Report of the Refining Committee—1941-1942

The Regional Soybean Laboratory has again carried the major share of the experimental work of the Refining Committee. Dr. D. H. Wheeler and his group carried on an extensive series of tests with a centrifuge which resulted in a suggested method using this means of separating the foots. The planning of the two cooperative studies made during the year and the tabulating and recording of results were also important contributions of this group. I am certain that the Society and the Refining Committee are appreciative of the splendid cooperation given by the Regional Soybean Laboratory in the continuing effort to find a suitable method of refining certain types of soybean oil.

The Committee, in its 1940-41 report, recommended that the silicate-acid refining method be given further tests by the group. Cooperative samples Nos. 1 and 2 were tested by the members, using both the Tentative A.O.C.S. method and the proposed silicate method. Contrary to what had been expected from previous tests, the proposed silicate method completely failed to give results on one of the two samples used. This serious failure would seem to eliminate the method from further consideration. The results are attached. (See Tables I and II.)

In view of the failure of the silicate method, attention was turned to the use of a centrifuge to give compact foots. Preliminary qualitative tests made by the Swift Research Laboratories and reported in last year's report indicated that this method had some promise.

TABLE I

Soybean Oil Refining Test—Cooperative Sample No. 1—A.O.C.S. Refining Committee—1941-42

	Tentative A.O.C.S. Method						Regional Soybean Laboratory Method			
Laboratory	F.F.A. Found	Max. 14° Bé. Lye	Grams Lye	Refining Loss	Color	Remarks	c.c. of 30° Bé. Used	Refining Loss	Color	Remarks
A. E. Staley Mfg. Company	0.58	2/3	22.8	5.88	9.8		6.9			Foots from Sample 1 grainy but not firm enough to recover oil.
			22.8	5.90	9.9					
		7/8	30.0	7.17	9.8					
			30.0	7.27						
Archer-Daniels-Midland Company	0.61	2/3	22.9	5.22			6.77	15.2		Loose granular.
		7/8	29.9	6.51				14.3		
Armour and Company	0.64	2/3	22.8	5.2	10.5	Semi-solid foots firm but would not adhere to bottom of cup.	6.75	7.3	11.7	One cup sloppy and one cup fair.
			22.8	5.2						
		7/8	30.0	6.7	10.7	One cup as above, the other cup had firm foots.				
			30.0	6.6						
Barrow-Agee Laboratories	0.64	2/3	23.0	5.5		Very satisfactory results	6.9	6.96		Soapstock on No. 1 granular and held entrained oil; difficult to separate.
			23.0	5.5				6.36		
		7/8	30.0	6.4						
			30.0	6.5						
Central Soya Company, Inc.	0.58	2/3	22.9	5.35		Foots in all cases fairly good.	7.15			Foots from No. 1 too sloppy to effect separation of oil.
		7/8	30.06	5.8						
Durkee Famous Foods	0.62	2/3	23.1	5.85		Foots firm—all oil recovered in first decantation.	7.0	16.52		Foots were too soft and grainy. Impossible to recover all the oil by remelting, chilling, and decantation.
			23.1	5.70				16.50		
		7/8	30.4	6.85						
			30.4	6.72						
H. E. Moore	0.6	2/3	22.77	5.3			6.84			Foots light and fluffy. Wouldn't settle out.
		7/8	29.88	6.7						
Lever Brothers Company	0.56	2/3	22.5	5.35	10.3	Globular, large-grained, oily looking, firm.	6.7	23.3	10.1	Fluffy suspension—very unsatisfactory.
		7/8	22.5	5.35	10.3	Satisfactory.		23.1	10.2	
			30.0	6.6	10.0	Same as above.				
			30.0	6.25	10.0					
Ralston-Purina Company	0.59	2/3	22.8	5.93			6.8			Foots too soft, poured with oil after chilling for 1 hr. at 10-15° C.
		7/8	29.9	7.68						
Regional Soybean Ind. Products Laboratory	0.55	2/3	22.80	5.7			6.9			Foots granular but too soft.
			22.80	5.6						
		7/8	29.90	6.2						
			29.90	....		Slippery foots				
Spencer-Kellogg and Sons, Inc.	0.60	2/3	23.0	6.0			6.9			Foots too soft.
			23.0	5.85						
		7/8	30.2	7.50						
			30.2	7.20						
Swift & Company	0.57	2/3	23.0	5.42		Foots fine grained, held together well.	6.8	5.28		Oil sample No. 1 only very slight break. Small grained foots which did not solidify.
			23.0	5.20		No unabsorbed water.		4.71		
		7/8	30.0	6.35		Same as above.		4.69		
			30.0	6.33						
The Fort Worth Laboratories	0.58	2/3	23.0	5.2	10.2		7.0	6.7		Poor soap stock. Required 7 remelts to determine loss.
			23.0	5.6	10.2			6.8		
		7/8	30.0	6.7						
			30.0	6.5						
Wilson & Company, Chicago, Ill.	0.59	2/3	24.1	6.3			7.0	8.6		
		7/8	29.6	6.2						
Wilson & Company, Oklahoma City, Okla.	0.60	2/3	23.0	4.8			6.96	4.7		Soft, wet, difficult to separate. Did not break well, foots loose.
			23.0	4.8				4.5		
		7/8	30.0	5.3						
			30.0	5.6						
T. C. Law, Atlanta, Ga.	0.70	2/3	23.5	8.3	11.0		6.8	....	12	Could not get satisfactory loss.
		7/8	30.5	6.1	9.8					
Procter & Gamble	0.6	2/3	22.8	5.7	10.0		7.0	9.2	10.5	Very sloppy foots.
				5.7	10.1			7.3	11.1	
		7/8	29.9	7.0	9.9					
				7.0	9.8					

A method was developed at the Soybean Laboratory which consisted of refining with 30°Bé. sodium hydroxide in an amount equal to five times the theoretical amount necessary to neutralize the free acid in the oil. A number of trials showed that a high Baumé lye was necessary to avoid water in the oil, to an even greater extent than when gravity settling was used.

The refining was done directly in the bronze centrifuge trunion cup in the regular refining apparatus, modified to hold the trunion cup and with the paddle blades shortened to clear the cup. The method was tested at the Soybean Laboratory on six samples of solvent-extracted oil. The oils were also refined by the Tentative A.O.C.S. method, with the results shown. (See Table III.)

In all cases, the foots obtained by the centrifuge method were very hard and compact, and no water in oil was observed, while three of the six samples gave trouble by the regular method. The oil decanted from the centrifuge refining was very clear in all cases.

A poll of the Refining Committee revealed that a total of six members had the necessary equipment and would be willing to try cooperative tests by the proposed centrifuge method. Arrangements were made to send cooperative samples Nos. 3 and 4 of solvent-extracted oil to these members for an evaluation of the method and comparison with the tentative A.O.C.S. method. The description of the proposed method as sent to those participating is as follows:

Proposed Centrifugal Refining Test Method

I. Apparatus

1. *Refining Apparatus:* As for the usual refining loss test (p. 12, A.O.C.S. Methods), except that the paddles are cut off so that they clear the walls of the centrifuge trunion cup by ¼ inch. Provision shall be made to hold the trunion cups in place in the refining apparatus. (A wooden collar has been used successfully as a temporary means.)
2. *Scales:* Sufficient capacity to weigh a trunion cup with 200 grams of oil. Sensitivity, 1/10 gram.
3. *Centrifuge:* Any type capable of spinning cups at about 1900 x gravity (e.g. International size 1, type

TABLE II

Soybean Oil Refining Test—Cooperative Sample No. 2—A.O.C.S. Refining Committee—1941-42

	Tentative A.O.C.S. Method						Regional Soybean Laboratory Method			
Laboratory	F.F.A. Found	Max. 14°Bé. Lye	Grams Lye	Refining Loss	Color	Remarks	c.c. of 30° Bé. Used	Refining Loss	Color	Remarks
A. E. Staley Mfg. Company	0.52	2/3 7/8	22.5 22.5 29.9 29.9	4.14 4.26 5.54 5.89	8.2 8.2 8.1 8.1		6.5	2.76 2.76	9.0 9.1	
Archer-Daniels-Midland Company	0.57	2/3 7/8	22.6 29.7	4.0 4.7			6.41	3.48 3.44		Firm granular.
Armour and Company	0.58	2/3 7/8	22.4 22.4 29.5 29.5	3.4 3.5 4.5 4.7	8.0 8.0	Foots in all cases were solid and adhered well to bottom of cup. One remelt.	6.40	5.4 5.0	9.2	Both cups good.
Barrow-Agee Laboratories	0.60	2/3 7/8	22.5 22.5 29.5 29.5	4.1 4.7 4.9 5.1		Very satisfactory results.	6.5	4.14 3.24		
Central Soya Company, Inc.	0.55	2/3 7/8	22.54 29.60	4.25 5.1		Foots in all cases were fairly good.	6.77	3.18 3.15		Large, firm foots.
Durkee Famous Foods	0.62	2/3 7/8	22.9 22.9 30.1 30.1	4.90 4.84 5.37 5.12			6.6	4.64 4.0		Coarse, firm, dry foots.
H. E. Moore	0.55	2/3 7/8	22.56 29.61	2.2 2.8			6.48	2.9		Coarse, hard-settled, good.
Lever Brothers Company	0.54	2/3 7/8	22.5 22.5 30.0 30.0	4.9 4.75 6.3 6.15	8.1 8.1 8.4 8.4	Medium close grained. Firm, satisfactory. Medium close grained. Firm, satisfactory.	6.4	4.30 4.25		Globular, medium grain, firm, satisfactory.
Ralston-Purina Company	0.53	2/3 7/8	22.6 29.65	4.13 5.14			6.5	3.10		Good firm foots.
Regional Soybean Ind. Products Laboratory	0.54	2/3 7/8	22.60 22.60 29.70 29.70	4.1 3.9 5.0 5.1			6.5	4.1 4.4		Foots granular and firm.
Spencer-Kellogg and Sons, Inc.	0.55	2/3 7/8	23.0 23.0 30.2 30.2	4.10 4.05 5.25 5.10			6.5	3.90 4.00		Foots hard.
Swift & Company	0.54	2/3 7/8	22.5 22.5 29.5 29.5	4.56 4.39 5.87 5.80		Well grained, solid foots. No unabsorbed water. Oil decanted off easily. Same as above.	6.5	3.63 3.67		Firm, large-grained foots. Oil decanted off easily.
The Fort Worth Laboratories	0.53	2/3 7/8	22.5 22.5 30.0 30.0	4.1 4.4 5.5 5.4			6.6	2.7 2.6		Very good soap stock. Required only two remelts.
Wilson & Company, Chicago, Ill.	0.57	2/3 7/8	22.5 29.2	2.8 3.0			6.7	3.2 3.2		Firm and granular.
Wilson & Company, Oklahoma City, Okla.	0.56	2/3 7/8	22.5 22.5 29.5 29.5	3.4 3.4 3.7 3.7			6.59	2.6 2.6		Good foots.
T. C. Law, Atlanta, Ga.	0.60	2/3 7/8	21.5 28.5	6.0 4.9	11.2 9.8		6.5	3.1	9.7	Firm foots.
Procter & Gamble	0.6	2/3 7/8	22.6 29.65	4.6 4.6 5.6 5.6	8.0 7.9 7.8 7.7		6.6	3.0 4.4	9.1 9.0	Firm but granular foots.

SB, at 2800 r.p.m., radius 20 cm.). If the centrifuge available does not produce this force, increase centrifuging time proportionately, but record the time and conditions of centrifuging. (The literature accompanying a centrifuge usually gives data on speeds and forces available with a given head.)

4. *Trunion Cups:* 250-c.c. capacity to fit the centrifuge used. The trunion cups are used as refining cups in this test.

II. Procedure

1. Weigh trunion cup.
2. Add 200 grams of oil to cup.
3. Add 30 Bé. sodium hydroxide equivalent to 5 times the theoretical amount necessary to neutralize free acidity, by direct weighing into the cup and oil on the balance. Weight of dry NaOH as 30 Bé. for 5 times theoretical for 200 gms. oil = 1.42 x per cent acid as oleic.
4. Place cup in refining apparatus bath at 20-24°C. and stir at 250 r.p.m. ± 10 for 60 minutes.
5. Immediately transfer to bath at 63-67° and stir for 30 minutes at 70 ± 5 r.p.m. Then raise paddles and allow to stand 15 minutes in 65° bath.
6. Remove from bath, place cups in centrifuge and centrifuge at approximately 1900 x gravity for 30 minutes.
7. Weigh cup and contents to determine evaporation loss.
8. Decant oil into a tared beaker and drain 30 minutes.
9. Weigh cup and refining foots.
10. Weigh refined oil in beaker.
11. Remelt cup contents at 75° ± 2°C. for 30 minutes without stirring.
12. Centrifuge cup 30 minutes.
13. Drain cup 30 minutes into separate tared beaker.
14. Repeat remelts (11, 12, 13) until 0.5 gram or less is obtained on remelt.
15. Calculate results by two methods and report average of the two methods.
    (a) Weight of crude oil minus weight of refined oil gives refining loss, to be reported as per cent of crude oil. Include the last remelt of 0.5 gram or less in weight of refined oil.
    (b) Weight of soap stock plus evaporation loss, minus weight of sodium hydroxide solution used gives refining loss, to be reported as per cent of crude oil.
16. Report
    (a) Grams of 30 Bé. NaOH used.
    (b) Number of remelts necessary to obtain 0.5 gram or less.
    (c) Refining loss.
    (d) Color of oil (Lovibond).

The results of the tests on cooperative samples Nos. 3 and 4 were as shown in the attached tables. (See Tables IV and V).

TABLE III

Comparison of A.O.C.S. Refining Method and Centrifuge Method (Run at Regional Laboratory)

		Regular Method					Centrifuge Method			
Sample	F.F.A.	Gm. 14°Bé. Lye	Max.	Refining Loss	Remelts	Condition of Foots	Gm. 30°Bé. Lye 5 x Theory	Refining Loss	Remelts (to less than 0.5 gm.)	Color
288	0.28	27.5 21.0	7/8 2/3	4.84 4.18	1 1	O.K. Compact but slippery	1.7	2.30	2	10.23
289	0.47	29.2 22.3	7/8 2/3	3.93 3.86	1 1	Water in oil O.K.	2.9 2.9	3.45 3.83	1 1	9.62 9.72
291	0.27	27.4 20.9	7/8 2/3	7.67 6.37	2 3	Soft Soft	1.6 1.6	2.95 2.70	2 2	11.22 10.91
292	0.26	27.3 20.8	7/8 2/3	7.49 1.75	3 3	Soft Soft	1.6 1.6	3.05 3.03	1 1	10.91 11.12
293	0.31	27.8 20.9	7/8 2/3	4.49 3.92	1 1	O.K. O.K.	1.9 1.9	3.18 3.15	1 2	10.58 10.91
294	0.90	33.0 25.2	7/8 2/3	4.22 4.45	1 1	O.K. O.K.	5.5 5.5	6.50 6.45	1 1	11.81 11.81

TABLE IV

Soybean Oil Refining Test—Cooperative Sample No. 3—A.O.C.S. Refining Committee—1941-42

Laboratory	F.F.A. (%)	Tentative A.O.C.S. Method			Centrifuge Method				
		Max. 14°Bé.	Ref. Loss %	Lov. Red	Gm. 30°Bé.	Ref. Loss %	Remelts	Lov. Red	Centrifuge
Soybean Laboratory	0.53	7/8 = 29.1 gm. 2/3 = 22.2 gm.	5.52 4.28	9.1 10.1	3.2 3.2	3.55 3.75	1 1	9.9 9.2	As directed
A. E. Staley Mfg. Company	0.39	7/8 = 27.9 gm. 2/3 = 21.3 gm.	5.29 4.24	8.8 9.0	2.40 2.40	2.88 2.85	2 2	9.0	2200 R.P.M. 50 min.
Swift & Company	0.5	7/8 = 29.5 gm. 2/3 = 22.5 gm.	5.5 4.4	8.5 8.7	2.96 2.96	3.5 3.4	1 1	8.7 8.7	2200 R.P.M. at 20 cm. 50 min.
Barrow-Agee Laboratories	0.4	7/8 2/3	5.3 4.6	....	2.4 2.4	3.3 3.3	1 1	7.8 8.3	No statement
Proctor & Gamble Company	0.45	2/3 = 22.0 gm.	4.4	8.3	2.7 2.7	4.8 6.3	0 0	8.4 8.3	1100 x gravity for 50 min.
Lever Bros.	....	7/8 = 29.5 gm. 2/3 = 22.5 gm.	5.7 4.4	10.0 9.5	3.05	5.2 avg. of dup.	1	9.0	No statement

The samples were further analyzed by the A. E. Staley Company for the following values in addition to those reported in connection with the test:

	No. 3	No. 4
Gardner break	0.04	0.17
Appearance	Clear	Clear
Moisture and volatile	0.3	0.1
Color	25 Yellow 4.6 Red	25 Yellow 4.9 Red

Inspection of the results reveals that neither of the samples tested caused the type of difficulty often encountered with the A.O.C.S. method with solvent extracted oils. For this reason they cannot be considered the most stringent test of the method as far as overcoming the difficulties of soft or sloppy foots and water in oil. On sample No. 3, only three out of six laboratories checked well with the centrifugal methods, while all six agreed when the A.O.C.S. method was used. On sample No. 4, four out of six agreed well by the centrifuge method, and with the A.O.C.S. method, four out of six agreed well. One of the laboratories which failed to check on both samples by the centrifuge method stated that their cup was so deep in relation to the diameter that stirring might not have been effective. On sample No. 3 the centrifuge method gave oils of about the same color as the A.O.C.S. method, while on sample No. 4 the centrifuge method gave oils consistently darker than the A.O.C.S. method.

These results certainly do not constitute any overwhelming argument in favor of the centrifuge method. However, consideration should be given to the fact that the A.O.C.S. method is familiar and more standardized, while the proposed method was being carried out for the first time, with improvised equipment, and unavoidable variations in cups and centrifuging. In view of these considerations, and in view of the results obtained at the Soybean Laboratory on certain samples which gave trouble by the A.O.C.S. method, further cooperative work would seem desirable, with efforts being made to standardize the centrifuge method, and to further compare it with the A.O.C.S. method.

Recommendations for Next Year's Refining Committee Activities

I. Consideration should be given to standardizing the centrifuge method with regard to stirring, cup dimensions, and centrifuging force and time. The amount of caustic to be used might be given consideration, using a definite excess over theory, instead of a definite number of times theory, as proposed.

II. Further cooperative tests should be made on solvent extracted soybean oil, using both the centrifuge method and the tentative A.O.C.S. method on enough samples to evaluate the former method. If the method should prove satisfactory on solvent-extracted oils, it should be tested on expeller and hydraulic oils.

III. Continued close cooperation by the Regional Laboratory is essential to the solution of the soybean refining problem. It is hoped that this group will again be in a position to carry the burden of the experimental work in the next year's program.

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TABLE V

Soybean Oil Refining Test—Cooperative Sample No. 4—A.O.C.S. Refining Committee—1941-42

Laboratory	F.F.A. (%)	Tentative A.O.C.S. Method			Centrifuge Method				
		Max. 14°Bé.	Ref. Loss %	Lov. Red	Gm. 30°Bé.	Ref. Loss %	Remelts	Lov. Red	Centrifuge
Soybean Laboratory	0.63	7/8 = 30.0 gm. 2/3 = 22.8 gm.	2.58 2.65	8.6 8.7	3.8 3.8	4.60 4.60	1 1	9.7 10.2	As directed
A. E. Staley Mfg. Company	0.63	7/8 = 30.0 gm. 2/3 = 22.9 gm.	2.92 2.90	8.2 8.2	3.8 3.8	4.73 4.75	2 2	9.0	2200 R.P.M. 50 min.
Swift & Company	0.7	7/8 = 31.0 gm. 2/3 = 23.5 gm.	3.0 2.9	7.4 7.7	4.13 4.13	4.75 4.85	1 1	8.7 8.7	2200 R.P.M. at 20 cm. 50 min.
Barrow-Agee Laboratories	0.6	7/8 2/3	6.0 4.5	....	3.6 3.6	4.6 4.5	1 1	8.5 8.5	No statement
Procter & Gamble Company	0.6	2/3 = 23.0 gm.	3.1	7.8	3.6 3.6	5.0 5.3	0 0	8.2 8.3	1100 x gravity for 50 min.
Lever Brothers	....	7/8 = 31.0 gm. 2/3 = 23.5 gm.	3.0 3.2	8.7 8.7	4.25 4.25	5.2 avg. of dup.	1	10.0	No statement