TABLE II

Cyclohexene Addition Products

Reactant	Yield of Addition Product	Carbon %		Hydrogen %		Hydroxyl %	
		Calc.	Found	Calc.	Found	Calc.	Found
Phenol	44	81.77	81.38	9.15	9.25	9.65	9.45
m-Cresol	62	82.06	82.03	9.54	9.75	8.94	8.70
2-Naphthol	25	84.91	83.90	8.02	7.99	7.52	7.42
o-Chlorophenol	48[a]	68.40	68.81	7.18	7.24	8.07	7.70
Methyl Salicylate	45[b]	71.77	71.86	7.74	7.80	7.26	6.60
Resorcinol	66	74.97	74.83	8.39	8.67	Not determined	
2,6-ditert-Butylphenol	50	83.27	83.17	11.18	10.99	Not determined	
p-tert-Butylbenzenethiol	65[c]	77.35	77.80	9.74	9.97	....	
2-Naphthalenethiol	21[d]	79.28	79.21	7.49	7.52	....	
Anisole	66	82.06	81.86	9.54	9.56	....	
Phenetole	62	82.30	82.17	9.87	9.90	....	
Thioanisole	No addition	....	....	....	....	....	
Benzoic acid	47[e]	71.44	72.94	7.90	7.85	....	

[a] Chlorine %: Calc. 16.83, Found 17.09.
[b] S.E.: Calc. 234, Found 233.
[c] Sulfur %: Calc. 12.91, Found 13.00.
[d] Sulfur %: Calc. 13.23, Found 12.90.
[e] S.E.: Calc. 204, Found 206.

nucleophiles were cyclohexyl substituted phenols; the products obtained from the addition of arylthiols were thioethers. In the preparation of the 2-naphthalenethiol addition product of cyclohexene, the excess of thiol was removed by dissolving the crude mixture in a small amount of methylene chloride and precipitating the unchanged 2-naphthalenethiol by the addition of hexane. Phenyl ethers were also capable of being added (See Table II).

Table II shows the yield data for the addition of phenols, phenyl ethers and arylthiols to cyclohexene. The products obtained from these additions are similar to the addition products from oleic acid. Cyclohexyl benzoate was formed by the addition of benzoic acid to cyclohexene. Methyl salicylate added to cyclohexene via the phenolic hydroxyl group. The 66% yield of cyclohexyl resorcinol is another example of the advantages of the methanesulfonic acid catalyst-solvent addition procedure. Bartlett and Garland (9) obtained only a 5% yield of product in condensing cyclohexyl chloride with resorcinol in the presence of aluminum chloride.

Thioanisole is included in Table II to show that thioethers do not add to cyclohexene. This experience is similar to that obtained with oleic acid. It was not unexpected since the end product of thiol additions was thioethers. Anisole and phenetole were successfully added under similar conditions.

The non-oxidizing behavior, and lower tendency to form dark colored side reaction products, was again observed in the above addition reactions using methanesulfonic acid catalyst.

ACKNOWLEDGMENT

Analytical results by E. T. Donahue and members of the analytical section of the Eastern Regional Research Laboratory.

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[Received February 5, 1963—Accepted May 8, 1963]

Report of the Commercial Fats and Oils Analysis Committee, 1963

THE MAY 1962 report was the last report of the long-standing Fat Analysis Committee. The following month, the Commercial Fats and Oils Analysis Committee was organized with nine subcommittees, most of which were carried over, with the exception of the Refining Subcommittee and the Bleaching Subcommittee. Both of these had separate identities of their own. Following are reports of activities of several individual subcommittees, submitted by R. C. Stillman, Chairman.

Bleaching Methods Subcommittee, B. N. Stuckey, Chairman

This subcommittee started the year under the chairmanship of T. C. Smith, who was replaced in November, 1962, upon his retirement, by E. R. Hahn. An interim report (dated 1962) prepared by Mr. Smith, and revised in some detail, contains essential information for future work on the selection of bleaching earths for distribution by AOCS.

"Negotiations were started in the fall of 1959 for a new supply of natural bleaching earth to replenish the supply of AOCS official natural earth, estimated at the time to be close to twelve hundred cans. As of July 15, 1960, there were 831 cans of the original 8,100 cans approved for distribution August 1, 1954, on hand. This indicates that the annual sales had averaged slightly over 1,200 cans or about 100/month; therefore, it was fair to say that the Society had sufficient earth on hand to meet normal sales for one year.

"It was our good fortune to have been favored with the foresight of the Bennett-Clark Co., supplier of the current official earth. The original lot of domestic natural earth was purposely produced in excess of the immediate requirements. The excess, amounting to approximately 12,000 lb in 80-lb multi-ply paper bags, was held in Bennett-Clark storage for future requirements without compensation by the Society. This earth had been evaluated for uniformity as a 'grand lot' of 450 eighty-pound bags. The sampling and testing of the 'grand lot' of earth, for the sake of completeness, will be discussed more fully elsewhere in this report.

"The foresight mentioned relieved the committee of a considerable amount of essential work involving the reestablishment of the uniformity of a new lot of earth, because this phase of the work had already been accomplished during September, 1951.

Bleach Tests on New Earth—1959–1961. "It is required that the earth be approved by the Chemists' Committee of the NCPA and the Technical Committee of the NSPA. The Chairmen were asked if they would accept the findings of the Bleaching Methods Subcommittee. Both Chairmen replied in the affirmative. Samples of proposed and official earth were sent out to each member of the subcommittee accompanied by

TABLE I

Collaborative Bleach Test Results on Refined Soybean Oil Comparing New Lot of Proposed Official Natural Bleaching Earth with Present Official Natural Bleaching Earth Approved August 1, 1954

3% Earth used in each test

Collaborator	Sample	Wesson (Lovibond) color			AOCS (Photometric) color		
		with proposed official earth	with present official earth	difference	with proposed official earth	with present official earth	difference
1	1 orig.	1.3	1.3	0	1.3	1.3	0
	1 dup.	1.3	1.3	0	1.3	1.3	0
	2 orig.	1.8	1.8	0	1.8	1.8	0
	2 dup.	1.8	1.8	0	1.7	1.8	—0.1
2	1 orig.	2.4	2.5	—0.1			
	1 dup.	2.3	2.5	—0.2			
	2 orig.	2.6	2.9	—0.3			
	2 dup.	2.7	2.8	—0.1			
6	1 orig.				1.77	1.71	0.06
	1 dup.				1.87	1.67	0.20
	2 orig.				1.90	1.91	—0.01
	2 dup.				2.02	1.86	—0.16
7	1 orig.	3.5	3.9	—0.4			
	1 dup.	3.0	4.0	—1.0			
	2 orig.	3.5	3.5	0			
	2 dup.	3.5	3.5	0			
9	1 orig.	2.65	2.55	0.05	2.21	1.97	0.24
	1 dup.	2.60	2.60	0	1.91	2.20	—0.29
	2 orig.	2.70	2.60	0.10	2.39	2.37	0.02
	2 dup.	2.65	2.60	0.05	2.34	2.40	—0.06
10	1 orig.	1.2	1.2	0	1.56	1.67	—0.11
	1 dup.	1.2	1.2	0	1.67	1.56	0.11
13	1 orig.	1.83	1.87	—0.04			
	1 dup.	1.85	1.83	0.02			
	2 orig.	1.63	1.70	—0.07			
	2 dup.	1.63	1.60	0.03			
14	1 orig.	2.0	1.9	0.1	2.35	1.94	0.41
	1 dup.	2.0	1.9	0.1	2.25	1.95	0.30
	2 orig.	2.5	2.5	0	2.83	2.63	0.20
	2 dup.	2.5	2.5	0	2.84	2.78	0.06
Average difference in bleach color with proposed official earth vs. present official earth				—0.07			+0.05

TABLE II

Collaborative Bleach Test Results on Refined Cottonseed Oil Comparing New Lot of Proposed Official Natural Bleaching Earth with Present Official Natural Bleaching Earth Approved August 1, 1954

4.67% Earth used in each test

Collaborator	Sample	Wesson (Lovibond) color			AOCS (Photometric) color		
		with proposed official earth	with present official earth	difference	with proposed official earth	with present official earth	difference
3	1 orig.	1.9	1.8	0.1			
	1 dup.	2.1	2.0	0.1			
	2 orig.	5.0	4.9	0.1			
	2 dup.	5.7	5.7	0			
4	1 orig.	2.40	2.40	0	2.39	2.35	0.04
	1 dup.	2.40	2.40	0	2.27	2.37	—0.10
	2 orig.	4.45	4.45	0	4.27	4.27	0
	2 dup.	4.45	4.45	0	4.27	4.27	0
5	1 orig.	2.0	2.0	0			
	1 dup.	2.0	2.0	0			
	2 orig.	5.1	5.0	0.1			
	2 dup.	5.0	5.0	0			
8	1 orig.	2.0	1.8	0.2	2.0	1.8	0.2
	1 dup.	2.0	1.9	0.1	2.0	1.9	0.1
	2 orig.	3.4	3.4	0	3.8	3.8	0
	2 dup.	3.4	3.4	0	3.9	3.9	0
10	2 orig.	2.4	2.5	—0.1	2.87	2.88	—0.01
	2 dup.	2.4	2.5	—0.1	2.70	2.88	—0.18
11	1 orig.	6.9	6.9	0	7.30	7.10	0.20
	1 dup.	6.9	6.9	0	7.20	7.10	0.10
	2 orig.	2.3	2.3	0	2.50	2.60	—0.10
	2 dup.	2.3	2.3	0	2.50	2.50	0
12	1 orig.	3.1	3.1	0			
	1 dup.	3.2	3.1	0.1			
	2 orig.	3.1	3.0	0.1			
	2 dup.	3.0	3.1	—0.1			
Average difference in bleach color with proposed official earth vs. present official earth				+0.02			+0.02

instructions as to how to proceed with the testing for bleaching equivalence.

"Instructions agreed to in a subcommittee meeting in Dallas, Texas, April 6, 1960, were as follows:

1. 'Duplicate bleaches are to be made on two oils using both earths. This will make a total of eight (8) bleaches to be run by each member.'
2. In order to get an equal distribution of oil types vs. earths, a breakdown of the committee was made, assigning soya and cottonseed oils on the basis of the most likely availability of types for testing.
3. The two oils selected, soya or cottonseed, should represent extremes of normal oils from the standpoint of bleachability.
4. The bleach tests should be made following strict adherence to the AOCS Official Method applicable to the oil being bleached.
5. The refined oil should be filtered to absolute clarity before bleaching.
6. Colors should be read according to the applicable method, with the oil to be read filtered completely free of the earth in order to prevent light scattering which could cause apparent discrepancies between the two earths.

The bleaching tests conducted on the new lot of proposed official natural bleaching earth in comparison with the present of final natural bleaching earth approved for use after August 1, 1954, are summarized in Tables I and II.

"Table III is a complete summary of the tests conducted by collaborator 2.

"The data of all the bleach tests were statistically analyzed and were submitted to the appropriate committees of the NCPA and the NSPA. With the approval of these two trade groups that the new earth is satisfactory for use, instructions were issued to the Bennett-Clark Co. on August 29, 1960, to proceed with the packaging of 3,000 one-half gallon cans.

"As of July 1, 1962, LaPine Scientific, distributor of AOCS supplies, reported an inventory of 24 cans of natural bleaching earth. On July 5, 1962, 600 cans were added to the inventory. This leaves 1224 of the 3000 cans of the last lot of earth still on hand in the Bennett-Clark warehouse in Nacogdoches, Texas, or a total visible stock of earth, in cans, equal to 1848 units, as of July 1, 1962. Assuming the average sales to be close to 100 three-pound cans/month, there is sufficient natural earth on hand for 18 months. There are still 34 eighty-pound, multi-ply paper bags or 2720 pounds of the original 'grand lot' of the earth on hand in Nacogdoches. There was an error made in the assumption that the natural earth packed four pounds/can instead of three, which is the sample representing the remainder of the original natural earth amounting to 12,000 pounds, evaluated for bleaching equivalence and authorized to be packaged on August 29, 1960.

"Based on subcommittee experience, at least one year or longer should be allowed for the preparation, evaluation and approval of a new lot of earth. By

TABLE III

Bleach Test Results on Refined Soybean Oil Comparing New Lot of Proposed Official Natural Bleaching Earth with Present Official Natural Bleaching Earth Approved August 1, 1954

3.0% Earth used in each test

	Refined oil used in tests		Lovibond color of bleached oil		ppm Chlorophyll of bleached oil	
	Lovibond color	ppm Chlorophyll	with proposed official earth	with present official earth	with proposed official earth	with present official earth
Oil A	70/7.5	1.24				
Test 1			25/2.4	25/2.5	0.02	0.02
Test 2			23/2.3	25/2.5	0.02	0.02
Average			2.35	2.50	0.02	0.02
Oil B	70/8.6	1.93				
Test 1			26/2.6	29/2.9	0.03	0.03
Test 2			27/2.7	28/2.8	0.03	0.03
Average			2.65	2.85	0.03	0.03

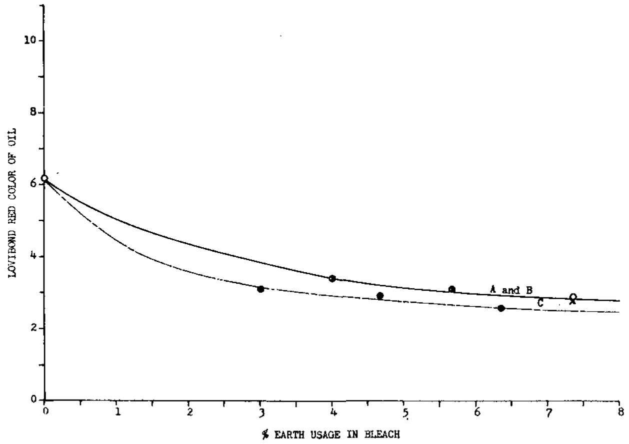

FIG. 1. Bleaching Refined and Filtered Cottonseed Oil—Red Color Removal.
A. Natural Bleaching Earth—Official until July 31, 1951
B. Natural Bleaching Earth—Official until July 31, 1954
C. Natural Bleaching Earth—Official since October, 1960

having the advantage of experience and the aid of more scientific techniques, the job of matching earths for equivalence has been made much easier and more reliable.

Background Information. "This report would not be complete without the inclusion of some background information. Rather than to be repetitious and include historical information already published (JAOCS, *31,* 127) this interim report has and will, for the most part, cover the activities of the Bleaching Methods Subcommittee during the period since the changeover from English natural bleaching earth to one of domestic origin. The changeover was motivated by a series of very unpleasant incidents which occurred during the use of the last lot of official English natural earth which expired on July 31, 1954, and was replaced by the earth of domestic origin. It was anticipated that some difficulty might arise in connection with Commodity Credit Corporation cottonseed oil graded into storage with the old English earth and out of storage with the new earth. For this reason the AOCS surplus stock of the former official English earth, consisting of 155 cans, was transferred to S. M. Harmon, Secretary-Treasurer of NCPA, Memphis, Tenn. This earth was to be used under the direction of the Chemists' Committee, in adjudication of hardship cases resulting from the change in the origin of the official natural earth.

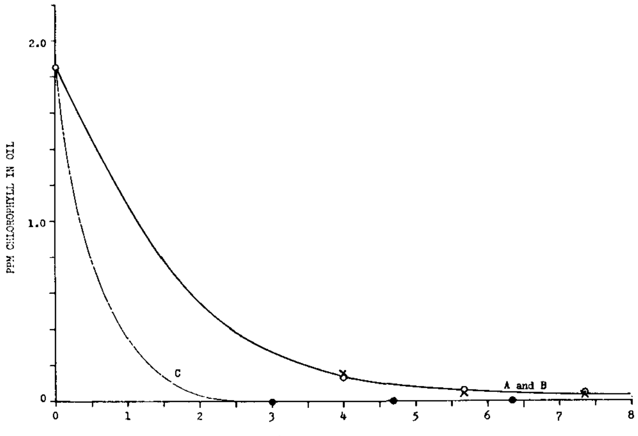

FIG. 2. Bleaching Refined and Filtered Cottonseed Oil—Green Color Removal.
A. Natural Bleaching Earth—Official until July 31, 1951
B. Natural Bleaching Earth—Official until July 31, 1954
C. Natural Bleaching Earth—Official since October, 1960

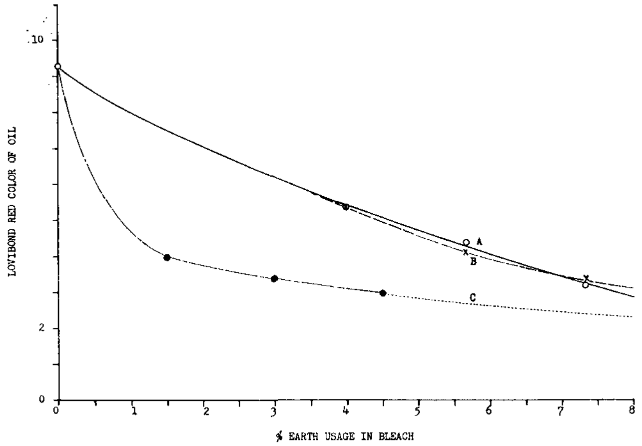

FIG. 3. Bleaching Refined and Filtered Soybean Oil—Red Color Removal.
A. Natural Bleaching Earth—Official until July 31, 1951
B. Natural Bleaching Earth—Official until July 31, 1954
C. Natural Bleaching Earth—Official since October, 1960

"Early in September, 1954, after use of the new earth was started at the dosages recommended by the Bleaching Methods Subcommittee, a number of complaints from the cottonseed oil industry, originating in isolated geographical locations, were received. These were claims that the new earth, at the recommended dosage of 3.67% adjusted by the Chemists' Committee of the NCPA from the 3.5% dosage recommended by the Bleaching Methods Committee, gave red colors higher than did 5.67% of the old English earth. It has been authentically stated that increases in the chlorophyll content of an oil will result in the masking of the red pigment, thus giving lower red readings by the Lovibond color standards (JAOCS, *31,* 311). The bleaching test has indicated that the new domestic earth has a greater affinity for the removal of chlorophyll. The Technical Committee of the NSPA was confronted with similar complaints. As a result, a meeting of this group was called during the fall meeting of the AOCS in Minneapolis, October 11, 1954, to discuss what action the Committee should take to alleviate the pressure from a very small minority of the soybean processors located in areas where green oil was giving color trouble. After considerable discussion there was a motion to follow the action of the Chemists' Committee of the NCPA and increase the dosage from 2.0 to 3.00%, effective immediately. The motion was unanimously approved. The Chemists' Committee had previously increased the dosage from 3.67 to 4.67% for bleaching cottonseed oil.

"To digress, the original 'grand lot' of earth had been tested for equivalence and uniformity by Law and Co. under the direction of D. L. Henry. The sampling scheme called for dividing the 'grand lot' of 450 eighty-pound bags numbered in the packing sequence, into three sublots of 150 bags each. The samples were taken from a randomized selection—one sample per bag and twenty-four per sublot, for a total of seventy-two samples across the 'grand lot.' Four refined cottonseed and four normal colored soybean oils, furnished by The Procter & Gamble Co., were used for the bleaching tests. The bleach tests were conducted at three earth concentrations, viz., 3.00, 4.12 and 5.67%, making up a set of twenty-four test conditions. By matching test conditions and sublot sam-

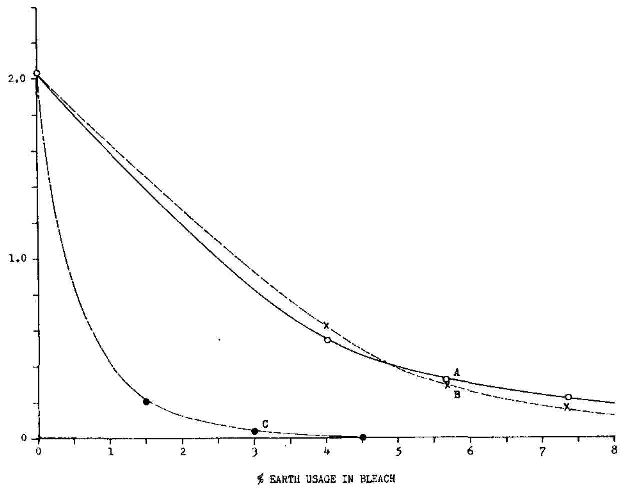

Fig. 4. Bleaching Refined and Filtered Soybean Oil—Green Color Removal.

A. Natural Bleaching Earth—Official until July 31, 1951
B. Natural Bleaching Earth—Official until July 31, 1954
C. Natural Bleaching Earth—Official since October, 1960

ples, it was possible to obtain one complete set of bleaches with each sublot and thus provide a basis for comparison of the three sublots. For controls, forty-eight similar bleaching tests (two replicate sets of twenty-four) were made at the same concentrations (3.00, 4.12 and 5.67%) with the same oils, using the then current official (English) earth. Bleached oil colors were read and recorded both by Lovibond and AOCS spectrophotometric systems. It may be observed from the foregoing that a very carefully planned program was followed for the establishment of the uniformity of the 'gross lot' of earth.

"On the basis of the results obtained from the testing of the earth for uniformity and the control bleaches for equivalence, these data indicated the following bleaching equivalents: 1) cottonseed oil required a concentration of 4.2 ± 0.4% of the proposed earth to give the bleaching equivalence of 5.67% of the official (English) earth; 2) soybean oil required a concentration of 2.4 ± 0.4% of the proposed earth to give the bleaching equivalence of 5.67% of the official (English) earth.

"It will be observed that the equivalent values differ from those derived from the average bleach equivalent data of the Bleaching Methods Subcommittee, viz., 4.2 ± 0.4% vs. 3.5% for cottonseed oil and 2.4 ± 0.4% vs. 2.0% for soybean oil. It is reasonable to assume that part of the difference could be attributed to the oils produced from seed grown in different years, 1951 vs. 1952 and 1953 vs. 1954.

Recent Comparisons of Natural Earths. "In order to have a permanent record of the comparative effectiveness of previous official natural bleaching (English) earth vs. present official natural bleaching (domestic) earth, three previously unopened cans of earth, having expiration dates of July 31, 1951 and 1954 (English) and domestic earth which had been approved as official since October, 1960, were employed for this purpose.

"This work is presented in the form of curves (Figs. 1–10); definition table and a tabulation of Lovibond color readings representing red and green color removal by the foregoing mentioned earths in refined cottonseed and soybean oils.

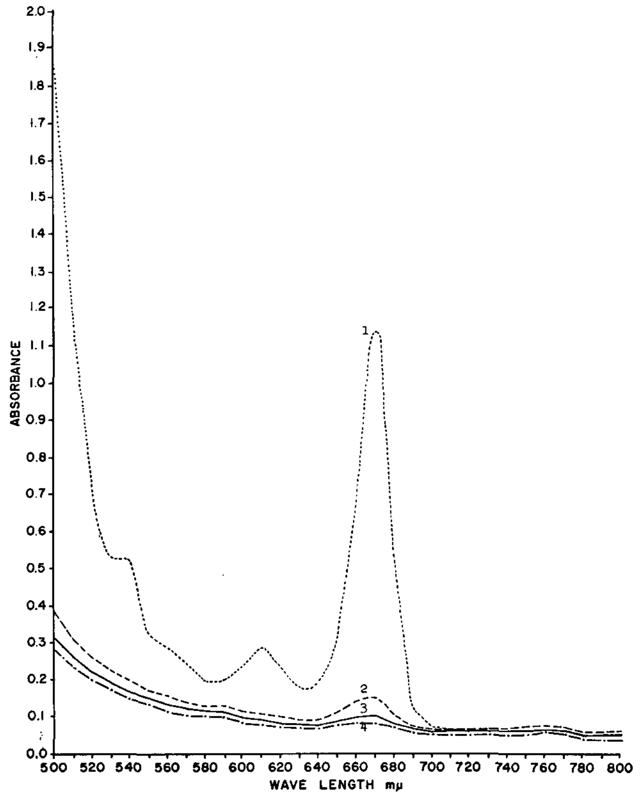

Fig. 5. Refined and Filtered Cottonseed Oil Bleached with Natural Bleaching Earth—Official until July 31, 1951.

Absorbance Curves 5 cm cell

1. Unbleached Oil	3. Bleached with 5.67% Earth
2. Bleached with 4.00% Earth	4. Bleached with 7.33% Earth

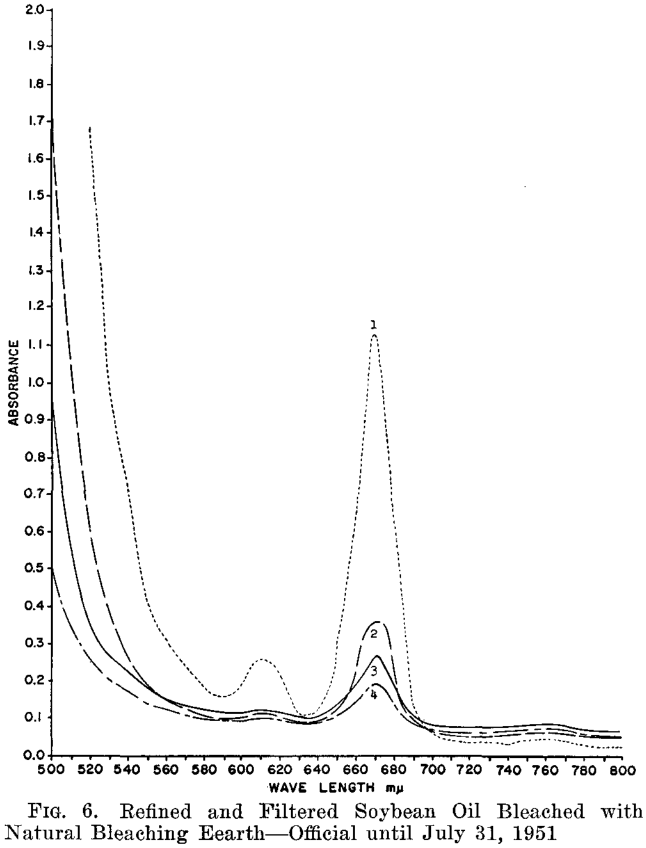

Fig. 6. Refined and Filtered Soybean Oil Bleached with Natural Bleaching Eearth—Official until July 31, 1951

Absorbance Curves 5 cm cell

1. Unbleached Oil	3. Bleached with 5.67% Earth
2. Bleached with 4.00% Earth	4. Bleached with 7.33% Earth

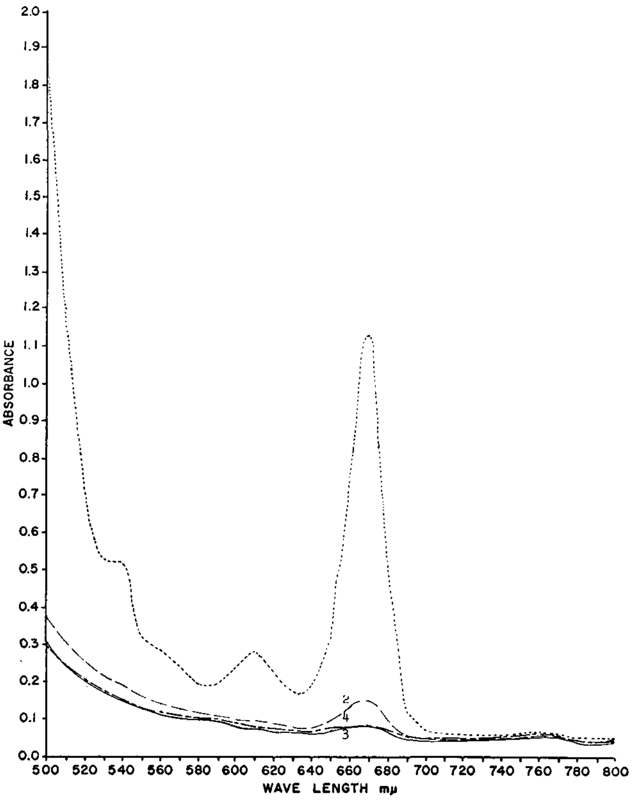

Fig. 7. Refined and Filtered Cottonseed Oil Bleached with Natural Bleaching Earth— Official until July 31, 1954.

Absorbance Curves 5 cm cell

1. Unbleached Oil
2. Bleached with 4.00% Earth
3. Bleached with 5.67% Earth
4. Bleached with 7.33% Earth

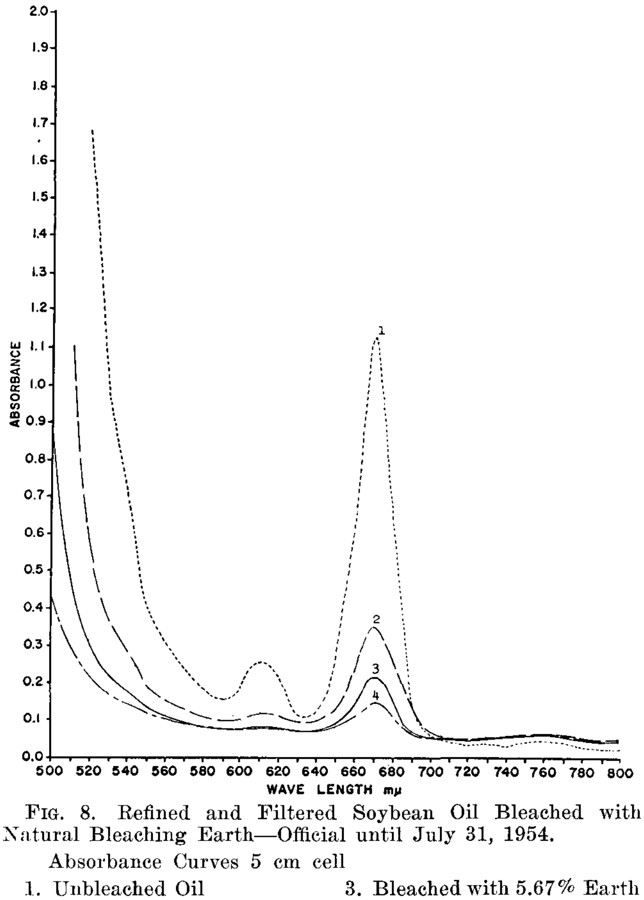

Fig. 8. Refined and Filtered Soybean Oil Bleached with Natural Bleaching Earth—Official until July 31, 1954.

Absorbance Curves 5 cm cell

1. Unbleached Oil
2. Bleached with 4.00% Earth
3. Bleached with 5.67% Earth
4. Bleached with 7.33% Earth

Other Activities. “Three lots, 2000 three-pound units each of activated bleaching earth were tested for bleaching equivalence in 1952, 1956 and 1962. The activated earth is used for bleaching green soybean oil and is tested for bleaching equivalence and approved for use by the NSPA Technical Committee. In 1956, a subcommittee of the Fat Analysis Committee on tallow and grease participated in the testing of bleaching equivalence against the previous lot of earth. In 1962, the Smalley group on tallow and greases participated in the testing.

“Two lots, 2000 units each, of Diatomaceous earth were tested by the members of the NSPA Technical Committee and the AOCS Bleaching Methods Subcommittee in 1953 and 1959.

Acknowledgments. “We wish to acknowledge the efforts of R. T. Clause of The Procter & Gamble Co. for the preparation of absorbance curves produced with a Carey Recording Spectrophotometer; K. E. Holt, W. E. Link, and L. D. McLean, Archer-Daniels-Midland Co., for the statistical analyses of bleaching data; Al Rich, of Bennett-Clark, for his assistance in having a new lot of official natural bleaching earth made ready for distribution and C. T. Millice, Plant Manager, Filtrol Corp., for his help in the preparation of a new lot of official activated bleaching earth, approved for use during 1962.”

COMPARISON OF THREE DIFFERENT AOCS OFFICIAL NATURAL BLEACHING EARTHS FOR THEIR EFFECT IN BLEACHING A REFINED COTTONSEED OIL AND A REFINED SOYBEAN OIL

AOCS natural bleaching earth used		Bleaching refined and filtered cottonseed oil		Bleaching refined and filtered soybean oil	
Kind	Amount %	Lovibond color	ppm Chlorophyll	Lovibond color	ppm Chlorophyll
None (refined & filtered oil)		70/6.2	1.87	70/9.3	2.03
English earth— Official until July 31, 1951	4.00	70/3.4	0.13	50/5.4	0.54
	5.67	30/3.1	0.06	35/4.4	0.32
	7.33	30/2.9	0.05	35/3.2	0.22
English earth— Official until July 31, 1954	4.00	30/3.4	0.15	50/5.4	0.62
	5.67	30/3.1	0.06	35/4.1	0.29
	7.33	30/2.9	0.04	30/3.3	0.16
Current official earth Approved October, 1960	3.00	30/3.1	0		
	4.67	25/2.9	0		
	6.33	20/2.6	0		
Current official earth Approved October, 1960	1.5			35/4.0	0.21
	3.0			30/3.4	0.05
	4.5			30/3.0	0.01

Color Standards Subcommittee, E. W. Blank, Chairman

This subcommittee should not be confused with the Color Committee of the Society which now reports to the Instrumental Techniques Committee in a subcapacity. The Color Standards Committee has reported briefly as follows:

“A prototype set of permanent F.A.C. glass color standards is being circulated to members of the Subcommittee on F.A.C. Colors for their approval. If the color standards prove acceptable to the Subcommittee, formal approval by AOCS of the standards will be sought. It is necessary to obtain assurance from AOCS that the glasses are officially acceptable before Lovibond (Tintometer Ltd., England) will initiate the expensive task of accumulating a glass bank preliminary to production of the standards.

“The design and construction of a viewer for the standards is being actively followed. Consideration is being given to both an inexpensive viewer for routine work and a more highly developed instrument for reference and research purposes.”

Determination of Free Fatty Acids Subcommittee, W. O. Lundberg, Chairman

This committee has reported in detail concerning acid value by both colorimetric titration and potentiometric titration, and have submitted recommended procedures to the Uniform Methods Committee for their approval. The report is as follows:

"Almost ten years ago, following a considerable amount of collaborative laboratory work by members of the Subcommittee, a revision of Method Ca 5a-40 (free fatty acids) was proposed. The proposal involved some rather marked changes in the present method, including, principally, a change in the solvent system, for the purpose of effecting complete solution of samples, and the introduction of an alternative electrometric method for dark-colored oils.

"The proposed revision was submitted to the Fat Analysis Committee in 1954 by the chairman of that Committee, under the title, 'Second Tentative Draft of Revised Procedure for Determination of Acid Value or Free Fatty Acids,' with the request that both the colorimetric and potentiometric procedures be tried out, and that members of the Committee then send comments to him, indicating whether the method should be submitted to the Uniform Methods Committee. Following is a quotation from a letter received by the chairman of the Subcommittee on the Determination of Free Fatty Acids from V. C. Mehlenbacher, who was then chairman of the Fat Analysis Committee, dated February 7, 1957, which summarizes the reactions that were obtained:

'Of replies from 19 people who have actually tried out the procedure, the votes were as follows:

6 approved the method
5 voted against the method
7 noncommital'

"Believe that you will agree that this leaves the situation somewhat up in the air. The comments returned were about as follows:

1. Retain the original sample weights.
2. Reduce the solvent to 75 ml.
3. Use pH as end point but the suggestions for the pH level varied from 5–11.5.
4. Safety, or lack of it, involved in the use of benzene was questioned.
5. Two phases sometimes resulted, apparently depending upon the free fatty acid content.
6. Several questioned the use of KOH.

"There were several suggestions for alternate solvents some of which you may have tried, but nevertheless I will list them here:

1. 91% isopropyl alcohol.
2. 99% isopropyl alcohol.
3. Isopropyl alcohol plus Skellysolve B. This is the solvent selected for the determination of gossypol in fats and oils, and according to some may be a very good fat solvent.

"I may say that at the same time I submitted this method to about a dozen Swift Laboratories. The comments from this group were far more favorable and much less critical.

"At any rate, I should like to resolve this situation now. I have given you a short summary of the comments I got. Insofar as my own comments are concerned, two will about cover it. I think we might

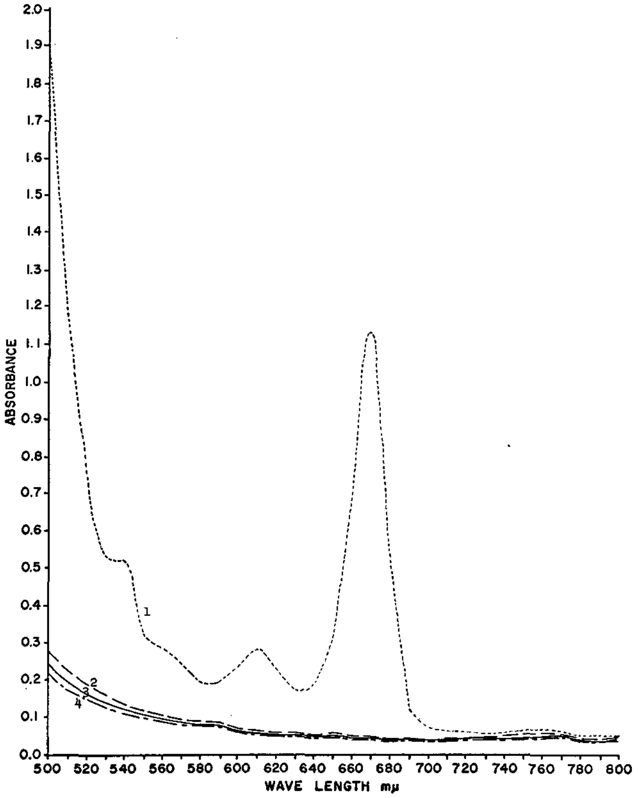

FIG. 9. Refined and Filtered Cottonseed Oil Bleached with Natural Bleaching Earth—Official since October, 1960.

Absorbance Curves 5 cm cell

1. Unbleached Oil
2. Bleached with 4.00% Earth
3. Bleached with 5.67% Earth
4. Bleached with 7.33% Earth

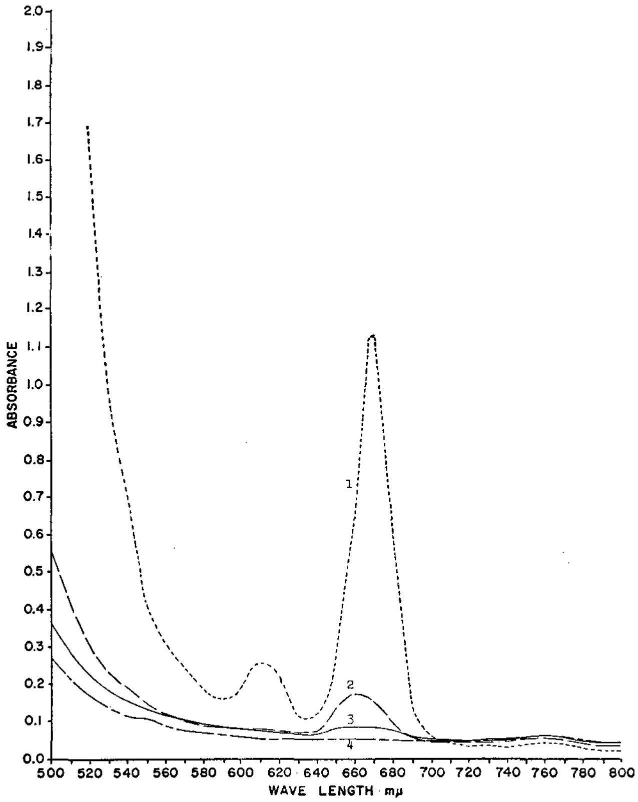

FIG. 10. Refined and filtered Soybean Oil Bleached with Natural Bleaching Earth—Official since October, 1960.

Absorbance Curves 5 cm cell

1. Unbleached Oil
2. Bleached with 1.5% Earth
3. Bleached with 3.0% Earth
4. Bleached with 4.5% Earth

use a little larger sample size than was specified in the procedure and I don't like the use of benzene. We are giving the gossypol solvent a little test in this laboratory and will let you know what we think about it after we have finished.

"Following a review of the foregoing, and of other comments concerning the proposed revision, it was decided at that time by the chairman of the Fat Analysis Committee and the chairman of the Subcommittee that there was no need for further Subcommittee work on the method. Nevertheless, the proposed revision was never submitted to the Uniform Methods Committee, but was held in abeyance for later consideration, apparently because the proposed revision involved rather marked changes from the existing official method.

"Recently, the proposed revision has been reviewed again by members of the Subcommittee. They have agreed unanimously that, after making several typographical corrections, the proposed revision in Method Ca 5a-40 should be recommended again for submission to the Uniform Methods Committee.

"It should be mentioned that the following additional comments and criticisms have been elicited:

1. That consideration should be given to adding the phenolphthalein indicator to the entire batch of solvent described in section B.2.
2. That a warning note concerning the toxicity of benzene should be included in the write-up of the method.
3. That possible differences in results obtained by the colorimetric and electrometric methods may occur in some samples and the validity of note E.2. is therefore questioned, on the grounds that no correction should be permitted in the instrumental method which would lead to a result different from that given by the colorimetric method.

"None of the members of the Subcommittee indicated any need for changing the proposed revision on any of the foregoing grounds, with the possible exception of point 3. The Subcommittee therefore recommends further that, if and when the Uniform Methods Committee considers the proposed revision, it should also consider whether point 3 is sufficiently important to merit further attention."

General Discussion

Although several of the subcommittees have not reported this year, this does not mean that they have not been active. It merely means that at the time of the report no work was ready to be reported. Other work than that reported above has been done by the Commercial Fats and Oils Analysis Committee and will be mentioned in some detail.

"1. *AOCS Procedure for the Determination of Neutral Oil Ca 9f-57.* Since the National Soybean Processors Association is seriously considering going over to the trading of oils on a neutral oil loss procedure, the method for neutral oil has been expanded to include determination of neutral oil loss. The procedure has been revised in detail, has been checked by a special committee and has been issued to the Smalley Committee for checking on cooperative samples during the fall and winter of 1962–63. Copies of the revised procedure, which was agreed upon at a special committee meeting in Chicago, have been sent to the Uniform Methods Committee for their consideration.

"2. *Bleach Test for Soybean Oil.* Since the use of the new procedure for determination of neutral oil loss will still require a single refining for the determination of bleach test, a modified bleach test for soybean oil has been investigated and the procedure written. This procedure has been designated for use with the neutral oil method, Ca 9f-57. Copies of this procdure have been given to the Uniform Methods Committee for their approval.

"3. *Sampling and Analysis of Commercial Fats and Oils.* Procedure D of AOCS methods Ca 1-47 is for 'Continuous Flow Method' for sampling tank or tank cars during loading or unloading. Work continues to be done on this procedure, in order to control more satisfactorily the amount of sample taken. It is hoped that recommendations on this method will be forthcoming next year.

"4. *Additional Work.* In 1963, such additional work by the Committee will be undertaken as is needed to revise existing procedures giving trouble or carrying out work suggested by the Uniform Methods Committee."

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• *Errata*

JAOCS, 40, page 301, July, 1963. ANDERSON AND WOOD: LANOLIN NEE WOOLGREASE. Table I should read as follows:

TABLE I

Composition of Acid and Unsaponifiable Fractions of Wool Wax

Composition	Weight %
A. Acid Fraction	
Alkanoic acids	60.0
α-Hydroxy acids	30.0
ω-Hydroxy acids	5.0
Undetermined	5.0
B. Unsaponifiable Fraction	
Hydrocarbons	0.3
Monoalcohols	9.5
α, β-Diols	6.5
Cholesterol	31.0
Lanosterol	44.0
Undetermined	8.7

JAOCS, 40, page, 32, January, 1963. FORE ET AL.; THE PREPARATION OF LAURYL ALCOHOL AND 6-HYDROXYCAPROIC ACID FROM PETROSELINIC ACID. Table I should read as follows:

TABLE I

Analyses of Crude Products from Reduction of Ozonized Ethyl Petroselinate

Component	Adsorption chromatography		
	Peak eluant volume	$NaBH_4$ reduction product	Catalytic hydrogenation product
	ml	%	%
Lauryl alcohol	480 ± 20	55.3	48.8
Ethyl hydroxycaproate	770 ± 70	41.7	40.7
Ethyl palmitate Ethyl laurate	145 ± 25	3.1	9.6
Diethyl adipate	310	0	0.9
Lauryl aldehyde	......	......	......
	Gas-liquid chromatography		
	Rentention time	$NaBH_4$ reduction product	Catalytic hydrogenation product
	min	%	%
Lauryl alcohol	7.8	55.5	45.0
Ethyl hydroxycaproate	17.9	42.3	42.9
Ethyl palmitate	21.0	1.3	1.2
Ethyl laurate	5.2	0	6.4
Diethyl adipate	11.2	0	4.1
Lauryl aldehyde	3.8	0.9	0.4