and 265, the effect of varying amounts of alkali was studied. On oil 258, the excess NaOH over theory per 100 grams of oil was varied from .35 to .70 gram for the A.O.C.S. method, and from 0.0 to .60 gram excess for the centrifuge method. On oil 265 the excess NaOH ranged from 0.0 to .91 gram on the centrifuge method. For both oils a rather consistent picture was obtained of the refining loss increasing with increasing alkali used, and this seems generally true for all the oils examined here. This is also supported by the 1941 results presented in Series I, referred to above. It appears that factor II, the formation of soaps by excess alkali, comprises the greater part of the foots when the centrifuge method is used, since, in general, the refining loss for a given oil bears more relation to the excess of alkali used than to the F.F.A. of the oil. This receives further support from the experiment on the 265 refined oil, where, when .90 gram of alkali was used in a "re-refining," a loss of 2.6 per cent was obtained, whereas a loss of 2.5 per cent was found using .80 gram alkali on the original oil containing .27 per cent F.F.A.

The use of five times theory is incorrect in that, for oils with a high F.F.A., too much alkali will be used, and in oils with very low F.F.A., too little alkali to give good foots may be used. A more logical choice would be to use alkali equivalent to theory plus .1 or .2 gram. It may also be possible to use the centrifuge method successfully with weaker alkali, and thus further reduce saponification. Inspection of column C in the table shows the large amounts of water used in the A.O.C.S. refining as compared with the centrifuge method. By centrifuging there will be more tendency for this water to separate and caution must be exercised to prevent the occurrence of free water in the foots-oil mixture. Alkali of 12° Bé was used for oil 265, and the results obtained with an equal amount of 30° Bé caustic shows the lower loss with the weaker alkali. On oil 258, a similar comparison was unsuccessful because of the unsatisfactory foots obtained with the weaker alkali.

Further work should be carried out on the following points:

I. Crude undried extracted oils and their effect on the A.O.C.S. method.
II. Limits of amount and strength of alkali in the centrifuge method.
III. Use of the centrifuge method on all types of oil.

Our second refining problem was referred to us by Mr. R. R. King of the Interstate Cotton Oil Refining Company, Sherman, Texas. Mr. King, in his letter of January 19, 1943, called attention to the fact that peanut oil from certain sections of the country was very difficult to refine by the official peanut oil method. He further suggested that the cottonseed oil "slow breaking" procedure was better adapted to this kind of oil.

Accordingly, two samples of peanut oil were sent out. One of these, marked "Georgia" was a normal oil which could be satisfactorily refined by the regular method. The other sample marked "Texas" did not respond to the usual treatment. The results of the tests of the committee are recorded in the attached tabulation and summarized at the end. These results clearly indicate that the "Texas" type of peanut oil does not refine satisfactorily by the official method. However, more work is necessary in the development of a procedure which will effectively handle this type of oil. The "slow breaking" method did not appear to have any particular advantage in the case of the Georgia sample.

Recommendations for Next Year's Refining Committee Activities

(1) It is possible that a refining loss basis of settlement for soybean oil will be established at the start of the next oil season. It is extremely important, in view of this, that we continue to have the assistance of the Northern Regional Research Laboratory. It is the hope of the committee that this will be possible.

(2) Further study should be made of the centrifuge method as outlined by Dr. Milner:
  (a) Limits of amount and strength of alkali in the centrifuge method.
  (b) Use of centrifuge method on all types of oil.

(3) Study of crude undried extracted oils and their effect on the A.O.C.S. method.

(4) Further study of the present A.O.C.S. refining test for peanut oil:
  (a) Application of slow breaking procedure to oils from Texas and Oklahoma.
  (b) Effect of longer settling time with regular method in the case of oils giving unsatisfactory foots with the regular procedure.

H. S. MITCHELL, *Chairman*
R. T. MILNER, *Vice-Chairman*

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C. B. CLUFF	N. F. KRUSE
G. A. CRAPPLE	T. C. LAW
M. M. DURKEE	H. E. MOORE
R. H. FASH	L. A. SPIELMAN
E. B. FREYER	B. L. STERNBERG
A. R. GUDHEIM	W. L. TAYLOR
ARTHUR KIESS	

Interim Report of the Refining Committee (A. O. C. S.)

As stated in the report of this Committee presented at the Annual Meeting, May 12-14, 1943, recent work has been on two different methods of refining soybean oil. As a result of a Committee meeting in Chicago on April 30, 1943, the Northern Regional Research Laboratory was requested to compare these two analytical methods (the present official and tentative A. O. C. S. cup methods and the proposed centrifugal method) on representative samples of all types of soybean oil to be furnished by Committee members. In addition, three sub-committees were established to study modifications of the present A. O. C. S. methods: Mr. S. O. Sorenson was assigned extracted oils; Mr. J. H. Sanders, expeller oils; and Dr. E. G. Freyer,

TABLE I

Oils Studied by Northern Regional Research Laboratory

NRRL No.	Type and Source	Break %	Phos. %	F.F.A. %	Crude Color (1") 70Y + R	Plant Loss and Bleach Color	
						Cent. %	Cent. Color
AO-41	Cl. Ext. Lever P-53	.29	.068	.89	6.3	3.40	44—4.1
AO-22	Ext. P + G 1	.19	.035	.37	5.0	......	....
AO-23	Ext. P + G 2	.06	.008	.66	4.7	......	....
AO-29	Ext. P + G 3	.06	.007	.64	4.3	......	....
AO-36	Ext. Lever P-42	.24	.044	.68	4.8	4.9	35—3.9
AO-27	Exp. Staley	.45	.081	.70	7.3	3.28	....
AO-37	Ext. P + G 1	.56	.111	.48	5.1	......	....
AO-38	Ext. P + G 2	.66	.120	.65	6.0	......	....
AO-35	Exp. Lever P-37	.46	.074	.82	9.6	4.98	35—3.6
AO-39	Exp. Lever P-46	.45	.087	1.24	3.7	5.20	....
AO-40	Exp. Lever P-50	.48	.049	.66	4.8	4.14	35—3.2
AO-43	Exp. Lever P-56	.36	.069	.51	4.8	3.05	35—3.5
AO-44	Exp. Lever P-59	.58	.099	.71	3.5	4.56	44—2.9

hydraulic oils. On July 7-8 a meeting of the Refining Committee was held at the Northern Regional Laboratory, Peoria, Illinois, to consider the results of these studies. Attendance was as follows:

Members and/or Representatives

Durkee, Maurice M.—A. E. Staley Mfg. Co., Decatur, Ill.
Freyer, Egbert—Spencer Kellogg and Sons, Buffalo, N. Y.
Houle, R. J.—Lever Bros. Co. (Edible Lab.), Hammond, Ind.
James, Edward M.—Lever Bros. Co., Winchester, Mass.
Kiess, Arthur—Armour and Company, Chicago, Ill.
Kishlar, Lamar—Ralston Purina Company, St. Louis, Mo.
Kruse, N. F.—Central Soya Co., Decatur, Ind.
McGee, Ray—Durkee Famous Foods, Chicago, Ill.
Milner, R. T.—Northern Regional Research Lab., Peoria, Ill.
Mitchell, H. S.—Swift and Company, Chicago, Ill.
Moore, H. E.—Capitol City Products Co., Columbus, Ohio
Ory, Harold — Ralston Purina Company, St. Louis, Mo.
Sanders, J. H.—Procter and Gamble Co., Ivorydale, Ohio
Sorenson, S. O.—Archer-Daniels-Midland Co., Minneapolis, Minn.
Spannuth, Hiram T.—Wilson and Company, Chicago, Ill.
Taylor, W. L.—Archer-Daniels-Midland Co., Chicago, Ill.

From Northern Regional Laboratory

Earle, F. R.
Goss, W. H.
Macmillan, D.
VonKorff, R. W.

In view of the present intense interest in this subject, this report is published to summarize the discussion at that meeting and the data accumulated since the report at the Annual Meeting of May 12-14. The meeting at Peoria was well attended with 15 members or representatives present. Two sessions were held: From 2-6 p. m. on July 7, and from 8:30 a. m. - 1 p. m. on July 8. The first session was largely devoted to presentation of the experimental data and discussion of it, and the second session to consideration of the conclusions to be drawn from the available knowledge.

The Regional Laboratory received four samples of clarified extracted, three of extracted, eight of expeller, and three of hydraulic oils on its original program to compare refining loss by the present A. O. C. S. methods with modifications of the centrifugal method. Actual plant losses were to be furnished on all of these oils, but it was found possible to do this only on three extracted and three expeller oils. Mr. E. M. James then offered to furnish oils and refining losses as determined in a very complete two-ton pilot plant which has been proved to be equivalent to commercial refinery practice.

Results on all oils were discussed but, to save space, the only data given here in tables I and II are for the oils on which plant losses are available. In table I are listed the crude oils as received, with plant losses. In table II are given the laboratory results. The green grade of all oils was 1 except expeller oil No. AO-39, which was 2. In table II, under "Alkali," there are given first, the strength of caustic soda solution used (°Bé); second, the percentage of alkali (%), that is, the grams of alkali solution per 100 grams of oil; and last, the weight of dry NaOH per 100 grams of oil in excess of that calculated for the amount of free fatty acid measured (Excess over Theory). It should be remembered that 500 grams of oil are used in the A. O. C. S. method and 200 grams in the centrifuge method. The theoretical amount of dry NaOH was calculated as .142 × F.F.A.

In discussing these results, members of the Committee suggested that weaker lye solutions would give better results on many oils and that the temperature of the hot agitation could be lowered to lower the refining loss. The advantages and disadvantages of the two methods were discussed in detail as well as the difficulty of relating any laboratory method to plant results. It was stated that for a given F.F.A., the refining loss will rise as the season progresses. Oil with a low break will give lower losses in December and January than oil of this same break value in June and July. Soybean oil of this season's crop is abnormal in having degenerated phosphatides and products of bacterial decomposition. Dr. E. G. Freyer discussed in some detail the "break factor." Dr. Freyer stated that experience on expeller oil has shown that on a good refining, the loss equals 3 × F.F.A. + 5.5 × Break. Instead of using 5.5, this factor may be calculated from the equation and called the "break factor," which, therefore, equals

Refining loss (actual) — (3 × F.F.A.). In Dr. Freyer's opinion, this Break factor offers a means of evaluating the refining of an oil. The factor would probably vary with type of oil, having an average value of 5.5 for expeller oils. Applying this to the data from the Regional Laboratory, he indicated that the high

TABLE II

Refining Loss and Bleach Color
(Results from the Northern Regional Laboratory)

NRRL No.	Method	Alkali			Loss %	Refined Color (5¼") 70Y + R	Bleach Color (5¼") Y + R	Remarks
		° Bé	%	Excess Over Theory				
Clarified Extracted:								
AO-41	AOCS ⅞ Max	14	6.5	.49	5.6	4.9	70—4.0	
	AOCS ⅔ Max.	14	5.0	.34	3.7	4.6	70—3.9	
	Centrifuge	30	1.9	.30	5.4	6.4	70—4.9	
	Centrifuge	30	3.2	.60	8.8	5.4	70—4.7	
	Centrifuge	30	3.2	.60	7.9	5.8	70—4.1	Only ½-hr. cold; ¼-hr. hot
	Centrifuge	16	3.4	.27	4.2	5.7	70—4.6	
Extracted:								
AO-22	AOCS ⅞ Max	14	5.6	.48	3.6	8.0	35—2.0	
	AOCS ⅔ Max.	14	4.2	.36	4.8	7.7	35—2.0	
	Centrifuge	30	1.5	.30	4.1	9.5	70—2.6	
	Centrifuge	30	2.8	.60	4.3	8.2	35—2.1	
	Centrifuge	30	2.8	.60	5.5	8.6	70—2.2	Only ½-hr. cold
AO-23	AOCS ⅞ Max	14	6.1	.50	5.2	9.1	35—1.7	
	AOCS ⅔ Max.	14	4.7	.37	3.8	9.6	35—1.6	
	Centrifuge	30	1.7	.30	4.0	9.6	35—2.0	
	Centrifuge	30	3.0	.60	6.7	8.8	70—2.3	
AO-29	AOCS ⅞ Max	14	6.1	.49	5.1	8.0	35—1.7	
	AOCS ⅔ Max.	14	4.6	.35	3.9	8.3	35—1.5	
	Centrifuge	30	1.9	.30	4.3	9.6	35—1.5	
	Centrifuge	30	3.0	.60	6.6	9.2	35—1.7	
	Centrifuge	30	3.0	.60	6.9	8.7	35—1.6	Only ½-hr. cold
AO-36	AOCS ⅞ Max	14	6.2	.49	4.7	7.4	70—4.8	
	AOCS ⅔ Max.	14	4.8	.35	4.5	6.9	70—5.1	
	Centrifuge	30	1.8	.30	5.3	8.1	70—5.7	
	Centrifuge	30	3.1	.60	7.1	9.1	70—5.5	Refined 6-16-43
	Centrifuge	30	3.1	.60	7.1	9.1	70—5.5	Refined 6-23-43
	Centrifuge	30	3.1	.60	4.6	7.6	70—5.3	Only ½-hr. cold
	Centrifuge	30	3.1	.60	4.2	7.8	70—5.4	Only ½-hr. cold; ¼-hr. hot
	Centrifuge	16	3.6	.30	4.5	6.6	70—4.5	
Expeller:								
AO-27	AOCS Max.	12	8.4	.57	5.2	7.9	70—3.0	
	AOCS ⅔ Max.	12	5.6	.35	5.1	8.0	70—3.1	
	Centrifuge	30	1.9	.30	5.6	9.6	70—3.7	
	Centrifuge	30	3.2	.60	6.8	8.7	70—3.1	
	Centrifuge	16	3.6	.30	4.6	8.6	70—3.4	
AO-37	AOCS Max.	12	7.9	.56	5.8	3.4	70—4.6	Refined color doubtful
	AOCS ⅔ Max.	12	5.3	.35	3.9	3.5	70—4.6	Refined color doubtful
	Centrifuge	30	1.6	.30	5.2	6.4	70—4.3	
	Centrifuge	30	2.9	.60	6.7	4.9	70—4.0	Refined 6-23-43
	Centrifuge	30	2.9	.60	6.8	3.6	70—3.7	Refined 7-2-43
	Centrifuge	16	3.3	.30	4.5	4.8	70—4.2	
AO-38	AOCS Max.	12	8.4	.58	6.4	3.2	70—5.5	Refined color doubtful
	AOCS ⅔ Max.	12	5.6	.34	5.6	3.7	70—4.6	Refined color doubtful
	Centrifuge	30	1.7	.30	6.1	5.1	70—4.1	
	Centrifuge	30	3.0	.60	7.3	3.9	70—4.2	
	Centrifuge	16	3.5	.30	4.7	4.1	70—4.1	
AO-35	AOCS Max.	12	8.7	.58	5.8	3.3	70—3.6	
	AOCS ⅔ Max.	12	5.8	.35	5.6	3.8	70—3.8	
	Centrifuge	30	1.8	.30	5.7	6.6	70—5.0	
	Centrifuge	30	3.1	.60	7.2	4.6	70—4.3	
	Centrifuge	30	3.1	.60	6.8	4.8	70—4.2	Refined 6-17-43 Only ½-hr. cold
	Centrifuge	30	3.1	.60	7.3	4.2	70—3.5	Refined 6-19-43
	Centrifuge	16	3.8	.30	4.8	3.8	70—4.2	
AO-39	AOCS Max.	12	9.9	.60	5.9	....	35—3.6	Bleach colors doubtful
	AOCS ⅔ Max.	12	6.5	.34	5.9	....	35—3.5	Bleach colors doubtful
	Centrifuge	30	2.1	.30	7.2	....	70—8.0	
	Centrifuge	30	3.4	.60	9.4	....	70—2.8	
	Centrifuge	30	3.2	.55	7.7	....	70—4.0	Only ½-hr. cold
	Centrifuge	16	4.3	.30	5.7	....	70—2.8	
AO-40	AOCS Max.	12	8.4	.58	6.7	3.2	70—2.8	
	AOCS ⅔ Max.	12	5.6	.36	6.6	3.3	70—3.2	
	Centrifuge	30	1.7	.30	6.2	4.7	70—3.4	
	Centrifuge	30	3.0	.60	6.9	3.8	70—3.3	
	Centrifuge	30	3.0	.60	6.6	4.5	70—2.9	Only ½-hr. cold
	Centrifuge	16	3.5	.30	5.2	4.0	70—3.3	
AO-43	AOCS Max.	12	8.2	.57	3.7	3.1	70—3.3	
	AOCS ⅔ Max.	12	5.5	.36	3.7	3.2	70—3.2	
	Centrifuge	30	1.6	.30	4.2	3.9	70—4.0	
	Centrifuge	30	2.9	.60	4.2	3.5	70—3.5	
	Centrifuge	30	2.9	.60	3.9	3.6	70—3.3	Only ½-hr. cold
	Centrifuge	16	3.3	.30	4.2	3.3	70—3.4	
AO-44	AOCS Max.	12	8.7	.58	5.6	4.1	70—3.0	
	AOCS ⅔ Max.	12	5.8	.35	5.1	4.7	70—2.3	All refined-colors doubtful
	Centrifuge	30	1.8	.30	6.0	11.5	70—2.8	
	Centrifuge	30	3.2	.60	7.0	5.1	70—3.1	
	Centrifuge	30	3.2	.60	6.9	4.6	70—2.8	Only ½-hr. cold
	Centrifuge	16	3.6	.30	4.9	6.2	70—3.0	

break factors found for hydraulic and extracted oils by the A. O. C. S. methods showed a poor refining. Dr. Freyer suggested weaker alkali for these cases. He also suggested that lye tables be constructed to take account of both break and F.F.A. in obtaining the caustic requirements.

Mr C. B. Cluff, who was not at the meeting, commented on the data as follows:

"In comparing two methods such as the present official procedure and the proposed centrifugal procedure, I think it important to eliminate other variables such as differences in the strength and amount of lye used, because any difference in result might be due to the lye instead of to the method used. If this should be the case, it would of course be entirely possible to change the lye specified in the official method. I do not feel myself that the requirement for using 12° lye on expeller oil and 20° on hydraulic oil is necessarily the best, for the reason that these lyes were established several years ago at a time when nobody had very much experience and the experiments made at that time were not based on a very large number of samples. The method adopted at that time was only tentative, but I believe was made official a year ago, but this was merely because nobody had complained of the requirements, and this in turn was probably due to lack of broad experience. I think this point could well be gone into again.

"Soft foots are indicated in several of your tests, but I believe only when relatively weak lye was used. This has never been a serious obstacle in making refining tests on cottonseed oil because it does not prevent determining the true loss. The foots can usually be hardened by chilling in cold water, or the condition could probably be prevented by using a stronger lye.

"The data you submit on the various kinds of oil shows as follows:

Clarified extracted oil, 4 samples, all show best results by the official method.

Extracted oil, 4 samples, all show best results by the official method.

Expeller oil, of 5 samples, 3 show best results by the official method and 2 by the centrifugal method.

Hydraulic oils, of 3 samples, all show best results by the centrifugal method.

Miscellaneous oils, omitting AO-39 on which colors are stated to be doubtful, of the 3 remaining samples, all show best results by the official method.

"In view of the data which you show, it seems to me that, on the whole, centrifugal method shows little, if any, advantage over the official method, and in any event we must bear in mind that a different concentration of lye was used in the centrifugal. I would think that other tests ought to be made comparing the two methods with the same lye before concluding that either method, considered as a method by itself, has any advantage over the other.

"In comparing the data shown, I have considered the best refining to be the one that gives the lightest bleach, provided that the difference in loss as compared with the next best test is not more than 2.5 times the difference in bleach. That is to say, if two tests vary in bleach by .2 red, then the loss must not be more than .5% higher in the test giving the lighter color. This ratio of higher loss to lighter bleach has been obtained by averaging a large number of tests made on oils wherein the bleach and loss varied, using, however, cottonseed oil instead of soybean oil, and has been checked many times. It is at least approximately reliable for small differences, but I would not use it for correcting large differences in bleach color.

"The object of any laboratory test is to serve as a basis for trading purposes, and the results must be easily and accurately reproducible in different laboratories. Also, the test would be expected to show somewhere near what would be obtained in the plant by ordinary refining procedures efficiently conducted, but this has not been considered the main object in the cottonseed oil refining test. The primary requisite is that all chemists must get the same result. . . . .

"Inasmuch as all oil laboratories are already equipped with apparatus for the present official method, I would suggest that it would be highly preferable to retain the same apparatus for use on soybean oils with such modifications as are necessary regarding the lye and temperatures, etc., rather than require all laboratories to purchase new equipment, such as would become necessary if the centrifugal method should be adopted. This, I think, could be justified only if the results obtainable are decidedly superior in consistency to any that can be obtained by suitable modifications of the official method."

Mr. E. R. Barrow, who could not attend, commented:

"I have read Dr. Milner's report with considerable interest, and, in scanning the results, they would appear to predict the selection of the centrifuge method for all three types of refinings. My opinion is that the important point is to ascertain whether the laboratory method approximates the results which are obtained in plant practice.

"Our laboratory is not equipped for the centrifuge method, but, in anticipation of this development, we have secured a priority rating from W. P. B. and are ready to order the centrifuge as soon as we know definitely the correct recommended type and accessories.

. . . .

"In Dr. Milner's description of the method, he proposes the use of the Trunion cup as the refining cup. According to Fisher's catalog, each Trunion cup made of bronze costs $8.50 each. In a laboratory handling a large number of oil samples, the use of these cups would make the cost prohibitive and furthermore we doubt that they could be obtained in quantities, made of bronze. The Duraluminum cups are prohibitive.

"Assuming that the refining test would be run in duplicate on settlement samples, only two samples could be handled at one time on the centrifuge. This method would undoubtedly require a heavy outlay for apparatus and would make the refining test more costly than by the present method. Also, the time consumed in the method would require a lot of manipulation and cut down the volume of work.

"We would like for you to take up with the Committee these detail points and seek an agreement on the exact type of equipment to be used, with catalog reference if possible. We would like to have a complete list of this equipment as soon as possible in order to equip our laboratory for this work and possibly in time to participate in some of the cooperative work of the Committee.

TABLE III

Summary of Refining Tests on Expeller Oils
(Results from J. H. Sanders)

	12° Bé Lye						14° Bé Lye						16° Bé Lye					
	% Loss			Bleach Color			% Loss			Bleach Color			% Loss			Bleach Color		
% Max. Lye:	66⅔	80	100	66⅔	80	100	66⅔	80	100	66⅔	80	100	66⅔	80	100	66⅔	80	100
Oil No. 1	8.0	8.0	8.0	30—2.8	35—2.3	25—2.4	7.7	8.1	8.2	30—2.9	35—2.4	25—2.5	7.6	7.9	7.9	35—2.7	35—2.7	35—2.3
Oil No. 2	4.2	4.3	4.0	35—2.5	35—2.4	35—2.3	4.1	4.7	4.6	35—2.7	35—2.7	35—2.2	4.4	4.5	4.7	35—2.5	35—2.4	35—2.2
Oil No. 3	6.3	6.3	7.3	30—2.4	30—2.4	30—2.4	6.4	6.0	7.2	30—2.4	30—2.3	30—2.3	6.0	6.0	6.8	30—2.6	30—2.2	30—2.3
Oil No. 4	6.2[1]	6.2	6.6	....	....	....	6.2[1]	6.2	6.7	....	....	....	6.2[1]	6.2	8.4	....	....	....
Average	6.2	6.2	6.5	32—2.6	33—2.4	30—2.4	6.1	6.3	6.7	32—2.7	32—2.4	30—2.3	6.1	6.2	7.0	33—2.6	33—2.4	33—2.3
With 0.5% Silicate:																		
Oil No. 1	7.7	7.4	8.7	25—2.7	35—2.8	25—2.4	7.5	7.6	7.8	30—2.9	35—2.8	25—2.5	7.2	7.2	8.0	35—2.9	35—2.7	35—2.5
Oil No. 2	4.4	4.0	4.1	35—2.9	35—2.9	35—2.7	4.0	4.0	4.2	50—2.7	50—2.6	35—2.4	4.3	4.1	4.4	35—2.8	35—2.5	35—2.4
Oil No. 3	5.8	6.5	6.4	35—2.8	30—2.5	30—2.6	6.2	6.2	6.2	35—2.7	35—2.6	35—2.7	6.2	6.0	6.4	35—3.1	30—2.4	30—2.3
Oil No. 4	6.5[1]	6.5	6.9	....	....	....	5.7[1]	5.7	7.6	....	....	....	6.1[1]	6.1	8.3	....	....	....
Average	6.1	6.1	6.5	32—2.8	33—2.7	30—2.6	5.7	5.7	6.5	38—2.8	40—2.7	32—2.5	5.8	5.7	6.6	35—2.9	33—2.5	33—2.4

[1] Assumed same as 80% maximum for same conditions.

"There is one detail of the method outlined on page 162, volume 19, OIL & SOAP, which appears to me as impractical, namely, numbers 11, 12, and 13 of the procedure. Would not the soapstock, heated to 75° C. and then centrifuged for 30 minutes, be too soft to drain for 30 minutes without loss of soapstock? Other than this point, the method appears practical, except for the time consumed and the expense of the apparatus involved."

In general discussion of the two methods, the A. O. C. S. and the centrifuge, it was stated that, although the centrifuge method required only four hours to complete, if a large number of determinations was run this apparent advantage would disappear, and in continuous working, as large a number of refinings could be made by the A. O. C. S. as by the centrifuge.

It was agreed that the availability of equipment (centrifuges) and the imminence of a new crop should not influence consideration of refining loss tests. These tests are to be established as the best analytical procedures available to give consistent reliable results.

In tables III, IV, and V are given results of tests on variations in the present A. O. C. S. procedures for expeller, extracted, and hydraulic oils as presented by Messrs. Sanders, Sorenson, and Freyer, respectively.

Mr. Sanders (table III) stated:

"On four different crude expeller oils, these conditions were varied: Amount and strength of lye, the use of silicate, the time in the cold, and the finishing temperature. Over one hundred cup tests have been made.

"These conclusions can be drawn from the work:

1. The present official method is satisfactory for trading purposes.
2. The foots produced with the present 12° Be′ lye are soft, and require extreme care in decantation of the oil. Strengthening the lye to 14° Be′ or 16° Be′ would reduce the risk of error, without any adverse effect.
3. One-half percent silicate added with the lye will reduce refining loss.

"The study should be extended to additional oils before any change in the existing method is recommended."

Mr. Sorenson (table IV) stated:

"For this work we obtained a quantity of extracted oil produced by our Decatur plant, having the following characteristics:

% Free Fatty Acids	.44
% Break	.20
Color in a 5¼″ cell	210 yellow—18 red

"In laying out this work it was decided to study the effect of refining with the theoretical amount of alkali as the basis and using multiples of this amount as excesses in succeeding work. Also as a start it was decided to use 25% sodium hydroxide solution in this work, making deviations from this concentration in the future as we thought desirable. The data on Run No. 1 were as follows:

"The refining was performed by cutting the oil at 85-90° F. (29.4-32.2° C.) with the alkali under slow agitation. The cut oil was agitated slowly at this temperature for an additional 20 minutes. The temperature was then raised to 130° F. (58° C.) in 60 minutes with slow agitation. The slow agitation was continued to 140° F. (60° C.) in an additional 30 minutes. Agitation was then stopped and the oil settled for 1 hour at 140° F.

	Amount of 25% NaOH	Soap Formation	Soap Settling at 140° F. (60° C.)
1.	1 c.c. = theoretical	Good break	Good
2.	2 c.c. = 2 × theoretical	Good break	Good
3.	3 c.c. = 3 × theoretical	Good break	Good
4.	1 c.c. NaOH + 10 c.c. H_2O	Fine grain	Good
5.	2 c.c. NaOH + 10 c.c. H_2O	Fine grain	Good
6.	3 c.c. NaOH + 10 c.c. H_2O	Med. grain	Good

"After settling the soap at 140° F., it was cooled in a bath at 65° F. (18.3° C.) for 1 hour and then allowed to stand overnight. After chilling an additional half hour at 65° F., the oil was poured from the foots with the following results:

1. Foots loose .... 3.2% loss
2. Foots loose but better......................... 5.0% loss
3. Foots loose .... 8.4% loss
4. Foots firm but slimy........................... 2.0% loss
5. Foots firm but slimy........................... 1.2% loss
6. Foots firm but slimy........................... 2.2% loss

TABLE IV[1]

Extracted Oil (Data from S. O. Sorenson)
Crude Extracted Oil, F.F.A.—.44%; Break—.20%;
Color (5¼″) 210—18

Procedure: Alkali was added to the oil at 85°-90° F. (29.4-32.2° C.) with slow agitation, continued for 20 minutes. The temperature was then raised to 130° F. (54.4° C.) in 60 minutes and to 140° F. (60° C.) in an additional 30 minutes, all with slow agitation. Agitation was then stopped and the oil settled for one hour at 140° F., cooled at 65° F. (18.3° C.) for one hour and stood overnight. After chilling one-half hour at 65° F., the oil was poured from the foots.

For this oil, theory (alkali) = .142 × .44 = .0625, maximum = .44/5.2 + .54 = .624.

Alkali					Loss %	Condition of Foots
c.c. 25%	c.c. H_2O	Max. %[1]	°Bé[1]	Excess Over [1] Theory		
1	0	.10	31	0	3.2	Loose
2	0	.19	31	.06	5.0	Loose but better
3	0	.29	31	.12	8.4	Loose but better
1	10	.10	4.2	0	2.0	Firm but slimy
2	10	.19	7.7	.06	1.2	Firm but slimy
3	10	.29	10.1	.12	2.2	Firm
4	10	.39	12.0	.18	4.5	Slippery
5	10	.48	13.7	.24	9.0	Slippery
8	10	.78	17.3	.42	14.0	Slippery
10	10	.97	18.5	.54	22.0	Loose
8.7	15	.86	14.8	.47	16.0	Loose
6.6	15	.65	13.0	.34	13.0	Loose
2	5	.19	12.1	.06	7.0	Loose
3	10	.29	10.1	.12	3.0	Firm
4	15	.39	9.2	.18	3.6	Loose
5	20	.48	8.8	.24	4.4	Loose
6	25	.58	8.5	.30	6.4	Liquid
7	30	.68	8.4	.36	10.0	Liquid

[1] Constructed from data submitted with values for Max. %, °Bé, and Excess Over Theory calculated and perhaps in slight error.

"Since the last result above gave the best results as far as separation of neutral oil was concerned, it was decided to make another series of refinings extending the amount of alkali used up to 10 times theory. Also included in these series of refinings were two refinings using the amount of alkali called for by the official A. O. C. S. formula for extracted oil. The data on these runs were as follows:

	Amount of Alkali	Soap Formation	Foots Characteristics
1.	4 c.c. of 25% + 10 H_2O	Good break	Slippery foots — 4.5% loss
2.	5 c.c. of 25% + 10 H_2O	Good break	Slippery foots — 9.0% loss
3.	8 c.c. of 25% + 10 H_2O	Good break	Slippery foots —14.0% loss
4.	10 c.c. of 25% + 10 H_2O	Med. fine	Loose foots —22.0% loss
5.	8.7 c.c. of 25% + 15 H_2O	Fine grain	Loose foots —16.0% loss
6.	6.6 c.c. of 25% + 15 H_2O	Fine grain	Loose foots —13.0% loss

"These results in conjunction with the results obtained in Run No. 1 indicate that the optimum amount of alkali to use for the best refining procedure thus far is 3 to 4 times the theory of alkali. Another series of refinings was made using from 2 to 7 times theory of 25% alkali solution plus an additional amount of water varying from 5 to 30 c.c. with the increasing amounts of alkali. The data on this series of refinings were as follows:

	Amount of Alkali	Foots Characteristics
1.	2 c.c. of 25% + 5 c.c. H_2O	Loose foots — 7.0% loss
2.	3 c.c. of 25% + 10 c.c. H_2O	Foots firm — 3.0% loss
3.	4 c.c. of 25% + 15 c.c. H_2O	Foots loose — 3.6% loss
4.	5 c.c. of 25% + 20 c.c. H_2O	Foots loose — 4.4% loss
5.	6 c.c. of 25% + 25 c.c. H_2O	Foots liquid — 6.4% loss
6.	7 c.c. of 25% + 30 c.c. H_2O	Foots liquid —10.0% loss

"These results again confirm that about 3 times the theoretical 25% alkali plus an additional 10 c.c. of water gives the best results as far as foots characteristics and oil yields are concerned. An additional series of refinings was made varying the general refining procedure as outlined at the beginning of this report, but the results obtained from this series was so conflicting that it was not thought the results would be of any significance; hence, they will not be included in this report. This work was performed by Mr. Max Kantor of our Technical Department."

TABLE V

Hydraulic Oils Refined by Hydraulic and Expeller Methods
(Results from E. G. Freyer)

Oil	Source	Break	F.F.A.	Refining Loss			
				Max Lye		⅔ Max. Lye	
				Hydraulic 20° Bé	Expeller 12° Bé	Hydraulic 20° Bé	Expeller 12° Bé
1	Edgewater	.41	.65	7.0	5.0	5.2	4.6
2	Edgewater	....	.70	....	....	5.3	4.4
1	Sotex	.81	1.3	9.1	8.9	8.5	7.8
2	Sotex	.97	1.5	10.1	9.0	10.4	9.9
3	Sotex	.89	1.5	9.9	11.8	8.7	10.0
4	Sotex	.89	2.0	15.3	15.6	15.5	15.3
5	Sotex	1.07	1.4	10.4	11.3	10.2	10.7
AO-20	Lever 1	.30	.7	8.1	6.5	6.1	5.4
AO-21	Lever 2	.31	.5	8.1	6.2	6.4	5.4
AO-24	Lever 3	.48	.68	8.3	7.4	6.3	5.5

Dr. Freyer's work may be summarized as follows:

"At the meeting of this Committee on April 30, 1943, it was pointed out that the present official method for hydraulic oil was established from tests on only a few samples. As table V shows, lower losses using the official expeller method were usually obtained on the ten oils tested this year and in only one case, Sotex 3, is loss by the hydraulic method appreciably lower than by the expeller method (8.7 against 10.0). Re-examination of the original data as published by this Committee in OIL & SOAP **14**, 173 (1937) shows that the losses using 20° Be′ lye were only .3% lower than when 12° lye was used and, too, the lower lye gave better bleach colors. On three of the oils in table V (AO-20, AO-21, and AO-24) the bleach colors by the expeller refining showed on the average .3 red lower than when the hydraulic refining was used. "Therefore, in view of these results and for the sake of simplicity, it is recommended that the present procedure designated for expeller oil be designated for expeller and hydraulic oils. It is also recommended that additional comparisons be made on the next crop's oil, especially if appreciable quantities of hydraulic oil are produced. An alternate course would be to continue both methods, designating the present expeller method as 'for expeller and hydraulic oils,' and designating the present hydraulic method as 'an alternate method for hydraulic oil'; then we might suggest to the trade associations using our methods that sellers be permitted to specify which method shall be used on their particular hydraulic oils, inasmuch as it is understood that some leeway of this sort is allowed in the case of slow-breaking oils." Mr. John Thoede of the South Texas Cotton Oil Company furnished data on five of these oils and Dr. R. T. Milner of the Northern Regional Laboratory data on three oils.

The chairman summarized the discussion as follows:

"The extensive investigation conducted by the Northern Regional Laboratory on the centrifuge modification of the official and tentative refining methods for soybean oil has indicated:

1. The centrifugal method of foots separation appears to be somewhat quicker and more certain than the settling method.
2. The proposed change in caustic strength of the lyes used, together with centrifugal separation of the foots, probably would overcome the difficulties encountered with watery foots, particularly in the case of extracted oil.
3. The losses obtained by the refining method are generally similar in magnitude to the lowest loss obtained by the present A. O. C. S. methods; but on the average, the bleach colors are higher.

"There are several disadvantages to the centrifuge method which must be considered:

1. Additional equipment will be needed over the present methods. It is possible that there would be need for the development of a new type of centrifuge with a greater number of cups.
2. There will be difficulties involved in synchronizing the centrifugal method with procedures followed with other oils."

An opinion on the suitability of these methods was asked for from all present, and the replies were as follows:

MR. SORENSON: "The A. O. C. S. expeller method is satisfactory, there is some question of the A. O. C. S. method as applied to hydraulic oil and the tentative method for extracted oil is not satisfactory. The centrifugal method shows promise in this case."

MR. SANDERS: "The A. O. C. S. methods will work on the great majority of oils including all of the 25 samples covering all types discussed at this meeting by Dr. Milner. Most refinery experience confirms these results. The present A. O. C. S. methods therefore would be reasonably good starting tools. Present knowledge plus actual experience in the trade would supply needed modifications."

MR. KIESS: "The expeller method of the A. O. C. S. gave reproducible results. It also works quite well on hydraulic oils. The method for extracted oils is reproducible on, say, 85 percent of the oils, and the foots are watery and sloppy at times."

MR. MCGEE: "On damaged oils where trouble is encountered, we do not finish the loss determination, but complete the bleach color test. Too much alkali is being used on some of these poor oils."

MR. KRUSE: (Mr. Kruse did not wish to add to the discussion presented to this point.)

MR. MOORE: "Do not believe that the centrifuge work should be dropped and think the Northern Regional Laboratory should continue their work on the centrifugal method for another year, if necessary."

MR. TAYLOR: "The time factor for the A. O. C. S. method is the factor to be considered, it being a 24-hour job. Perhaps the centrifugal method would require more laboratory time but not as much over-all time."

MR. ORY: "A 10-gram, or some such weight, sample of oil might offer possibilities for a micro method approaching a more quantitative aspect."

MR. KISHLAR: "In my opinion the centrifugal method should not be dropped. The probabilities of the photoelectric colorimeter being employed for color measurement and a micro-refining method seem to offer some hope for the future."

MR. DURKEE: "No action is required if the A. O. C. S. methods are to be retained. The methods for extracted and clarified extracted oils should continue to be accepted as tentative. The time factor on the A. O. C. S. method is discouraging. The centrifugal method work should be continued. The A. O. C. S. calculation of alkali needed requires further study."

MR. SPANNUTH: "The A. O. C. S. methods offer the only course open for the present, but the work on the centrifugal method should be continued. Color grading should also be given consideration."

MR. JAMES: "Which firms have centrifuges available?" (A. E. Staley and Company, Procter and Gamble Company, Lever Brothers Company, Armour and Company, Wilson and Company, and Swift and Company have centrifuges available. Spencer Kellogg Company and Archer-Daniels-Midland Company are attempting to complete their equipment so that it can be used for the centrifuge method.) "The A. O. C. S. method gives too light a color on the laboratory refining compared to plant color. On poor oils the difference amounts to as much as a 1-unit spread in red color."

MR. HOULE: "In our experience over a long period, there have been few cases where the refinings on expeller oils and extracted oils failed to give losses. Study of both methods should be continued. It seems that toward the first of the year the two-thirds maximum gives the best refining, whereas during the middle of the year the maximum alkali gives the best results."

A Sub-Committee of S. O. Sorenson (chairman), E. M. James, E. Freyer, N. F. Kruse, A. Kiess, and J. H. Sanders, appointed by Mr. Mitchell, presented the following two resolutions:

(1) WHEREAS, The intensive investigation conducted by the Northern Regional Research Laboratory on the centrifuge modification of the official and tentative refining methods has indicated that:

1. The centrifuge method always gives compact foots on all types of soybean oil with reproducible results when using the recommended conditions and is decidedly quicker.
2. The proposed change in caustic lye strength used, together with centrifugal separation of the foots, probably would overcome the difficulties encountered with sloppy foots, particularly in the case of extracted oil.
3. The centrifugal method losses, in general, are of similar magnitude to the lowest loss obtained by the present A. O. C. S. cup methods, being sometimes lower and sometimes higher depending mainly upon oil type.

4. The centrifugal method gives higher refined bleached colors with the recommended 30° Be′ lye, but with weaker lyes the refined bleached colors are closer to those obtained by the cup methods.

And Whereas, There are several disadvantages to the centrifugal method which must be considered; namely,

1. Additional equipment will be needed over the present, facilities for the A. O. C. S. cup method.
2. There will probably be need for changes in equipment design necessary to place the centrifugal method on a completely practical basis.
3. Under present conditions, the securing of new equipment is uncertain.
4. There will be difficulties involved in synchronizing the centrifugal method with procedures followed on other oils.

And Whereas, The ultimate centrifugal refining loss test should correlate with commercial practice,

Be It Resolved, That the A. O. C. S. Refining Committee suggest that at some future date the work on the development of the centrifugal refining method be continued at the Northern Regional Research Laboratory under the direction of Dr. R. T. Milner.

(2) Whereas, The present A. O. C. S. cup methods for hydraulic and extracted soybean oils have produced unsatisfactory results in many cases.

And Whereas, The trading experience of the industry for many oils has been based upon the cup refining methods of the A. O. C. S.,

Be It Resolved, That the Refining Committee continue their studies on modifications of the present A. O. C. S. cup methods which are now under way in Sub-Committees with the Northern Regional Research Laboratory collaborating in this work at the present time ,and the new recommended procedures be subjected to collaborative work for possible adoption at the earliest time, if possible by September 1, 1943. Such methods to be tentative and subject to further modifications as conditions warrant.

Mr. Taylor moved that these resolutions be adopted; seconded by Mr. Durkee. They were carried.

Mr. Kruse offered the following resolution:

The present method for expeller oil is satisfactory and gives reproducible results. Modifications of the present methods for extracted and hydraulic oils would improve these from the standpoint of reproducibility and such modifications will be presented at the earliest possible time.

This was seconded by Mr. Sorenson, and carried.

Another meeting of this Committee will be called as soon as additional data are available for consideration.

BOOK REVIEW

"Practical Emulsions," by H. Bennett, Editor-in-Chief, *The Chemical Formulary*; Technical Director, Glyco Products Company, Inc. Chemical Publishing Company, Inc., Brooklyn, New York, 1943; 462 pp. illustrated; 15 x 22 cm. Price $5.00.

This is a highly practical book which makes no attempt to delve into the theoretical aspects of emulsions. It concisely sets forth much helpful information on the production and preservation of good emulsions.

The book is divided into two parts. The first is concerned with emulsifying agents, type of emulsions, methods, formulation, equipment, stability of emulsion, and a discussion of several of the more important technical emulsions such as asphalt, cosmetic, detergent, lubricating, latex, food, leather and paint, polish and wax emulsions. Also included in this section is a chapter on dispersing and wetting agents. The section finally contains a list of some 600 emulsifying agents and 1,000 emulsions with references to their composition or source, and a list of demulsifying and defoaming agents.

The second part of the book gives formulas and methods for making many types of emulsions. Among them are agricultural sprays, cutting and soluble oils, cleaners and soaps, as well as emulsions in the fields of asphalts, cosmetics, drugs, foods, lacquers, leather, lubricants, medicines, paints, paper, polishes, resins, and textile.

H. C. Black,

Abstracts

Oils and Fats

Edited by
M. M. PISKUR and SARAH HICKS

Testing edible oils. Official methods of analysis in Portugal. *Oil & Colour Trades J. 104,* 130 (1943).

New methods for determination of fat in foods. A. Schloemer and K. Rauch. *Z. Untersuch. Lebensm. 83,* 289-305 (1942). Extn. of fat from milk by the Grossfeld method, in which the sample is hydrolyzed with HCl in presence of $CHCl:CCl_2$ gave results on fat content and the butyric acid no. of the extd. fat that agreed with those using the Gerber fat detn. method. Replacing the $CHCl:CCl_2$ with CCl_4 at first gave lower results, but on increasing the time of heating for hydrolysis and adjusting the ratio of reagents results were obtained which agreed with those of the Gerber and the Roese-Gottleib methods. The modified method for milk is as follows: 10 g. milk, 10 cc. CCl_4, 15 cc. concd. HCl and some pumice stone are refluxed 20 mins. After cooling add 10 cc. 96% alc. and 40 cc. benzine, shake vigorously for 15 secs. and allow to stand 15 mins. The wt. of fat is detd. in a 25 cc. aliquot from the fat soln, phase and the results are obtained with the use of Table 4, p. 336 of "Anleitung zur Untersuch. Lebensm." The new method produces less carmelization of lactose than the method using $CHCl:CCl_2$ or the Schmid-Bondzynski-Ratzlaff method. With dried milks 2 g. of sample, 10 cc. HCl, 10 cc. CCl_4 and 10 cc. alc. should be used as reagents and the addn. of the alc. should be followed by a 5 min. addnl. refluxing. In work on cheese, the Grossfeld Hoth method was as reliable and simpler than any modification of the CCl_4 method. Procedures for the use of the new method on dried eggs and bakery products were also prepd.

New method for determining fat in cheese. J. Grossfeld and A. Zeisset. *Z. Untersuch Lebensm. 84,* 193-201 (1942), Bring 5 g. sample, 6x6-cm. "Zellglass," some pumice stone and 10 cc. 25% HCl to a boil in a reflex app. After 10 min. add 20 cc. CCl_4. Boil 10 min., cool to 20°, add 30 cc. benzine (b.p. 60-70°), shake ½ min. and allow to stand over night. Amt. of fat in a 25 cc. aliquot is detd. and fat content is read from Table 4 in "Anleitung zur Untersuch. Lebensm.," p. 384. The method checks well with the international method and requires less reagents.