[CONTRIBUTION FROM THE OIL, FAT AND WAX LABORATORY, BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

OKRA SEED OIL.[1]

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The object of this investigation was to prepare several samples of okra seed oil and determine the so-called constants for each sample, in addition to making a study of the chemical composition of the oil.

Several lots of the seed of okra (*Abelmoschus esculentus*, Malvaceae) were received at various times from E. A. McIlhenny, of Avery Island, La., through David Fairchild of the Department of Foreign Seed and Plant Introduction of the Bureau of Plant Industry. The first samples of okra seed were received about two years ago, and H. S. Bailey obtained the cold pressed oil from these seeds by means of an expeller. Shortly afterwards, H. S. Bailey and J. M. Johnson made a preliminary examination of these samples of oil, and their results, which have recently been confirmed by us, have been incorporated in this paper.

Last January another 150-pound sample of the Avery Island okra seed was sent to this laboratory. The seed were pressed in the expeller, with the result that 12 pounds of virgin okra seed oil were obtained. The oil had a pleasing, greenish yellow color, and a slight but distinct fragrant odor. This seed was found to contain 15.60% of oil and 5.63% of moisture. The press cake contained 5.88% of oil.

It is interesting to observe that okra seed oil, as well as oils obtained from other members of the Malvaceae, has been found to give the Halphen color test.[2]

TABLE I.

Sample number.	1.	2.	3.	4.
Iodine number (Hanus)	93.2	100.3	95.5	95.2
Saponification number	195.5	195.6	195.6	195.2
Polenske number	....	....	....	0.23
Reichert-Meissl number	....	....	....	0.26
Acetyl value	23.9	16.2	11.5	21.4
Acid value	....	0.66	0.34	1.42
d_{25}^{25}	0.9187	0.9182	0.9160	0.9172
Refractive index at 25°	1.4692	1.4693	1.4695	1.4702
Unsaponifiable matter, %	....	....	....	0.37
Soluble acids, %	....	0.12	0.09	0.14
Insoluble acids, %	....	95.90	96.27	96.20
Unsaturated acids, %	....	....	....	67.33
Saturated acids, %	....	....	....	29.22

Sample 4 was the oil obtained from the 150-pound sample of seed mentioned above.

[1] Published by permission of the Secretary of Agriculture.

[2] S. L. Ivanov and N. F. Kokotkena, *Soobsclich. Biuso Chastu Rast*, [2] 1915, No. 7, 3 to 24.

When the Renard test for arachidic acid was applied to a 20 g. portion of the oil, 2.2 g. of fatty acids were obtained, which were found to melt at about 58°. After two recrystallizations of these fatty acids from alcohol, the melting point was raised only a degree; this indicated, as in the case of tomato seed oil,[1] that the Renard test was not applicable to this oil. The preceding table contains the results of the analyses of 4 okra seed oils.

The table given below contains the analysis and other data obtained from the insoluble acids, saturated and unsaturated acids, of Sample 4.

	Insoluble acids.	Saturated acids.	Unsaturated acids.
Iodine number (Hanus)	97.3	5.7	137.9
Saponification number	210.6	215.6	199.2
Mol. wt	266.4	260.1	281.6
Acetyl value	..	...	25.4
M. p	40–41°	...	...
Titer	38.5°	...	...

The unsaturated acids had a beautiful green color.

In order to identify and to determine the approximate amount of unsaturated fatty acids, a portion of 30 g. of okra seed oil was taken and these acids were separated by the well-known lead-salt ether process. Precautions were taken to prevent the oxidation of the unsaturated liquid acids by evaporating the ether solutions of these acid in an atmosphere of dry carbon dioxide. For the identification and separation of the unsaturated acids the bromine method of Eibner and Muggenthaler[2] was employed. A 10 g. sample of the acids, dissolved in 90 cc. of absolute ether, was cooled to —10°, and 5 g. of bromine was slowly added, drop by drop. During the addition of the bromine, the temperature was not allowed to rise above —5°. After the addition of the bromine, the contents of the bromination flask were allowed to stand at —5° for 2 hours. The small amount of crystalline material which had separated was filtered in a weighed Gooch crucible, washed with cooled absolute ether, dried at 100°, and weighed. The crystals weighed 0.3727 g.; they melted at 114°, which showed that the compound was the tetrabromide of linolic acid, and that the unsaturated acids which form insoluble octa- and hexabromides were absent. The excess of bromine was removed from the filtrate in the usual manner with sodium thiosulfate, and after the ether was removed by distillation, the bromides were dissolved in hot petroleum ether. After cooling, the solution was allowed to stand for several hours, and then the separated tetrabromide was filtered and weighed. This crop of crystals weighed 4.9839 g. The filtrate was concentrated to less than half of its original volume, and again cooled and allowed to stand as before. After filtering this crop of tetrabromide crystals, which weighed 0.5296 g., the filtrate was evaporated to a volume of 25 cc., and allowed

[1] *J. Ind. Eng. Chem.*, 11, 850 (1919).

[2] Lewkowitsch, 5th Ed., 1, 573.

to stand in the ice box overnight, and no more crystals separated. Each crop of crystals was found to melt at 114°, the melting point for tetrabromo-linolic acid. As it was impossible to get any more of the tetrabromide to separate, all of the petroleum ether was removed, and the residue of tetrabromide linolic acid and dibromoleic acid was weighed. The bromine content was determined by boiling portions of the residue with conc. nitric acid and solid silver nitrate. Oleic dibromide contains 36.18% of bromine, and linolic tetrabromide contains 53.33%. Therefore, knowing the bromine content of the residue, it is possible to calculate the proportions of dibromide and tetrabromide present. The data obtained from the examination of the unsaturated acids are given in the following table:

	G.
Sample of unsaturated acids	10.0000
Linolic tetrabromide crystallized	5.8862
Residue (di- and tetrabromide)	12.4153
Dibromide in residue 80.47% or	9.9900
Tetrabromide in residue 19.53% or	2.4247
Total tetrabromide	8.3109
Linolic acid = tetrabromide	3.8784 or 38.78%
Oleic acid = dibromide	6.3741 or 63.74%

The percentages of linolic and oleic acids in the unsaturated acids were calculated into the percentages of the glycerides in the original oil as follows:

	Found. %.	Calc. to basis of 100%. %.	In original oil. %.	Glycerides in oil. %.
Oleic acid	63.74	62.17	41.86	43.74
Linolic acid	38.78	37.83	25.47	26.62
Total	102.52	100.00	67.33	70.36

Examination of Saturated Acids.

From 242 g. of okra seed oil, 70.7 g. of saturated acids were obtained. The methyl esters of these acids were prepared in the same manner as the esters of the saturated acids of tomato seed oil,[1] and distilled under diminished pressure. The following table contains the data of the distillation of 64.9 g. of the methyl esters:

Fractions.	Temperature. T °.	Pressure. Mm.	Wt. of fractions. G.
1	170–173.5	7.0	24.7
2	170–174	5.0	22.7
3	174–175	5.0	11.2
4	178–186	5.0	2.1
5	187–195	5.0	1.8
6	(195)200–222	5.0	0.8
7	222–227	5–7	0.5
Residue	....	...	0.9
Total	....	...	64.7

[1] *Loc. cit.*

The fractions in turn, as their boiling points were reached, were returned to the distilling bulb and refractionated in order to get a better separation of the esters. The following table contains the results of the analyses of 7 fractions, obtained by the redistillation of the fractions:

Fractions.	Wt. of fractions. G.	Iodine number.	Saponification value.	Mean mol. wt.	Palmitic acid. G.	Palmitic acid. %	Stearic acid. G.	Stearic acid. %	Arachidic acid. G.	Arachidic acid. %	Unsaturated acids. G.	Unsaturated acids. %
1.....	23.2	..	...	...	22.00	94.82	..	...	...	..	..	...
2.....	21.8	3.1	206.4	271.8	19.46	89.46	0.72	3.32	...	..	0.49	2.25
3.....	10.8	5.9	204.3	274.6	8.63	79.93	1.15	10.68	...	..	0.46	4.28
4.....	2.3	12.8	193.9	289.3	0.48	20.98	1.49	64.93	...	..	0.21	9.28
5.....	1.0	14.9	190.8	294.1	0.14	14.06	0.70	70.37	...	..	0.10	10.00
6.....	0.8	16.3	...	...	...	...	0.62	77.50	0.051	6.38	0.09	11.80
7.....	0.63	..	...	...	...	...	0.47	75.50	0.051	8.10	0.08	12.00

The distillation was continued until the small residue remaining in the distilling bulb charred. An attempt was made to isolate and identify the acids in this residue, but on account of the small amount of ester present it was found impossible.

It should be observed that the saponification values of Fractions 6 and 7 were not determined on account of the smallness of these fractions. The free fatty acids were obtained from these fractions by saponification, and precipitation with hydrochloric acid in the usual manner, and the arachidic acid was separated from the stearic acid by fractional crystallization from 95% alcohol. The arachidic acid obtained from Fractions 6 and 7, which was found to melt at 76°, was analyzed by Mr. Charles E. F. Gersdorff with the following results:

Subs. (Fraction 6), 0.0507: CO_2, 0.1428; H_2O, 0.0604.
Calc. for $C_{20}H_{40}O_2$: C, 76.85; H, 72.91. Found: C, 76.82; H, 13.24.
Subs. (Fraction 7), 0.0509: CO_2, 0.1435; H_2O, 0.0609. Found: C, 76.89; H, 13.29.

Both of the analyses as well as the melting points proved that it was arachidic acid.

The free acids recovered from Fraction 5 were repeatedly crystallized from 95% alcohol until the constant melting point of 67 to 68° was obtained, then this acid was analyzed by Mr. Gersdorff with the following results:

Subs., 0.1058: CO_2, 0.2946; H_2O, 0.1214.
Calc. for $C_{18}H_{36}O_2$: C, 75.98; H, 12.76. Found: C, 75.94; H, 12.75.

The analysis showed that the compound was stearic acid.

Acids.	Wt. G.	%	Acids in oil. %	Glycerides in oil. %
Palmitic.....	50.71	88.36	25.82	27.23
Stearic.....	5.15	8.98	2.62	2.75
Arachidic.....	0.10	0.17	0.05	0.05
Unsaturated.....	1.43	2.49	0.73	...
Total.....	57.39	100.00	29.22	30.03

The percentages of the saturated acids and their glycerides in the original oil have been calculated from the analytical data with the preceding results.

Summary.

The chemical characteristics of 4 samples of cold pressed okra seed oil have been determined. These oils vary slightly in composition. An exhaustive study has been made on the composition of the recently expressed oil No. 4, the results of which are given in the following table:

Glycerides of.	%.
Palmitic acid	27.23
Stearic acid	2.75
Arachidic acid	0.05
Oleic acid	43.74
Linolic acid	26.62
Unsaponifiable matter	0.37

WASHINGTON, D. C.

NEW BOOK.

The Condensed Chemical Dictionary. Compiled and edited by the Editorial Staff of the Chemical Engineering Catalog, of which F. M. TURNER, JR., is Technical Editor and D. D. BEROLZHEIMER, W. P. CUTTER and JOHN HELFRICH are Assistant Editors. The Chemical Catalog Company, Inc., 1 Madison Ave., New York, 1919. 525 pp. $5.00 net.

The title page of this useful book describes it as being "a reference volume for all requiring quick access to a large amount of essential data regarding chemicals and other substances used in manufacturing and laboratory work." While it is intended primarily to supply in readily available form the outstanding facts regarding such substances to people not chemically trained who are being brought into contact with the chemical industries in greater numbers with the growing importance of these industries, the book will serve as a great time-saver for the chemist who keeps it at hand.

Many substances of scientific interest but not important commercially have been omitted. Therein lies the chief significance of the word "condensed" in the title for, although it is stated that no attempt has been made to produce an exhaustive work, the field of commercially important chemical substances, excepting dyes, appears to have been pretty thoroughly covered.

Under the various headings information is given, with such variations as the nature of the substances may require, on derivation, habitat, color and other properties (restricted to those properties likely to be of commercial importance), constants, method of purification, grades, containers, uses, impurities, fire hazard, railroad shipping regulations and occasionally other information. Under "Derivation" a general idea of the method of