iodine value was then corrected for these components to give the amount of methyl oleate. The proportions of the saturated esters were computed from the saponification equivalents after correction for the known amounts of the unsaturated esters.

In Table IV are compared the original composition of the two mixtures and the composition calculated from a summary of the analyses of all the fractions.

Discussion

THE results of these analyses indicate that an accuracy of somewhat less than one unit per cent of the methyl ester in question can be obtained by using the above procedure. In fact, the calculated analyses and the actual analyses of the *individual components* differ on the average by less than 4% of the amount present.

It is recognized that hydrogenated fats may have complicating factors which might tend to decrease the accuracy somewhat but a series of duplicate analyses of hydrogenated shortenings in this laboratory have been found to agree well within the one unit per cent range. Natural fats such as milk fats (9, 11) and guinea pig body fats (12) have been analyzed using the above method and it has been our experience that the complex highly unsaturated C_{20} esters require further study by bromination or crystallization techniques to characterize completely their individual components. However, this is also true of the other procedures for fatty acid analyses. It should be remembered, as Hilditch has pointed out, that the methyl ester distillation technique of fat analysis is insufficient in itself for the identification with certainty of very small amounts of any specific component. The spectrophotometric analyses will indicate quantitatively diene, triene, and tetraene material and this information together with iodine values and saponification equivalents will afford a very close approximation to the composition of most fractions resulting from a methyl ester distillation.

Summary

Mixtures of known composition of purified methyl esters of lauric, myristic, palmitic, stearic, oleic, linoleic and linolenic acids have been prepared. Fractional distillation under reduced pressure followed by spectrophotometric determination of methyl linoleate and methyl linolenate in each of the *fractions* and determination of iodine values and saponification equivalents allowed calculation of the compositions which agreed well with the compositions of the original mixtures.

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Report of the Bleaching Methods Committee 1944-45

THIS Committee's work during the 1943-44 season was concentrated on studying the bleaching response of refined soybean oil against various natural and activated clays as the oils were aged under normal storage conditions. A rather marked change, a deterioration of the bleach in the case of the more active natural clays and activated clay led to a further study designed to establish whether the change was primarily in the oil, or in the bleaching material, or both. As reported in a supplement to last year's report, Oil & Soap, *22*, 22 (1945), this change was shown to reside in the refined oils, and early work during the current season furnished evidence that the deterioration was associated with the fairly rapid development of peroxide in stored refined oil samples.

Since the 4% activated clay bleach test was proposed specifically for the grading of the refined bleach color of crude soybean oils, the attention of the Committee was then directed to learning if the bleaching response on stored crude oil (freshly refined just before making each bleach test), also deteriorated and at what rate.

The results were brought together at a meeting of the Committee in Chicago in October, 1944. Bleach response-aging data were presented covering seven crude expeller oils studied in five different laboratories. The results are shown in Table I and indicate that the deterioration of refined bleach color in stored crude is negligible or non-existent. One member determined the change in bleaching response on storing a quantity of refined oil derived from the same crude, at the same time that the crude itself was being held and periodically tested. Peroxide values were run on both crude and refined. The results rather strikingly show the apparent connection between formation of peroxide in the refined oil and partial inactivation of the activated clay. (See Sander's data, Table I and Fig. 1; also see Table III). In this particular case the bleaching response of the refined oil against official A.O.C.S. Fuller's Earth also deteriorated whereas in most cases that remains substantially constant. Attention is called to the constancy of the 4% activated clay bleach in the case of the crude oil.

At the meeting in Chicago one member pointed out that all of these results were obtained on only one type of crude soy i.e., expeller. Arrangements were then made to get similar data on extracted oil, degummed and non-degummed, and on hydraulic oil. The results on these have just been completed and are given in Table II. Again the bleaching response remained uniform well within the normal irregularities of reading colors. At least one of the laboratories ran peroxide numbers on the oil and found them to remain very low as compared with the rapid increase noted in the case of holding refined oils. We con-

TABLE II.

Lab. and Member	Kind	Holding Time	2% Activated		4% Activated		6% A.O.C.S.	
			Y	R	Y	R	Y	R
P. & G. (Sanders)	Degummed Extracted	0	20	1.8	7	0.7	30	2.3
		14 days	17	1.2*	7	0.8	30	1.9
		28 days	16	1.6	8	0.8	30	1.9
		56 days	20	1.9	8	0.8	30	1.9
Staley (Marmor)	Degummed Extracted	0	9	1.1	4	0.8	15	1.4
		14 days	9	1.2	4.3	0.8	15	1.5
		60 days	9	1.2	4.2	0.8	15	1.5
Central Soya (Kruse)	Degummed Extracted	0	12	0.8	6	0.3	35	1.1
		14 days	12	0.8	6	0.3	35	1.1
		44 days	12	0.8	6	0.3	35	1.1
		75 days	12	0.8	6	0.3	35	1.1
Central Soya (Kruse)	Non-Degummed Extracted	0	12	0.8	6	0.3	35	1.1
		14 days	12	0.8	6	0.3	35	1.1
		44 days	12	0.8	6	0.3	35	1.1
		75 days	12	0.8	6	0.3	35	1.1
Spencer Kellogg (Freyer)	Hydraulic	0	35	3.5	20	2.1	35	4.5
		44 days	35	3.6	25	2.3	35	4.7
		120 days	....		25	2.3	35	4.7
Lever Bros. Co. (Houle)	Extracted						4%	
		0	18	1.5	8	0.8	35	2.4
		57 days	20	1.6	9	0.9	35	2.4
		103 days	20	1.5	10	0.9	30	2.2

* Report stated, "probably due to testing errors."

Filter Paper and Clarity for Color Reading. The Committee's attention was called to variable color readings obtained, according to which portion of the filtrate was taken, due to variable clarity. Investigation showed that this resulted from the use of large pore, fast filtering paper of the type designated for refined oil, and that when a paper of fine texture was used as specified in the bleaching method, the entire filtrate was of uniform clarity, except of course the first 10 to 20 ml. passing. There was no opportunity to act upon the suggestion of one member that specific brands and grades of paper be named in the method. That is a job for the new Committee to consider.

This Committee realizes that the methods have recently undergone extensive editorial revision and in recommending the following additions understands that considerable editing may be necessary to fit them into the new scheme. It is understood that much of the revision is concerned with reclassification and rearrangement, i.e., the revision of headings, etc. In this particular connection, since the new bleach test is designed specifically for grading crude oil, we believe that it should fall under the general heading "Crude Soybean Oil" and subheading, "Refined Bleached Color." Since the revised methods are not available to us now, however, we can but specify that the insertions be made at those points where they seem most logically to belong as the methods are now written and titled.

Recommendations

On p. 17a: Rewrite section under REFINED OILS—BLEACHING, as follows:

(a) *Apparatus:* Scales, weights, refining cups, and stirring machine are to be similar to those specified under REFINING, but with T-shaped paddles one-half inch wide instead of one inch wide.

Gas burners or electric heaters to heat the oil in the cups.

Official Fuller's Earth: This is obtained from the office of the American Oil Chemists' Society. A fresh supply must be used each year beginning August 1.

Official Activated Clay (for Refined Bleached Color on Crude Soybean Oil): This is obtained from the office of the American Oil Chemists' Society. Renewal of the supply is to be in accordance with information on the container label.

(b) *Determination:* Cottonseed and Other Oils, except Soybean—Weigh 300 grams of refined oil into a refining cup; heat to 120° C. and add 6 per cent of Official Fuller's Earth. Stir mechanically at 250 r.p.m. (plus or minus 10) for five minutes, not allowing temperature to fall below 105° C.

Filter through an unused filter paper of fine texture. After sufficient oil has passed the filter to insure clearness, collect a sample for color reading. Cool and read color immediately as prescribed under COLOR.

TABLE III.

Effect of Aging on Bleaching Response of Expeller and Extracted Soybean Oil Stored as Crude and as Refined Oil.

Earth Dosage Date	Age in Days	Stored as	N.S.P.A. Official Activated Clay		Commercial Earth A		Imported Activated		Commercial Clay C		A.O.C.S. Official		O.P.N.
			2%	4%	2%	4%	2%	4%	2%	4%	2%	4%	
Expeller Oil													
9- 9-44	0	Crude Oil	30/2.3	16/1.4	35/3.4	20/1.7	35/2.7	25/1.9	35/2.8	30/2.0	35/5.5	35/2.8	......
		Refined Oil	35/2.7	18/1.6	35/4.3	25/2.1	35/3.0	25/2.1	35/3.9	35/2.1	35/6.9	35/4.0	......
10- 9-44	30	Crude Oil	30/2.4	18/1.6	44/4.1	20/1.9	30/2.7	25/2.0	35/2.8	25/2.0	44/5.9	35/3.1	......
		Refined Oil	35/3.1	20/1.8	44/4.7	25/2.3	35/3.3	25/2.1	44/3.9	35/2.3	44/7.5	44/4.2	......
11-14-44	66	Crude Oil	30/2.4	16/1.6	44/3.8	25/2.2	30/2.7	20/1.9	44/3.3	30/2.0	44/5.9	35/3.3	......
		Refined Oil	35/3.6	20/1.8	44/5.4	30/2.9	35/3.6	30/2.4	44/4.1	35/2.6	44/7.5	44/5.0	......
1- 8-45	121	Crude Oil	30/2.2	16/1.4	35/3.6	20/1.7	30/2.4	25/1.8	35/2.9	30/1.9	44/6.0	35/3.1	2.2
		Refined Oil	44/3.7	25/1.9	44/5.4	35/3.0	35/3.5	35/2.3	44/4.3	35/2.4	44/7.7	44/5.4	10.4
3-14-45	186	Crude Oil	30/2.5	18/1.6	Insufficient Earth		35/3.0	25/2.1	35/3.0	30/2.0	35/5.7	35/3.0	2.7
		Refined Oil	35/3.9	35/2.5			35/3.7	35/3.1	35/4.3	35/2.7	35/7.7	35/4.8	15.8
Extracted Oil													
12-17-44	0	Crude Oil	18/1.5	8/ .8	20/1.9	10/1.0	18/1.7	12/1.1	35/2.0	18/1.4	35/5.4	35/2.4	......
		Refined Oil	20/1.8	9/ .9	35/2.4	12/1.1	25/1.9	16/1.3	35/2.9	20/1.6	35/6.8	35/3.2	......
2-12-45	57	Crude Oil	20/1.6	9/ .9	35/2.3	16/1.2	25/2.0	18/1.2	35/2.0	25/1.4	35/5.8	35/2.4	0.6
		Refined Oil	35/2.3	16/1.3	35/3.6	35/2.0	35/2.3	30/1.9	35/3.4	30/1.6	35/7.3	35/3.9	9.7
3-30-45	103	Crude Oil	20/1.5	10/ .9	25/2.3	16/1.1	25/1.9	16/1.3	30/1.9	20/1.4	35/5.5	30/2.2	2.2
		Refined Oil	30/2.4	20/1.6	35/3.4	35/2.7	25/2.4	30/2.2	35/3.0	20/1.7	35/7.3	35/3.5	15.6

All crude oil freshly lab. refined after aging and before bleaching.
Crude oil refined by A.O.C.S. methods using lowest amount of prescribed lye for the expeller and the highest amount for the extracted oil.
Official A.O.C.S. Bleaching Method used.
Refined oil from numerous cups composited before bleaching.

(Data submitted by R. J. Houle, Lever Brothers Company, Hammond, Indiana)

Soybean Oil: Method I. (Adopted as tentative, May, 1942.)

Weigh 300 grams of refined oil into a refining cup; add 6% of Official Fuller's Earth and, using mechanical agitation at approximately 250 r.p.m., heat immediately to 120° C., taking not more than five minutes. Then stir mechanically at 250 r.p.m. (plus or minus 10) for five minutes, not allowing temperature to fall below 105° C.

Filter through an unused filter paper of fine texture. After sufficient oil has passed the filter to insure clearness, collect a sample for color reading. Cool and read color immediately as prescribed under COLOR.

Method II (for Refined Bleached Color on Crude Soybean Oil): Use 4% of Official Activated Clay under the same test conditions given in Method I.

NOTE—Method I is inapplicable to certain oils having an unusually high chlorophyll content (green types). Method II is not recommended for use on commercial refined soybean oil unless it has been refined within a few days. Method II is designed especially for use on green type and damaged crudes, after test refining. Method I and II do not give the same bleaching results.

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Chairman.

Report of the Refining Committee
1944-45

TWO meetings of the Refining Committee were held during the year, one in New Orleans on May 9, 1944, and the other in Chicago on October 24, 1944. Sub-Committees have been active as follows:

Sub-Committee on Refining of Extracted Soybean Oil—S. O. Sorensen, chairman.
Sub-Committee on Centrifugal Refining of Extracted Soybean Oil—E. M. James, chairman.
Sub-Committee on Modified Cup Refining of Extracted Soybean Oil—J. H. Sanders, chairman.
Sub-Committee on Expeller vs. Hydraulic Method for Hydraulic Oil—E. B. Freyer, chairman.

Copies of the minutes of the two meetings held and of the reports of the Sub-Committees for the year have been furnished the members of the main Committee. This report will constitute a brief review of the most important points covered at the meetings and in the Sub-Committee reports.

REFINING COMMITTEE MEETING, MAY 9, 1944, NEW ORLEANS:

Present: 13 members, 4 alternates, and 4 visitors.

Sub-Committee on Centrifugal Refining (R. R. King, chairman) recommended that work on this method be continued. Recommendation was adopted.

Sub-Committee on Expeller vs. Hydraulic Method for Hydraulic Oil (E. B. Freyer, chairman) reported as follows:

> The substance of my report covers the refining of 32 samples of hydraulic oil by the laboratories of Swift and Company at Chicago and 11 samples of hydraulic oil by the laboratory of the South Texas Cotton Oil Co. at Houston, by both the method for expeller soybean oil and the former official method for hydraulic soybean oil, which it was voted by the Committee last October in Chicago should be abandoned. While the results of the tests show that 44% of the samples gave a lower loss by the expeller method, 49% by the hydraulic method and the balance showed no difference, the net average difference in losses shows the expeller method to be lower by 0.024%. Further, in the case of the widest loss differences between the two methods, the hydraulic results were the higher ones. The color of the refined oil is somewhat lighter by the expeller method although this point is possibly of small significance. On the whole, I consider the expeller method the better of the two, and its adoption has the advantage of making it possible to have only two standard procedures, one for hydraulic and expeller, and one for solvent extracted oil.

This report was unanimously accepted as confirmation of the previous year's recommendation of the Committee that the expeller method be made the official method for hydraulic oil.

Sub-Committee on Extracted Oil Refining (S. O. Sorensen, chairman) reported on new proposed kettle refining method for extracted oil. Messrs. Sorensen and James described their results which appeared to be favorable. It was decided to follow actively the investigation of the kettle method.

The 1943-44 report of the chairman covering the Refining Committee meetings at Peoria on July 7-8, 1943, and at Chicago on October 5, 1943, (Oil & Soap, February, 1945) was approved.

The following action was then taken with respect to a portion of the 1943-44 report:

> It was moved and seconded that the paragraph in the 1943-44 report reading as follows: "Be it resolved that since the present tentative A.O.C.S. method has proved satisfactory on several additional extracted oils from this year's crop and seems satisfactory for general use it should be recommended as an official method" be rescinded. This motion was passed unanimously.
>
> Another motion was then made, seconded, and passed unanimously as follows: "It is moved that since discrepancies in the analytical findings continue in the present method for the refining of various types of soybean oils that the present method for extracted oil be continued as a tentative method."

REFINING COMMITTEE MEETING, OCTOBER 24, 1944, CHICAGO:

Present: 11 members, 2 alternates, and 7 visitors.

Dr. Freyer's Sub-Committee to define the term "concordant results" as used in the soybean oil refining methods presented a report which suggested a definition. This report was accepted, and the chairman was instructed to poll the committee as to whether the proposed definition should be presented to the Society. A definition revised by Dr. Freyer for clarification was later submitted. The two proposed definitions for concordancy as follows were submitted to the Refining Committee. The preferred definition is to be suggested for incorporation in the Methods of Analysis of the A.O.C.S. immediately after the two lines of the last paragraph of page 16D:

I. Original Proposed Definition

On the test to be reported the chemists' report must be based upon the average of duplicate tests with the same lye agreeing within a tolerance of 0.3% on losses up to 6.0%, or within a tolerance of 5.0% of the lower loss when losses are above 6.0%.

II. Modification by Dr. Freyer

The result to be reported shall be the lower loss of the two obtained using different lyes, and it shall be based upon the