4690	1.4744-1.4750[a]
Free fatty acids, %	0.67	4.84	0.55	0.52	0.5-5.8[b]
Hydroxyl number	3.7	4.0	6.0	2.9	2.0-12.5[c]
Saponification value	189.7	191.7	191.3	191.3	186.2-192.8
Unsaponifiable matter, %	0.41[d]	0.90	0.77	1.02	0.52-0.96
Iodine value	144.3	149.2	144.8	144.5	142.4-150.1
Thiocyanogen value	82.5	86.0	84.0	85.5	82.2-86.2
Saturated acids, %	8.2	7.4	7.9	8.2	5.5-7.9

[a] Determined at 25°C. [b] Acid values. [c] Acetyl value. [d] Determined on alkali refined oil.

The glyceride composition of the four safflowerseed oils calculated from ultraviolet absorption data, using the revised constants of Swain *et al.* (22) for linoleic acid and from iodine and thiocyanogen values, are given in Table III together with the range in composition previously reported in the literature.

A considerable range in values has been reported for the fatty acid composition of safflowerseed oils as indicated in Table III. The results of the present authors agree with the higher values reported for

TABLE III

Composition of Safflowerseed Oils

	Percentage composition calculated as glycerides						
	Linoleic		Oleic		Saturated		
	A[a]	B[b]	A[a]	B[b]	A[a]	B[b]	C[c]
Nebraska 852	76.4	74.2	13.8	17.1	9.8	8.0	8.6
Nebraska 55	78.1	80.5	16.0	10.6	5.8	8.5	7.7
Indian	75.2	75.1	16.9	16.5	7.9	8.1	8.3
Nebraska 804-32	72.9	74.6	21.1	17.0	6.0	8.0	8.6
Literature values[d]	39-79		13.4-37.6		4.8-18.8		

[a] Calculated from iodine and thiocyanogen values.
[b] Calculated from ultraviolet absorption data using the revised constants of Swain *et al.* (22) for linoleic acid.
[c] Determined by a modified Bertram oxidation method.
[d] Various authors have reported the presence of 0.04 to 5.7% linolenic acid in safflowerseed oils of different origins.

TABLE IV
Characteristics, Composition, and Stability of Hydrogenated Safflowerseed Oil

Hydrogenated oil sample number	Iodine value	Thiocyanogen value	Percentage composition calculated as glycerides							Keeping time. A.O.M., hrs.
			Linoleic		Oleic		Saturated			
			A[a]	B[b]	A[a]	B[b]	A[a]	B[b]	C[c]	
1	115.1	82.5	40.0	33.8[d]	52.3	54.1	6.8	6.2	9.0	....
2	87.9	81.5	7.4	6.4	87.3	89.0	5.3	4.5	10.1	27
3	82.8	79.8	3.2	2.4	89.9	90.9	6.9	6.4	11.1	36
4	78.0	77.7	0	0.3	91.0	90.0	9.0	9.6	13.2	52
5	73.3	73.5	0	0.07	86.8	85.0	13.2	14.8	16.8	124
6	68.7	69.0	0	0.02	81.6	79.8	18.7	20.2	22.5	ca. 70[e]

[a] Calculated from iodine and thiocyanogen values. [b] Calculated from ultraviolet absorption data. [c] Determined by a modified Bertram oxidation method. [d] Also contained 6% conjugated diene. [e] Sample No. 6 had an abnormally poor stability, possibly because of poor deodorization.

linoleic acid, the lower values for oleic acid, and intermediate values for saturated acids. The compositions calculated from spectrophotometric data and from the iodine and thiocyanogen values agree fairly well. The content of saturated fatty acids determined independently by the Bertram oxidation method more nearly agree with the values calculated from the spectrophotometric method. Linolenic acid was not detected in any of the oils.

Processing

A sample of the crude oil from Nebraska 852 was refined by the official method of the American Oil Chemists' Society for solvent-extracted soybean oil. The refining was normal in all respects except that the soap stock was partly liquid even at 15°C. Losses of 3.3% and 3.1% were obtained on refining with 87.5% and 66.6%, respectively, of the maximum 14° Bé sodium hydroxide. The refined oil color was the same in both cases, namely, 35 yellow and 3.5 red Lovibond units.

Bleaching with 6% of official A.O.C.S. bleaching earth produced an oil with a Lovibond color of 3.0 yellow and 0.5 red units.

Approximately 36 pounds of the same oil were refined in a stainless steel refining kettle under the same conditions employed in the refining loss determination with the weaker caustic (66.6% of maximum 14° Bé NaOH). The oil was allowed to stand over-night to settle the soap stock, and the following day the refined oil was drawn off, filtered, and bleached by heating in an open kettle for 20 minutes at 105°C. with 4% of a commercial activated bleaching clay. The oil, which was filtered immediately after bleaching, had a Lovibond color of 3.0 yellow and 0.4 red units.

A portion of the bleached oil was deodorized at 215°C. for two hours at 1 mm. pressure of mercury. The oil was cooled to 40°C. before exposure to air, at which time it was completely bland with respect to odor and flavor. However within a few hours the oil developed an off-flavor which may best be described as "rubbery." The off-flavor increased perceptibly for several days, after which the flavor remained relatively unchanged.

Another batch of the same oil was deodorized after the addition of 0.01% of citric acid in the form of a 10% solution in ethanol. The same sequence of off-flavor development occurred, but at a slightly slower rate. The change in flavor was particularly affected by light as indicated by the fact that a sample of freshly deodorized oil exposed to light from a north window developed a "rubbery or polymerized" flavor within 15 to 30 minutes.

The stability of the deodorized oil by the active oxygen method (2) was 8 hours and was not increased by addition of citric acid prior to deodorization.

Hydrogenation

A batch (17.5 pounds) of the refined oil was selectively hydrogenated in a laboratory hydrogenator (4) at 15 p.s.i. pressure and 190°C., in the presence of 0.1% nickel catalyst prepared by dry reduction of electrolytically precipitated nickel hydroxide (3). Hydrogen absorption corresponded to a reduction of approximately one iodine unit per minute. Samples of approximately one pound each were withdrawn periodically at predetermined iodine values, filtered through precoated filters to remove suspended catalyst, and analyzed with the results shown in Table IV.

The composition based on ultraviolet absorption data (Table IV) was calculated with the equations adopted by the American Oil Chemists' Society (1) for this method. Since the hydrogenated linoleic glycerides probably contain both cis and trans isomers, this method of calculation is believed to be as satisfactory for hydrogenated oils as that using the revised constants (20a, 22) for natural linoleic acid.

The percentage of conjugated constituents was found to be less than 1% in all except the first hydrogenated sample which contained 6% conjugated diene. The composition of the hydrogenated samples calculated from iodine-thiocyanogen values and ultraviolet absorption data agree fairly well, but the calculated value for the saturated glycerides in neither case agrees very well with those obtained independently by the Bertram oxidation method.

The consistency of the hydrogenated samples was determined by the micropenetration technique described by Feuge and Bailey (10). The samples were maintained at 0°C. for *ca.* 16 hours and tempered for 30 minutes successively at each temperature at which the micropenetration was measured. From the micropenetration curves shown in Figure 1 it is apparent that samples 2 and 3, having iodine values of 87.9 and 82.8, are softer and samples 4, 5, and 6, having iodine values of 78.0, 73.3, and 68.7, are somewhat harder than shortening consistency (curve C. S.) which is taken as 100 micropenetration units at 25°C. Under the conditions of hydrogenation employed, which were highly selective, the plastic range of the hydrogenated safflowerseed oils is shorter than that of a typical commercial shortening.

The hydrogenated oils were deodorized as described for the unhydrogenated oils, stored in a refrigerator, and examined periodically with respect to flavor and odor. Immediately after deodorization all of the samples were free of any taste and odor. One week later sample No. 2 had a pronounced "oily" taste, and sample No. 3 a similar, but less intense taste. At the

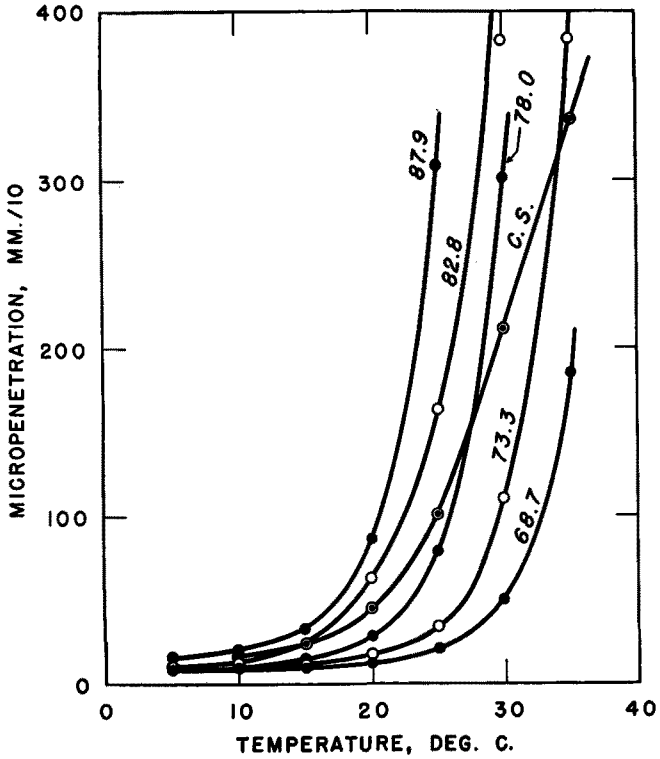

FIG. 1. Micropenetration curves of safflowerseed oil hydrogenated to various iodine values compared with the micropenetration curve of a commercial shortening (C. S.).

end of two weeks sample No. 2 had a "beany" or "grassy" flavor, and the "oily" taste of sample No. 3 was more pronounced. Samples 4-6 were still free of off-flavor and odor at the end of the two weeks' storage period. The results of the organoleptic examination parallels the stability values shown in Table IV. From the data shown in this table it is apparent that it is necessary to harden safflowerseed oil to an iodine value in the vicinity of 73 to obtain an accelerated stability value of 100 or above. However at this consistency safflowerseed oil is unsuitable for use in an "all-hydrogenated" shortening.

Salad Oil

The low stability of unhydrogenated safflowerseed oil can undoubtedly be attributed to the high content of glycerides of linoleic acid. These can be decreased by selective hydrogenation to convert them to oleic acid glycerides without any significant increase in the amount of saturated acids. However the formation of a certain amount of iso-oleic acid glycerides cannot be avoided, and the hydrogenated product would require winterization to produce an acceptable salad oil. A winterized oil of this type was prepared from sample No. 1 of Table IV.

About 600 grams of this oil was melted at 60°C., cooled to 26°C., followed by a gradual reduction of the temperature to 9°C. during a period of 6 hours, and holding the oil at this temperature for 18 hours. The chilled oil was filtered at the same temperature with a yield of 77.5% of winterized oil. The iodine value of the winterized oil was 117.0 and that of the stearine fraction 107.3. The winterized product passed a 7-hour cold test of the American Oil Chemists' Society.

The winterized hydrogenated oil and the corresponding unhydrogenated oil were deodorized at the same time. Both products were free of off-odor or flavor when removed from the deodorizer. After storage for several days both products had slight off-flavors. The winterized oil had an "oily" taste, and the unhydrogenated oil had a "beany" or "grassy" flavor. The stability by the active oxygen method was the same for both oils.

Summary

Four varieties of safflowerseed grown in Nebraska and Colorado were examined with respect to the composition of the seed, hulls, extracted meal, and oil.

The oil from one variety (Nebraska 852) was (A) refined, bleached, and deodorized; and (B) refined, bleached, hydrogenated, and deodorized. The liquid oil and the hardened fat were analyzed with respect to their fatty acid compositions and their stabilities to rancidity and flavor reversion were determined.

All of the results indicate that safflowerseed oil is not as suitable for edible purposes as other domestically produced edible oils.

Acknowledgment

The authors wish to express their appreciation to Carl E. Claassen for the safflowerseed used in this investigation; to L. J. Molaison for assistance in processing the seed; to J. R. Loeb, H. Damare, and R. T. O'Connor for the spectrophotometric analyses of the oils; and to J. F. Jurgens, A. F. Cucullu, and V. O. Cirino for the analyses of the seed, meal, and hulls reported here.

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[Received November 8, 1950]