Report of the Bleaching Methods Committee 1945-1946

THIS committee has been occupied during the past season on assignments largely growing out of the Uniform Methods Committee meeting in Memphis [1] a year ago, primarily in connection with the methods' revision.

Designation of Filter Paper. An earlier committee, with good reason, introduced the requirement that the filter paper used be one of fine porosity. It was felt that at least one and preferably two or three grades should be named as meeting the requirements. Accordingly, as the result of work done by several members of the committee on six or seven different filter papers covering two types, fine porosity and coarse porosity, the committee recommends by unanimous vote that four of these fine porosity papers be named in the method, where at present only one brand and number is named. (One member favors, in addition, the naming of the coarse porosity papers, possibly designating that two such be used together.) These four were shown to be substantially equivalent in respect to filter rate and retentiveness or porosity. It might be remarked that these papers begin to pass clear filtrate when about 20 ml. has run through and that if the papers are fluted, considerable time is saved in collecting a color tube full, as compared with the case of using a single coarse porosity paper even though the latter filters at a much faster rate. The coarse paper requires the build-up of a layer of spent earth before filtration becomes effective, and hence there is a relatively long period during the first half to two-thirds of the filtration when oil of some degree of turbidity could be taken by a careless or inattentive technician. This objection would be overcome to some extent by using two such papers.

TABLE I.

Additional Data Correlating 4% Activated Clay Bleach Test and 6% Official A.O.C.S. (English) Fuller's Earth Bleach Test on Fresh-Refined Soybean Oils.

Laboratory and Member	Sample	Refined Oil Color	Activated Clay (N.S.P.A. Official) 2%	Activated Clay (N.S.P.A. Official) 4%	Official A.O.C.S. Fuller's Earth 6%
Barrow-Agee (G. Worthen Agee)	1	70y/7.8r	35y/3.3r	20y/2.0r	35y/3.5r
	2	70 6.8	18 1.8	10 1.0	22 2.2
SK & S (Egbert Freyer)	1(JG)	70 11.6	20 1.8	10 1.1	35 2.5
	2(JF)	70 7.6	20 2.1	12 1.1	25 2.1
	3(VS)	.....	.....	10 0.8	30 2.5
	4(VS)	.....	.....	10 1.1	35 3.1
	5(VS)	.....	.....	11 1.0	35 3.2
	6(VS)	.....	.....	11 1.0	30 3.3
Procter & Gamble (J. H. Sanders)	1	35 9.3	20 2.4	14 1.6	25 2.5
	2	35 8.1	15 1.7	8 1.0	20 2.0
	3	35 8.2	15 1.8	8 0.8	20 2.0
	4	50 9.5	20 2.3	12 1.4	25 2.4
	5	50 7.2	13 1.5	7 0.8	20 2.0
	6	50 7.7	14 1.6	7 0.9	20 2.0
Lever Brothers (R. J. Houle)	1	.....	20 1.7	8 0.7	30 2.1
	2	.....	20 1.8	8 0.7	30 2.0
	3	.....	30 3.0	14 1.3	30 2.7
	4	.....	30 2.9	14 1.3	30 2.7
Central Soya Co. (N. F. Kruse)	1	.....	.....	7 0.7	20 1.4
	2	.....	.....	7 0.7	20 1.4
	3	.....	.....	7 0.7	20 1.4
	4	.....	.....	7 0.9	20 1.6
	5	.....	.....	7 0.9	20 1.6

This is the fourth set of such data assembled over a three-year period. When plotted (A.O.C.S. vs. N.S.P.A.—red colors), the points constituting all sets fall in the same area on the graph and are closely intermingled, thus revealing no evidence that the activated clay stock has changed measurably in activity in respect to the A.O.C.S. official fuller's earth, as would be manifest by a uniform displacement of one set of points in relation to the ones representing the earlier comparisons.

The question of whether there might be some slight bleaching effect resulting from adsorption of color on the filter paper fibers was considered and tested. There was no evidence that this occurred in the case of the filter papers studied, which varied considerably in bulk; hence we are led to dismiss this as being a possible cause of color reading discrepancies ascribable to the use of different kinds of filter paper.

Checking the Stability of the N.S.P.A. Official Activated Clay. Inasmuch as last year's committee work led to the adoption of the activated clay bleach test for use on soybean oil, the reference in our method to an official activated clay naturally presupposes the availability of such. Assuming that the N.S.P.A. stock of activated clay could be brought under A.O.C.S. auspices, the chairman of the Uniform Methods Committee requested that additional evidence be furnished demonstrating the stability of that clay over the period of its history to date. Therefore five different laboratories supplied comparative bleach test data on a total of 23 different refined soybean oil samples showing the relation of the bleach tests using 6% of official fuller's earth and using 4% N.S.P.A. activated clay. The data is given herewith (Table I) and its interpretation clarified by the following paragraph quoted from a committee letter:

> The test of stability has been uniformity of the average relationship between the activated clay test and the old A.O.C.S. test. Assuming that the official A.O.C.S. fuller's earth has remained unchanged and continues to be unchanged, constancy of the above relation may be considered to indicate that the official N.S.P.A. activated clay has also remained constant in activity.

In view of all the evidence accumulated in the past three years, the committee voted unanimously to endorse the N.S.P.A. Official Activated Clay.

Bleaching Method for Cottonseed Oil. Another point brought out at the Memphis meeting in discussing the methods' revision was that it would be a distinct forward step and make for simplicity in the Methods' Book and less possibility for confusion if the different bleach tests could be consolidated. More important than that however would be the advantage that laboratories regularly testing both soybean and cottonseed oils would have to be geared only to one procedure, instead of two—a distinct convenience in the routine. Now the committee for 1941-42 went to considerable length to confirm the work of Gudheim, Munsberg, and Robertson (Oil and Soap, *16,* 153, 1939) showing that the old bleach test procedure (the current one for cotton oil) gave unrepresentative high results due to heat darkening when applied to soybean oil. The outgrowth of that work was the present method for soybean oil in which the earth is added to the cold oil before heating to 120° C. with agitation. It would thus be a distinctly backward step to revert to the cottonseed oil bleach procedure for soybean oil, besides resulting in changing the bleaching standard on the latter oil by .3 to .5 red.

Recalling that the above-mentioned authors had reported that cottonseed oil did not undergo deleteri-

[1] Spring, 1945.

ous heat darkening during the bleach test and that consequently the same bleach test results were obtained regardless of which procedure was used, the chairman arranged to attempt to confirm this contention on a more elaborate scale with the object of recommending, if the results justified such action, that the bleach test procedure for cottonseed oil be unified with that now designated for soybean oil. Specifically for this job, he enlisted the aid of several men named below who are not members of the Bleaching Methods Committee, with the object of having this job largely done by people whose primary interest is cottonseed oil. This group therefore combined with the regular Bleaching Methods Committee constituted in effect a task committee for this assignment.

The results of making comparison tests on refined cottonseed oil samples covering a fairly good range of quality, using both the official method for cottonseed oil (adding earth to hot oil) and the present tentative procedure for soybean oil (adding earth to cold oil) are given in Table II. It is noted that the difference in the bleach test results on these oils bleaching prime or better is negligible, where any difference is apparent at all, in the large majority of cases. In those instances wherein the difference was significant: i.e., the cottonseed oils bleached slightly lower by the soybean oil procedure, these were on off-bleaching oils and thus have no relationship to any trading standard, which is concerned only with oils bleaching at or near 2.5 red. One collaborator remarked: "In my opinion some cottonseed oils (particularly the darker ones) are slower in reaching maximum bleach than others." It is considered that the intent of the designation of 5 min. bleaching time is to attain the optimum bleach on the great majority of oils. It is of course to be expected that certain individual samples might require a minute or two longer to attain the optimum bleach. It thus appears that when a slight or moderate difference in results between the two bleach tests does occur, as in a few instances, this is a result, not of heat darkening of the cottonseed oil when bleached by the official procedure, but by the attainment of a slightly lower color by the soybean oil method consequent upon the presence of the earth in the oil during the minute or so when it passes through the 80-120° temperature range.

As implied above, the only significance that a slight drift in the test indications would have in the case of samples with bleach tests falling *well within* either the prime bleach category or the off bleach category is of concern only to refinery people in connection with operations control. As appears below, the representatives of this group are unanimous that this constitutes no barrier or valid objection to unifying the bleach tests. As concerns the extent of the indicated drift in the bleach test results on the grade standard, these results show that the standard grade's cutoff would be shifted by not more than .05 red on the average; and if we may disregard differences of ±.1 red in this work then we can say that there would be no drift at all for about three-fourths of the samples; whereas the other fourth might be upgraded into the prime category where they may otherwise be close to the edge; and if we may consider 0.2 red difference to be of doubtful significance (and that is indeed close to the commonplace discrepancy in color readings), then 94% of the oils bleaching prime or better can be said to have exhibited no significant difference in response to the two procedures. However in line with the above quoted comment it seems to the committee that this small minority of samples, which we might consider to be slightly abnormal in requiring a minute or so additional bleaching time incident to using the soybean procedure, really deserves to be graded up in this slight degree; for, whether or not the slight difference noted results from a small degree of heat darkening to which this particular minority of oils might be susceptible, or merely to the fact that these oils need a minute or two more bleaching time (which they get under the proposed procedure), it is appropriate to consider that in actual plant practice the oil will be given a bleaching time considered to be optimum. Hence the indication that a small fraction of oils might be thrown just over the line into the prime bleach category is not considered by the committee to be objectionable and of sufficient cause to prevent unification of the two bleach test procedures. Indeed we consider that the test procedure wherein the earth is added to the oil cold *actually gives more characteristic and truer bleaching results* in those few instances where there may be any difference at all.

TABLE II.

Effect of Using Soybean Oil Bleaching (Test) Procedure on Cottonseed Oil.

Members and Laboratory	Official Bleach	Red Reduction
R. R. King	20y/2.7r	0
A. D. Campbell, Jr.	20 1.2	0
Interstate Cotton Oil Ref. Co.	20 1.4	0.1
	20 3.0	0.2
	20 3.2	0
G. Worthen Agee	15y/1.5r	0
Barrow-Agee Laboratories	30 2.8	0
	30 3.1	0.1
	35 3.8	0.1
	35 8.0	0.2
J. H. Sanders	15y/1.8r	0
The Procter & Gamble Co.	25 2.6	—0.1
	25 2.9	—0.1
	25 3.1	0
	20 2.2	—0.1
P. A. Williams	35y/3.8r	0.4
South Texas Cotton Oil Co.	20 1.6	0.3
	20 2.7	0.2
	35 4.3	0.3
	35 4.4	0.3
J. J. Ganucheau	17y/1.7r	0
The Southern Cotton Oil Co., New Orleans	20 2.0	0
	35 4.5	0.1
	35 5.5	0.1
	35 6.5	0.1
W. T. Maxwell	20y/3.7r	0.8
The Southern Cotton Oil Co., Savannah	20 2.7	0.2
	20 3.0	0.2
	20 2.0	0
	20 5.1	0.4
R. J. Houle	17y/1.7r	0.1
Lever Brothers Co., Hammond	17 1.8	0.1
	16 1.9	0.1
	30 2.6	0.2
A. D. Rich	25y/2.6	0.1
The Filtrol Corp.	25 2.8	0.2
	50 5.4	0.2

Frequency Distribution and Summary of Differences

1. On those giving a prime bleach by lower result (total comparisons, 17):

Difference	—0.1	0.0	0.1	0.2	0.3 red
Number	2	6	5	3	1
% of Total	12	35	29	18	6
% Within 0.1r		76			

Net Average Diff. 0.05 red

2. *All Tests*—total number of comparisons, 37:

Difference	±0.1	0.2	0.3	0.4	0.8 red
Number	23	8	3	2	1
% of Total	62	22	8	5	3
% Within 0.2r		84			

Net Average Diff. 0.13 red

For conciseness, the bleach results by the Soybean Procedure have been omitted. The column "Red Reduction" shows the amount by which the result obtained using the Soybean Procedure was lower than the result by the Official Procedure for Cottonseed Oil.

Those members, not on the Bleaching Methods Committee who collaborated in this work, were the following:

R. R. King, Interstate Cotton Oil Refining Co., Sherman, Texas
P. A. Williams, South Texas Cotton Oil Co., Houston, Texas
J. J. Ganucheau, Southern Cotton Oil Co., Gretna, La.
W. T. Maxwell, Southern Cotton Oil Co., Savannah, Ga.
J. H. Sanders, The Procter and Gamble Company, Ivorydale, Ohio

There was no dissenting vote out of the eleven who voted to recommend that the bleach test procedure for cottonseed oil be unified with that now current for soybean oil. If adopted, this change would of course be tentative for at least a year.

Evaluating Bleaching Clay Activity by an Absolute Method. It was proposed that the committee study the possibility of developing some method of determining bleaching activity of fuller's earth independent of (a) a comparison of the activity with that of some other earth and (b) the necessity to use vegetable oils, which are notoriously unstable with respect to their bleaching response. There was some discussion of various approaches to this problem in correspondence but the consensus of opinion in the committee was that it represented a type of investigation which was beyond the scope of committee work, that it constituted really a research problem that might well be the subject of a fellowship investigation. However, two members objected that any method of evaluation which depended upon the bleaching power manifest in use on some "synthetic" oil which could be reproducibly constituted, would not be valid in evaluating bleaching earths as to their activity on natural vegetable and animal oils, which among themselves offer considerable diversity in their response to various bleaching earths.

Recommendations.

1. That in Cc 8a-28 (A-6), where now occurs "E. & D. 192," there shall be substituted the following: "E. & D. 192, R-A 871, Whatman, No. 12, S. & S. 596."
2. That the Society endorse the stock of activated clay which has been standardized and packed in sealed cans especially for use in grading the bleachability of soybean oil; and it is further recommended that this lot be designated Official A.O.C.S. Activated Clay, and a label to that effect be applied to the containers, assuming that suitable arrangement to permit this can be made with the present sponsors of that stock of clay.
3. Combine Methods Cc 8a-28 and Cc 8b-44, particularly with respect to (a) under Procedure in the latter, which will constitute the wording of the unified method. The statement of scope will therefore be broadened to read, "Applicable to refined cottonseed and soybean oils." The line at B. (b), Dc 8b-44, *For Green Oils—High Chlorophyll Content,* will have added to it, "Intended only to apply to soybean oil." This recommendation proposes that in the bleach test procedure for cottonseed oil the 6% official earth be added to the cold oil before heating to 120° C. with agitation in not over 5 minutes.[1]

The committee believes that it has completed the tasks which were assigned to it or which it had assumed, insofar as they can be considered to come within the scope of committee work, and therefore asks that it be discharged.

G. WORTHEN AGEE	A. D. RICH
N. F. KRUSE	HENRY ODEEN
R. A. MARMOR	EGBERT FREYER, chairman
R. J. HOULE	

[1] The recommendations embodied in this paragraph were subsequently approved, but on incorporating the indicated changes into the methods, (Second Edition), editorial requirements made it necessary to depart from the wording suggested here.

The Solvent Extraction of Oil From Acorns*

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THE possibility of using acorns for food has often been discussed in the literature (18,21,27), but except in times of scarcity of other foods, acorns have been utilized in any quantity only by wild animals or semi-domestic pigs (22). There are some references (14,18) to the possible uses of acorn oil, but there is no record of any large scale work with this material. This investigation was undertaken to handle a quantity of acorns, 847 pounds, (a) to disclose what particular problems there are in processing this material for acorn oil, (b) to demonstrate a practical method of obtaining acorn oil, and (c) to relate solvent- extraction theory to the experimental extraction of this oil.

There are three general methods for obtaining oil from oleaginous seeds, expression, expulsion, and solvent-extraction. Solvent-extraction was selected as the most promising method for obtaining oil from acorns since it can be applied to any oil-bearing material and is particularly suited to the removal of oil from materials of low oil content. The oil content of acorns is reported to vary from 2.5% (25) to 16.0% (23).

Acorns. One of the problems of this investigation was to determine what type of material could be expected on offering a price for acorns. Potential suppliers in Arkansas, Virginia, New York State, Florida, and South Carolina expressed a willingness to collect acorns for three cents a pound. There were 361 pounds of acorns received from Gainesville, Florida, and 486 pounds from Anderson, South Carolina. It is our opinion, based on our personal observation and our attempts to get acorns, that they are seldom as abundant as popularly supposed and that harvesting for commercial utilization would be difficult.

* This work was done in the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Va.