# **Report of the Refining Committee 1940-41**

THE Refining Committee, in its 1939-40 report, recommended that the tentative method for extracted soybean oil be given further study and that suitable combinations of alkalies, as well as method of application, be given consideration. This has been done in this year's program of the Committee.

Another 1939-40 recommendation was that the Soybean Regional Laboratory take an active part in the work, collaborating with the Refining Committee in the investigational program necessary for the development of methods to be used in cooperative study. This has also been done and it will be noted in this report that practically all of the investigational work was carried out at the Regional Laboratory. The mass of data presented, covering the extensive tests made by D. H. Wheeler and P. Krauczunas, of the Soybean Laboratory, indicate how well they carried out their portion of the 1939-40 recommendations of the Committee. The Refining Committee and the Society owe these men and the Regional Laboratory a debt of gratitude for their efforts in the difficult task of finding a method suitable for Extracted Soybean Oil.

A meeting of the Refining Committee was held at the U. S. Regional Soybean Industrial Products Laboratory on April 24, 1940. Complete minutes covering this meeting, dated April 25, 1940, were distributed at the 1940 Spring Meeting of the Society. For the purpose of the record, however, the suggested experimental procedure agreed upon at this meeting is given again in this report since the work at the Regional Laboratory covered most of the suggestions made. These suggestions were as follows:

"1. Some work shall be done in refining with straight alkali.

"2. Concerning the strength of alkali to use, it was suggested by Kiess that some tests should be made with  $16^{\circ}$  and  $18^{\circ}$ , as well as with the usual  $12^{\circ}$  and  $14^{\circ}$  Baumé; and also the ratio of excess should be higher for higher free fatty acid.

"3. It was agreed that  $\frac{1}{2}$ ,  $\frac{2}{3}$ , and  $\frac{3}{4}$  of the maximum amount of lye should be used.

"4. The time and temperature of agitation in the cold should be 10° C. or lower, for one hour. (This suggestion was subsequently withdrawn.)

"5. Hot agitation time should start at 12 minutes, and the temperature should be 60° C., plus or minus 2°. Amended to  $65^{\circ} \pm 2^{\circ}$  C.

"6. This concerns the settling period. The Committee believes that the time should be overnight. At the end of the refining period, it was agreed to take it out and allow it to come to room temperature, record the room temperature, then rechill to  $10^{\circ}$  C. or lower for 30 minutes the next morning before pouring off the oil.

\* "7. Remelting and draining. The evaporation losses were discussed, and it was felt that the wording of the method should be clarified. It was agreed to weigh the foots immediately after pouring oil, then make the evaporation loss correction.

then make the evaporation loss correction. "8. A combined alkali. The Chairman reported that he had used 5% sodium metasilicate powder in making up the mixture. The Committee felt that there should be an investigation of the action of various combinations in refining, and that someone should write the Philadelphia Quartz Company for their past experience. "9. It was generally agreed to use the old standard bleach test and adopt 40 yellow for collaborative work, but to accept the colors only from those who have standardized glasses. The readings of others shall be recorded with a mark to indicate that their results cannot be averaged. It was agreed to run bleaching tests on refined oil in case a promising refining method is found in order to determine if the color has been 'set.'

"10. When collaborative samples are received at the Laboratory, the top of the can should be cut open to be sure that all settled out material is incorporated with the main part of the sample and that the sample should be warmed to 50° C. to help get any settlings in solution. "11. The Regional Laboratory chemists will deter-

"11. The Regional Laboratory chemists will determine the free fatty acid on collaborative samples. Individual laboratories will run the determinations, but will use the value set for the samples; in this way, disturbing variations will be eliminated. The lye will be specified. The fatty acid determinations shall be reported by individual laboratories to 0.01% and should use tenth-normal NaOH for titration."

The next section of this report was prepared by Wheeler and Krauczunas, of the Soybean Laboratory.

The Regional Soybean Laboratory first carried out a refining loss program on solvent extracted oils as outlined by the Committee. The strength of lye was varied, using 12°, 14°, 16°, and 18° Baumé and making two tests on each oil, using  $\frac{1}{2}$  and  $\frac{3}{4}$  of the maximum of sodium hydroxide as calculated from the formula F.F.A.

----+ .54. The tabulation of results is attached. 5.2

(Series I). These tables show that, whereas good results can be obtained in some cases, it is not true with many samples.

Most of the trouble is with extracted clarified oils. The lye frequently separates from the foots, giving water in the oil. Realizing this, Mr. Kiess of Armour and Company strongly urged the trial of smaller amounts of stronger caustic. Following this suggestion, tests were made using  $20^{\circ}$ ,  $22^{\circ}$ , and  $30^{\circ}$  Baumé lye. The improvement obtained with the clarified oils indicated that this step was in the right direction, but there was still trouble with soft foots in many cases.

Pursuant to further suggestions of the Committee, the Philadelphia Quartz Company was consulted and a series of experiments run, using silicates (Series II). In general, the use of silicate gave very good results with clarified oils, eliminating the difficulties of water in the drained oil. However, soft foots were still encountered in some samples.

Having exhausted these possibilities, a miscellaneous lot of reagents were tried, as tabulated in Series III. Nothing promising was uncovered.

It seemed that, whereas a satisfactory method could be worked out for a given oil, the same procedure would not be especially desirable for other oils. This work, as well as the results of cooperative samples of previous years, emphasized the fact that to get concordant results a method is required which will give firm foots that retain the lye. Retention of water is accomplished by using smaller amounts of stronger caustic, whereas

some sort of binder is indicated to secure firmness of the foots.

Proceeding on this basis, a method was worked on (Series IV) in which a solution of metasilicate was pipetted in, followed by concentrated HCl and then the calculated amount of high Baume' caustic. This method looked promising and after preliminary work, a trial cooperative run was made on three samples of widely varying oils in the Armour, Soybean, and Swift Laboratories. Whereas good firm foots were obtained, the results reported by the different laboratories were not in good enough agreement.

In an attempt to discover the cause of the discrepancies, the same three analysts made a determination on one of the samples in the presence of each other so that all used exactly the same details (Series V). The attached table gives the results of this run, as well as the figures obtained in the separate laboratories. From this and the chart of variations in procedure, it can be expected that if the directions are explicit in all details and if everybody follows these directions to the letter, results which check reasonably well can be expected, provided the foots are firm enough.

The following method is recommended:

Apparatus

Official refining loss apparatus (page 12, Official and Tentative Methods of A.O.C.S., amended 1938).

Refining cups.

Balance of at least 2-pound capacity and sensitive to 0.1 gm.

Analytical balance.

Clocking device.

- Pipettes: 1 cc.; 10 cc., graduated to 0.1 cc.
- Flask, Ehrlenmeyer, ground glass stoppered (ca 250 cc.).

Beakers, 50 cc. or 100 cc.

Some support for cups (in drainage).

# Reagents

1-2 N. standard acid.

- Stock saturated NaOH of determined percentage. As the formation of carbonate is noted, filter periodically through asbestos. Such filtration does not necessitate restandardization.
- 30° Baume' caustic. To be prepared each week from the above stock. After allowing sufficient time for the freshly prepared solution to come to room temperature, exactly 10.00 cc. are weighed in g.g.s. Ehrlenmeyer on an analytical balance and standardized. There is thus determined the density, in grams per cc., and the exact strength, which must be 23.5 per cent plus or minus 0.4 per cent, by weight, as calculated from the formula,  $\% = N \times cc. \times 4/wt.$  of 10 cc. (N = normality, cc. = cc. of standard acid to titrate 10 cc. to phenolphthalein endpoint).
- 50 percent, by weight, solution of Na<sub>2</sub> SiO<sub>3</sub>5H<sub>2</sub>O prepared the previous evening, using heat to get solution and letting it come to room temperature overnight.

Conc. HC1 (sp.g. 1.19).

#### Procedure

Weigh out 500 gm. oil (properly sampled) into cup and let settle for at least 2 hours, preferably overnight.

While stirring in the refining apparatus at 250 r.p.m. and 20°-24° C., pipette 21/2 cc. of the 50 per cent Na2SiO35H2O solution, following with 1 cc. concentrated HC1. Continue this cold stirring for 10 minutes.

Then, while still stirring, pipette in the 30° Be' NaOH as calculated from the formula: (F.F.A.) (3.55)

(percentage of 30° Be'/100) (density  $\frac{\text{wt. 10 cc.}}{10}$ )

(This is the volume in c.c. of lye for the 500 gm. of oil and represents 5 x theory.

Continue agitation at 250 r.p.m. and 20°-24° C. for 1 hr. Change to 63°-67° C. and agitate at 70 r.p.m. for 15 min.

Let settle at 63°-67° C. for 1 hr.

Chill in bath for 1 hr. at 10°-15° C.

Let stand at room temperature overnight.

Chill in bath for 1 hr. at 10°-15° C.

Weigh the cups and contents to determine loss by evaporation.

Pour off the oil into tared cup or beaker, allowing exactly 30 minutes for drainage.

Weigh the oil; weigh the soap stock.

Remelt : Place in water bath at 75° C. for 30 minutes. Chill in bath at 10°-15° C. for 1 hr.

Drain oil into tared 50 cc. or 100 cc. beaker for exactly 30 minutes.

Weigh the drained remelt oil; if this is more than 1.5 gm., continue the above remelting procedure until the drained oil is less than 1.5 gm.

Calculations, per cent refining loss

Method No. 1:

500 - (decanted oil + total remelt oil)5

Method No. 2:

(Wt of soap stock\* + evap. loss) -

 $(4.6^{**} + \text{calctd. wt. of } 30^{\circ} \text{ Bé} + \text{total remelt oil})$ 

5

\* Weight after *first* drainage.

\*\* Weight of the 2.5 cc. silicate + 1 cc. HC1.

In the following tables, covering the work done by the Regional Laboratory, the sample number is an identification number given by the Laboratory in which the tests were made and the test number is merely a consecutive number for reference purposes. Free fatty acid is expressed as per cent oleic and the Baume' strength is within the limits specified in the A.O.C.S. handbook. The values in the next three columns are obtained by three different methods of calculating the amount of dry sodium hydroxide to be used per 100 gm. of oil. Since for any particular determination a specific quantity of lye is added, obviously these values are interconvertible. In the official tentative method, the formula F.F.A.

- + 0.54 gives the maximum amount of alkali 5.2

and the fraction of this maximum, given in the "Max. times" column, is used in the test. The amount of lye to be used can also be calculated on the basis of the amount of sodium hydroxide necessary to neutralize the free fatty acids, so-called "theory," equal to (F.F. A.) x (0.142) grams. For a given test, a certain excess is used and the grams of dry sodium hydroxide calculated as theory times a certain number, given in the "Theory times" column, or as theory plus a certain amount, given in the "Theory plus" column. Of the three, that figure which is not enclosed in parenthesis is the one actually used in calculating the amount of dry NaOH, while the other two are conversions from it. (Text continued on page 214)

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SERIES I-SODIUM HYDROXIDE

SERIES I-SODIUM HYDROXIDE (Continued)

	Theory (Per 100 gm. Oil)													The (Per 100	eory ) gm. Oil	)	
Sample	Test No.	FFA	Be'	Max. Times	Times	Plus	Refining Loss	Remarks	Sample	Test No.	FFA	Be'	Max. Times	Times	Plus	Refining Loss	Remarks
252 252 253 253 254 255 255 255 255 255 255 255 255 255	$\begin{array}{c} 1 \\ 2 \\ 3 \\ 4 \\ 5 \\ 6 \\ 7 \\ 8 \\ 9 \\ 0 \\ 11 \\ 12 \\ 3 \\ 14 \\ 5 \\ 6 \\ 7 \\ 8 \\ 9 \\ 0 \\ 11 \\ 12 \\ 22 \\ 24 \\ 5 \\ 22 \\ 22 \\ 22 \\ 22 \\ 22$	0.45 0.45 0.45 0.34 0.34 0.34 0.28 0.83 0.80 0.50 0.50 0.45 0.45 0.45 0.45 0.45 0.4	12° " " " " " " " " " " " " " " " " " " "	***************************************	(7.35) (4.9) (9.4) (6.27) (9.4) (6.27) (11.2) (7.47) (4.45) (2.97) (4.58) (3.05) (6.72) (4.48) (8.72) (6.67) (7.35) (4.9) (7.35) (6.79) (9.4) (7.35) (6.79) (9.4) (7.35) (6.79) (9.4) (7.35) (6.27) (7.35) (6.27) (7.35) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (9.4) (7.35) (6.27) (7.35) (1.27)	(.406) (.249) (.406) (.254) (.406) (.257) (.407) (.232) (.407) (.232) (.407) (.232) (.406) (.247) (.406) (.247) (.406) (.249) (.406) (.249) (.406) (.249) (.406) (.254) (.406) (.254) (.406) (.254) (.406) (.254) (.406) (.257) (.407) (.233) (.406) (.257) (.407) (.233) (.406) (.247) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.233) (.407) (.356) (.477) (.358) (.357) (.406) (.249)	$\begin{array}{c} 1.24\\ 1.205\\ 3.995\\ 2.437\\ 1.870\\ 4.526\\ 3.995\\ 3.4595\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 3.995\\ 5.724\\ 5.3897\\ 1.655\\ 3.44\\ 4.99\\ 5.726\\ 1.65\\ 3.75\\ 4.299\\ 4.6\\ 8.390\\ 5.946\\ 4.991\\ 2.1\\ 2.00\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 4.950\\ 5.946\\ 5.946\\ 4.950\\ 5.946\\$	Water in oil Foots soft Foots soft Foots soft Water in oil Foots Soft Water in oil Foots Soft Foots soft Foots soft Foots soft Foots Soft Foots Soft Foots OK Foots OK Water in oil Water in oil Foots soft Foots OK Foots Soft Foots Soft Foots Soft Foots Soft Foots Soft Foots OK Foots OK Foots OK Foots OK Foots OK Foots Soft Foots Soft Foots Soft Foots Soft Foots Soft Foots OK Foots OK	252 253 253 253 255 255 255 255 255 255	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{c} 0.41\\ 0.34\\ 0.34\\ 0.34\\ 0.49\\ 0.49\\ 0.49\\ 0.34\\ 0.28\\ 0.34\\$	$16^{\circ}$	$\frac{1}{3}$	$\begin{array}{c} (7.09)\\ (3.55)\\ (9.4)\\ (6.27)\\ (6.27)\\ (6.27)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (11.2)\\ (7.47)\\ (4.48)\\ (3.05)\\ (6.72)\\ (4.48)\\ (5.33)\\ (4.9)\\ (9.4)\\ (4.81)\\ (6.55)\\ (6.72)\\ (4.81)\\ (6.55)\\ (6.72)\\ (4.81)\\ (6.55)\\ (6.72)\\ (4.81)\\ (6.55)\\ (11.2)\\ (11.36)\\ (6.55)\\ (11.36)\\ (6.55)\\ (11.36)\\ (6.55)\\ (11.36)\\ (6.55)\\ (11.36)\\ (11.36)\\ (6.55)\\ (11.36)\\ (11.36)\\ (6.55)\\ (11.36)\\ (11.36)\\ (6.55)\\ (11.36)\\ (11.$	$\begin{array}{c} (.354)\\ (.148)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.257)\\ (.406)\\ (.257)\\ (.406)\\ (.257)\\ (.407)\\ (.232)\\ (.407)\\ (.232)\\ (.407)\\ (.233)\\ (.406)\\ (.247)\\ (.406)\\ (.244)\\ (.406)\\ (.244)\\ (.406)\\ (.244)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.406)\\ (.254)\\ (.247)\\ (.232)\\ (.406)\\ (.247)\\ (.232)\\ (.406)\\ (.247)\\ (.243)\\$	4.88         6.01         3.92         6.01         3.92         6.01         3.92         6.01         3.92         6.21         4.90         4.90         4.00         3.96         5.02         7.267         6.35         4.09         2.227         6.35         4.09         2.227         6.35         4.98         2.227         6.35         4.98         5.724         4.42         4.93         4.11         4.67         4.5         3.93         4.9	Foots slippery Foots too soft Foots soft Foots soft Foots soft Foots soft Foots soft Foots soft Foots soft Foots Soft Foots OK Foots OK Foots Soft Foots S
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SERIES II-SODIUM HYDROXIDE-SODIUM SILICATE MIXTURES

Sample	Test No.	FFA	Be'	Max. Times	Th (Per 100 Times	eory gm. oil) Plus	Addition	Refining Loss	Remarks
252 253 256 252 253 253 253 256 256 256 256 256 256 256 256 256 256	121 122 123 124 125 126 127 128 129 130 131 132 133 134 135 136	.45 .34 .83 .45 .45 .34 .34 .83 .83 .83 .83 .83 .83 .83 .83 .83 .83	14° " 16° " " 18° " " " 20°	***************	(7.35) (9.4) (4.45) (7.35) (4.9) (9.4) (6.27) (4.45) (6.27) (4.18) (6.27) (1.98) (6.27) (1.98) (6.27)	(.406) (.406) (.407) (.406) (.249) (.406) (.254) (.407) (.232) (.254) (.154) (.232) (.115) (.254) (.254)	½       40 percent Na silicate         ½       40 percent SiO2 as silicate "S"         5 percent SiO2 as silicate "S"	0.91 and 1.01 0.66 and 0.61 2.22 and 1.67 1.41 and 1.35 1.03 and 1.16 0.71 and 0.91 0.92 and 0.94 1.85 and 2.06 1.78 and 1.80 0.92 and 0.91 0.93 and 0.81 1.96 1.98 1.68 1.94 1.32	Water in oil Water in oil Water in oil Foots OK Foots OK Water in oil Water in oil Water in oil Water in oil Water in oil Water in oil Foots OK Poured after chilling 5 hrs. at 10° C. Foots OK Water in oil; 5 hrs. at 10° C. Foots OK Water in oil; 5 hrs. at 10° C.
253 253 255 255	137 138 139	.34 .34 .28 28	  	1/3 1/3 1/2	(4.18) (4.18) (7.47) (7.47)	(.154) (.154) (.257)	5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S"	0.79 0.62 0.51	temp., 2 hrs. Water in oil; poured after standing room temp., 2 hrs. Water in oil Water in oil
255 256 256 256 266 <sup>1</sup>	141 142 143 144 145	.23 .83 .83 .83 .19	** ** ** **	72 1/2 1/2 1/3 1/3 (.309)	(7.47) (2.97) (1.98) (1.98) (6.55)	(.232) (.232) (.115) (.115) .15	5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S" 1+34 gm. silicate "G" dissolved in 5 cc. H <sub>2</sub> O	2.05 2.16 2.11 2.10 0.86	water in oil; poured after standing over night Foots OK Poured after standing overnight Foots OK Poured after 5 hrs. at 10° C. Foots OK

# SERIES II-(Cont'd)

Sample	Test No.	FFA	Be'	Max. Times	Tl (Per 10 Times	heory 0 gm. oil) Plus	Addition	Refining Loss	Remarks
2661	146	.19	20°	(.640)	(13.67)	.342	1+34 gm. silicate "G" dissolved		Water in oil; foots too slippery.
252 2521 2521	147 148 149	.45 .41 .41	22° "'	1/8 1/3 1/3	(3.26) (3.55) (3.55)	(.145) (.148) (.148)	5 percent SiO <sub>2</sub> as silicate "S" 1 percent SiO <sub>2</sub> as silicate "99" 1 percent SiO <sub>2</sub> as silicate "99"	1.46	Foots OK. Foots too soft. Foots too soft; poured after standing
2521 2521	150 151	.41 .41	**	1% 1%	(3.55) (3.55)	(.148) (.148)	2 percent SiO2 as silicate "99" 2 percent SiO2 as silicate "99"	•••••	Foots too soft, Foots too soft; poured after standing
2521 2521	152 153	.41 .41	**	1/3 1/3	(3.55) (3.55)	(.148) (.148)	4 percent SiO2 as silicate "99" 4 percent SiO2 as silicate "99"		Foots too soft, Foots too soft; poured after standing overnight without chilling
2521 2521	154 155	.41 .41	**	₩ ₩3	(3.55) (3.55)	(.148) (.148)	5 percent SiO2 as silicate "99" 5 percent SiO2 as silicate "99"	2.92 3.01	Foots OK. Poured after standing overnight without chilling
2521 2521 2521 2521 253 253 253	156 157 158 159 160 161	.43 .43 .43 .43 .34 .34	66 66 68 68 68 68 68	1/3 1/3 1/3 1/3 1/2 1/2 1/2	(3.4) (3.4) (3.4) (3.4) (6.27) (6.27)	(.146) (.146) (.146) (.146) (.254) (.254)	5 percent SiO <sub>2</sub> as silicate "99" 5 percent SiO <sub>2</sub> as silicate "99" 5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S"	2.9 1.43 1.12	Foots too soft. Foots too sloppery. Foots OK. Foots OK. Water in oil; poured after 2 hrs. at room
253 253	162 163	.34 .34	"	1/3 1/3	(4.18) (4.18)	(.154) (.154)	5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S"	$\begin{array}{c} 1.16\\ 1.20 \end{array}$	temperature. Foots OK. Poured after standing overnight without
2531 2531	164 165	.49 .49	 	1/2 1/3	(3.04) (3.04)	(.142) (.142)	2 percent SiO2 as silicate "99" 2 percent SiO2 as silicate "99"		chilling. Foots too soft. Foots too soft; poured after standing
2531 2531	166 167	.49 .49	22°	1/3 1/3	(3.04) (3.04)	(.142) (.142)	3 percent SiO2 as silicate "99" 3 percent SiO2 as silicate "99"	*****	Foots too soft; poured after standing over
2531 2531	168 169	.49 .49	**	1/3 1/3	(3.04) (3.04)	(.142) (.142)	4 percent SiO2 as silicate "99" 4 percent SiO2 as silicate "99"	·····	Foots too soft; Foots too soft; poured after standing
2531 2531	170 171	.49 .49	**	1/3 1/3	(3.04) (3.04)	(.142) (.142)	5 percent SiO <sub>2</sub> as silicate "99" 5 percent SiO <sub>2</sub> as silicate "99"	4.04 4.16	Foots OK. Poured after standing overnight without
2531 2531 2531 2531 2531 2531	172 173 174 175 176	.51 .51 .49 .49	44 44 44 44	1% 1% 1% 1%	(2.94) (2.94) (2.94) (3.04) (3.04)	(.140) (.140) (.14 <b>0</b> ) (.142) (.142)	5 percent SiO <sub>2</sub> as silicate "99" 5 percent SiO <sub>2</sub> as silicate "99" 5 percent SiO <sub>2</sub> as silicate "99" 5 percent SiO <sub>2</sub> as silicate "metso" 5 percent SiO <sub>2</sub> as silicate "metso"	3.2 3.0 3.05	Foots too soft. Foots too soft. Foots OK. Poured after standing overnight without
2531 255 255	177 178 179	.51 .28 .28	44 44 44	1/3 1/2 1/2	(2.94) (7.47) (7.47)	(.140) (.257) (.257)	5 percent SiO2 as silicate "S" 5 percent SiO2 as silicate "S" 5 percent SiO2 as silicate "S"	2.4 1.11 1.04	Foots OK. Foots OK. Water in oil; poured after standing over-
255 255	180 181	.28 .28	**	1/8 1/3	(4.98) (4.98)	(.158) (.158)	5 percent SiO2 as silicate "S" 5 percent SiO2 as silicate "S"	0.86 0.84	night without chilling. Foots OK. Water in oil; poured after standing over-
255 255 255	182 183 184	.28 .28 .28	** ** **	⅓ ⅓ ⅓	(4.98) (4.98) (4.98)	(.158) (.158) (.158)	5 percent SiO2 as silicate "metso" 5 percent SiO2 LaPine metasilicate 5 percent SiO2 LaPine metasilicate	0.81 1.13 0.98	night without chilling. Foots OK. Foots OK. Poured after standing overnight without
255 255	1 <b>85</b> 186	.20 .20	" 22°	1/3 1/3	(6.79) (6.79)	(.164) (.164)	5 percent SiO2 as silicate "metso" 5 percent SiO2 as silicate "metso"	0.73 0.79	chilling. Water in oil. Water in oil; poured after standing over-
255 255	187 188	.20 .20	66 66	⅓ ⅓	(6.79) (6.79)	(.164) (.164)	5 percent SiO2 as silicate "99" 5 percent SiO2 as silicate "99"	0.76 0.84	Foots OK. Poured after standing overnight without
2551 2551	189 190	.26 .26	**	½ ½	(5.33) (5.33)	(.160) (.160)	5 percent SiO2 as silicate "metso" 5 percent SiO2 as silicate "metso"	2.26 2.35	Foots OK. Poured after standing overnight without chilling
256 256	191 192	.83 .83	**	$\frac{1/2}{1/2}$	(2.97) (2.97)	(.232) (.232)	5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S"	3.05 3.20	Foots OK. Poured after standing overnight at room temperature
256 256	193 194	.83 .83	**	½ ½	(2.97) (2.97)	(.232) (.232)	5 percent SiO <sub>2</sub> as silicate "S" 5 percent SiO <sub>2</sub> as silicate "S"	3.30 3.21	Foots OK. Poured after standing overnight without chilling
264 265 265 265 266 266 266 <sup>1</sup>	195 196 197 198 199 200 201	.26 .26 .23 .23 .17 .17 .19	48 66 66 66 66 66	⅓ ⅓ ⅓ ⅓ ¼ (.281)	(5.33) (5.33) (5.96) (5.96) (7.98) (7.98) 6	(.160) (.160) (.162) (.162) (.167) (.167) (.135).	<ul> <li>5 percent SiO<sub>2</sub> as silicate "S"</li> <li>5 percent SiO<sub>2</sub> as silicate "G"</li> <li>5 percent SiO<sub>2</sub> as silicate "S"</li> <li>5 percent SiO<sub>2</sub> as silicate "G"</li> <li>5 percent SiO<sub>2</sub> as silicate "G"</li> <li>5 percent SiO<sub>2</sub> as silicate "G"</li> <li>2 gm. silicate "G" dissolved in 3 cc. H<sub>2</sub>O.</li> </ul>	3.13 and 3.20 3.17 1.29 1.14 and 1.08 1.2 1.7 and 1.1 1.20	Foots OK. Foots OK. Foots OK. Foots OK. Foots OK. Foots OK. Foots OK.
2661	202	.19	22°	<b>(</b> .567)	(12.12)	.3	2 gm. silicate "metso" dissolved in 3 cc. H <sub>2</sub> O.	1.31	Water in oil.
266 268	203 204	.1 <b>7</b> .21	**	¥3	(7.98) (6.49)	(.167) (.164)	<ol> <li>1.5 gm. solid silicate "metso" (order of addition of silicate and lye reversed in duplicates).</li> <li>1.5 gm. solid silicate 'metso" (order of addition of silicate and lye reversed in duplicates).</li> </ol>		Foots too soft. Foots too soft.

# SERIES III-MISCELLANEOUS TESTS

Sample	Test No.	FFA	Be'	Max. Times	T (Per 10 Times	heory 0 gm. oil) Plus	Addition	Refining Loss	Remarks
24 <b>9</b>	205	.39	14°	7⁄8	(9.71)	(.483)	2.5 gm. A1 Stearate + NaOH =	3.8	Foots soggy.
252	206	.45	16°	3⁄4	(7.35)	(.406)	2 gm. A1 <sub>2</sub> (SO <sub>4</sub> ) $_{3.18H_2O}$ dis-		Foots too soft.
252	207	.45	"	1/2	(4.9)	(.249)	$2 \text{ gm. A1}_2 (SO_4)_{3.18}\text{H}_2\text{O}$ dis-	•	Foots too soft.
253	208	.34	**	3⁄4	(9.4)	(.406)	2 gm. A12 (SO4) 8.18H2O dis-		Foots too soft.
252	209		••••	•		•••••••	5 gm. Na <sub>3</sub> PO <sub>4</sub> .12H <sub>2</sub> O dissolved	1.10 and 1.24	Foots fairly firm.
2521	210	.41	14°	2⁄3	(7.09)	(.354)	Straight lye then 1 gm. of 25 per-	1.72 and 1.64	Water in oil.
2521	211	.41	"	⅓	(3.55)	(.148)	Straight lye then 1 gm. of 25 per-	1.84 and 1.72	Foots OK.
2521	212	.41	22°	3	(3.55)	(.148)	Straight lye then 1 gm. of 25 per-		Foots too soft.
2531 2531	213 214	.49 .49	• •	⅓ ⅓	(3.04) (3.04)	(.142) (.142)	20 percent solution Na <sub>3</sub> PO <sub>4</sub> .12H <sub>2</sub> O 20 percent solution Na <sub>3</sub> PO <sub>4</sub> 12H <sub>2</sub> O 20 percent solution Na <sub>3</sub> PO <sub>4</sub> 12H <sub>2</sub> O	•••••	Water in oil; foots too soft. Foots too soft; poured after standing overnight without chilling.
2531 2531	215 216	.49 .49	13° 13°	1⁄3 1⁄3	(3.04) (3.04)	(.142) (.142)	DuPont 57X clay mixture. DuPont 57X clay mixture.	3.02 3.00	Foots OK. Poured after standing overnight without chilling.
2531 265	217 218	.49 .23	20°	¾ (.641)	(3.04) (11.47)	(.142) .342	10 percent solution of Na <sub>2</sub> CO <sub>8</sub> . 2 gm.Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .18H <sub>2</sub> O dissolved in 1 2 gm meteo dissolved in 5 cc H	ye;	Foots too soft. Foots too soft.
2661 2661	219 220	.19 .19	68 66	(.644) (.644)	(13.67) (13.67)	.342 .342	2 gm.A1 <sub>2</sub> (SO <sub>4</sub> ) <sub>3.18H<sub>2</sub>O dissolved in 1 2 gm.A1<sub>2</sub>(SO<sub>4</sub>)<sub>3.18H<sub>2</sub>O dissolved in 1 1 + <math>\frac{3}{4}</math> gm. silicate "G" dissolved</sub></sub>	lye, ye; 1.86 in	Foots too soft. Foots OK.
2661	221	.19	"	(.644)	(13.67)	.342	5 cc. H2O. 2 gm.A12(SO4)3.18H2O dissolved in 1 1 + 34 gm. silicate "G" dissolved	ye; 1.59 lin	Foots OK.
2661	222	.19	**	(.644)	(13.67)	.342	2 gm.A12(SO4)s.18H2O dissolved in	lye; 1.34	4 Foots OK.
<b>2</b> 68	223	.21	**	(.641)	(12.47)	.342	2 gm. metso dissolved in 5 cc. in 2 gm.A1 <sub>2</sub> (SO <sub>4</sub> ) <sub>8.18H<sub>2</sub>O dissolved in 1</sub>	20. ye; 6.56	5 Foots scummy.
269	224	.65	"	(.653)	(4.7)	.342	2 gm. Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .18H <sub>2</sub> O dissolved lye; 1 + 3/4 gm. silicate "G" dissol	20. in 4.28 wed	Foots OK.
266	225	.17	14°	⅔	(15.82)	(.358)	2 gm. silicate "99" dissolved in 5	cc	Water in oil.
266 266	226 227	.17 .17	••	<del>%</del>	(15.82)	(.358)	5.7 gm. B.W.; 5 gm. HC1; then the lye. 1.5 gm. metso dissolved in 5 cc. He	lye 20;	Foots too slippery. Foots too scummy.
266	228	.17	22°	⅓	(7.98)	(.167)	1.4 gm. HC1; 1.5 gm. solid silid	cate 0.98	Foots fair.
268	229	.21	14°	⅔	(12.98)	(.357)	2 gm. silicate "metso" dissolved in 5	cc	Water in oil, foots slippery.
268	230	.21	**	⅔	(12.98)	(.357)	5.7 gm. silicate "B.W."; 5 gm. H	C1;	Foots too soft.
268	231	.21	22°	⅓	(6.49)	(.164)	2.5 gm. silicate metso dissolved in 5	cc. 0.93	Foots OK.
268	232	.21	"	1/2	(6.49)	(.164)	1.4 gm. HC1; 1.5 gm. solid silicate "n so"; then lye.	net	Fogts too soft.
In the f r.p.m., r	ollowing lext 20	, first t minutes	he silic at 65	ate and ° and 7	acid are 0 r.p.m.,	added, foll finally, 1 l	owed at once while stirring, by the lye, nour's standing at 65° C. From then on	then there is 1 , the regular p	hour of cold stirring at 22° C. and 250 rocedure is followed.
266	233	.19	20°	(.480)	(10.27)	.25	5 cc. 40 percent silicate "metso" } 8.1	gm	Water in oil.
268	234	.21	"	(.482)	(9.38)	.25	$1 + \frac{1}{2}$ cc. con. HCl. 5 cc. 40 percent silicate "metso" 8.1 ( $1 + \frac{1}{2}$ cc. con. HCl.	gm. 2.97	Foots OK.
In the for and acid then on.	ollowing are ad the ree	, the all ded while ular pro-	cali is le stirr ocedure	added ar ing at 7 is follo	nd there i '0 r.p.m.	is 1 hour of and 65° C.	stirring at 22° C. and 250 r.p.m., then , and the stirring continued for 10 mir	1 10 minutes at nutes longer; fin	: 65° C. and 70 r.p.m.; then, the silicate nally, 1 hour's standing at 65° C. From
0661	015	10	000	( 400)	(10.07)				Franks to a soft

2661	235	.19	20°	(.480)	(10.27)	.25	5 cc. 40 percent silicate "metso"; 4 cc.	•••••	Foots too soft.
266	236	.17	"	(.479)	(11.36)	**	con. HC1. 4 cc. con. HC1., 5 cc. 40 percent silicate	•	Foots too soft.
268	237	.21	"	(.482)	(9.38)	**	5 cc. 40 percent silicate "metso," 4 cc.		Foots too soft.
268	238	.21	**	(.482)	(9.38)	**	con, HCl., 5 cc. 40 percent silicate "metso."	•••••	Foots too soft.



### SERIES V.

Tests made on sample No. 258 by representatives of three laboratories at the Armour Laboratory on April 10 and 11, 1941. While stirring at 250 r.p.m. and  $22^\circ$ , plus or minus  $2^\circ$  C. 2% cc. of 50 percent sodium metasilicate pentabydrate are pipetted in, then 1 cc. conc. HCl, and the cold stirring continued 10 minutes. Next, 6.6 cc. of lye pipetted in and the cold stirring continued for 1 hr.; change to 65°, plus or minus  $2^\circ$  C., and 70 r.p.m. for 15 minutes, and settle 1 hour at 65° plus or minus  $2^\circ$  C.

			Grome	Mar	Th (Per	Theory (Per 100 gm.		Evaporation Loss*		Remelt Oil*		No. of Remelts*		ning ss*	Pomorle	
Laboratory	FFA	Be'	Lye (percent)	Times	Times Plus		· A B Gm. Gm.		A Gm.	B Gm.	Α	В	A %	B %	Keniai ks	
Armour	0.50	28°	1.64	(.558)	5	(.284)	2.2	2.8	9.5	10.5	3	3	5.0	5.3	One of duplicates with some	
Soybean	0.50	28°	1.64	(.558)	5	(.284)	2.4	<b>.</b>	11.6	•••••	3	3	5.4	<b></b>	One of duplicates with soft	
Swift	0.50	28°	1.64	(.558)	5	(.284)	2.8	2.6	11.7	9.4	3	3	5.2	5.1	foots; other firm. Foots firm in both dupli- cates.	
* Run in dupli	cate.															
Results of a	previou	is test	made on	the same	e sampl	e of soy	bean oi	l in th	ne differ	ent labo	ratori	es.				
Armour Soybean Swift	0.50 0.50 0.50 0.50	30° 30° 30° 30°	1.51 1.50 1.50 1.51	(.558) (.558) (.558) (.558)	5 5 5 5	(.284) (.284) (.284) (.284)	2 1 	.7	0 1 	.8		1	3 4 5 4	.2 .6 .97 .2	Foots firm. Foots OK Foots OK Well grained foots	

#### oil & soap

# SERIES V-Cont'd).

ARMOUR	SWIFT	SOYBEAN		
Weighing samplesTare and weights	Tare	Weights		
Cold stirringWater run in at ca 10° C., temperature raised to 20°-24° with steam.	Tap water at room temperature, less than 24° C. In summer ice used to keep down temperature.	Tap water at ca 20° C. run in and tem- perature kept constant at 22° C. with thermoregulator.		
Hot stirring Preheated to ca 67° C. in separate tank; kept at temperature with steam,	Steam used to raise temperature of cold stirring bath, requiring ca $1 + \frac{1}{2}$ min-	Preheated in separate tank, kept at 65° C. with thermoregulator.		
Chilling	Cold tap water run in and let stand 12°- 20° C.	In refrigerator at 10° C. for 1 hr.		
OvernightIn water bath at room temp.	In water bath at room temp.	On work bench at room temp.		
Chilling	Cold tap water run in and let stand 1 hr. (ca 12°-24° C.).	In refrigerator at 10° C. for 1 hr.		
Decanting	For 30 min. into tared steel cup; wipe off at end.	For 30 min. into tared beaker.		
Remelting	30 min. in separate open water bath at ca 75° C.; cups tilted; kept at temp. with steam.	In refining apparatus at 70°, plus or minus 2° C.; kept at temperature with steam.		
Chilling	Cold water (tap) run in and let stand 1 hr. (ca 12°-24° C.).	In refrigerator at 10° C. for 1 hr.		
DrainageDirectly into first pour off and let drain 30 min.; weigh soap stock cup both be- fore and after drainage; allow for evap- oration loss.	Into tared beaker for ca 2 min. and wipe off excess with finger.	Into tared beaker for 30 min.		

#### SERIES IV

In the following, while stirring at 250 r.p.m. and 22°, plus or minus 2° C., 5 cc. of 40 percent sodium metasilicate pentabydrate are pipetted in, then  $1+\frac{1}{2}$  cc. con. HC1., and the cold stirring continued 10 minutes. Next the lye is pipetted in the cold stirring continued for 1 hour; change to 65°, plus or minus 2° C., and 70 r.p.m. for 15 minutes, and settle 1 hour at 65°, plus or minus 2° C. From then on, the regular procedure is followed.

			•	Grams	3	71		D.C.	
Sam	- Test			Iye	Mari	'ner 100	ary arm oil)	ing	
ple	No.	FFA	Be'	cent	Times	Times	Plus	Loss	Remarks
254	239	0.34	20°	1.72	(.410)	(5.14)	0.2	2.25	Water in oil.
254	240	0.34	-,,	1.68	(.399)	5	(.193)	2.20	Water in oil.
257	241	0.80	"	2.18	(.452)	(2.76)	0.2	3.73	Floating scum.
257	242	0.80	**	3.94	(.819)	5	(.454)	3.63	Foots OK.
258	243	0.50	**	1.88	(.426)	(3.82)	0.2	1.71	Water in oil.
258	244	0.50	44	2.46	(.558)	5	(.284)	1.91	Water in oil.
265	245	0.23	**	1.62	(.398)	(7.12)	0.2	0.77	Water in oil.
266	246	0.23	"	1.14	(.280)	5	(.131)	1.42	Water in oil.
266 <sup>1</sup>	247	0,19	**	1.58	(.394)	(8.41)	0.2	••••••	Water in oil.
2661	248	0.19	"	0.94	(.234)	5	(.108)		Water in oil
268	249	0.21		1.60	(.396)	(7.71)	0.2	2.93	Foots OK.
268	250	0.21		1.04	(.257)	<u> </u>	(.119)	0.98	Foots UK.
In t 50 p	he follow ercent s	ving, t ilicate	the j solu	proced	ure is as and 1 cc.	above e con. H	xcept fo C1.	or usin	$1g 2 + \frac{1}{2}$ cc. of
254	251	0.34	30°	1.06	(.410)	(5.14)	0.2	1.85	Foots firm.
254	252	0.34	"	1.06	(.410)	(5.14)	0.2	1.85	Foots firm.
254	253	0.34		1.02	(.399)	5	(.193)	1.79	Foots hrm.
254	254	0.34		1.02	(.399)	5	(.193)	2.11	Foots hrm.
257	255	0.80		1.32	(.452)	(2.76)	0.2	3.33	Foots scummy.
257	256	0.80	"	1.33	(.452)	(2.76)	0.2	4.4	Foots scummy,
257	Switt	0.80		1.55	(.452)	(2.70)	0.2	4.4	sloppy.
257	Armour	0.80	"	1.33	(.452)	(2.76)	0.2	3.6	Foots firm.
257	257	0.80		2.40	(.819)	5	(.454)	4.64	Foots OK.
257	258	0.80		2.40	(.819)	5	(.454)	4.57	Foots OK.
257	Swift	0.80	••	2.42	(.819)	5	(.454)	5,1	Foots well
257	Armour	0.80	"	2.43	(.819)	5	(.454)	4.4	Foots firm.
258	259	0.50	"	1.14	(.426)	(3.82)	Ò.2	4.23	Foots OK.
258	260	0.50	**	1.14	(.425)	(3.82)	0.2	4.28	Foots OK.
258	Swift	0.50	**	1.15	(.425)	(3.82)	0.2	3.8	Well grained
258	Armour	0 50	**	115	( 426)	(3.82)	0.2	35	foots. Foots firm
258	261	0.50	""	1 50	(558)	5	(.284)	4.60	Foots OK.
258	262	0.50	"	1.50	(558)	š	(.284)	5.97	Foots OK.
258	Swift	0.50	"	1.51	(.558)	Ĵ.	(.284)	4.2	Well grained
						2			foots.
258	Armour	0.50		1.51	(.558)	17 10	(.284)	3.2	Foots hrm.
265	263	0.23	"	0.98	(.398)	(7.12)	0.2	1.18	Foots UK.
265	264	0.23	"	0.98	(.398)	(7.12)	(121)	2.50	Foots UK.
203	203	0.23		0.70	(280)	5	(131)	1.90	Foots OK.
205	267	0.23	"	0.70	(394)	(8 41)	$\hat{0}_{2}^{(101)}$	1 08	Water on re.
200-	207	Ų.19		0.90	(.334)	(0.41)	0.2	4.00	melting.
2661	268	0.19	"	0.96	(.394)	(8.41)	0.2	0.71	Foots OK.
2661	Swift	0.19	£4	0.97	(.394)	(8.41)	0.2	1.1	Well Grnd.
									foots. 0.5 gm.
2661	Armour	0.10	"	0.97	( 394)	(0 11)	0.2	1 2	Water.
2661	269	0.19	"	0.58	(234)	5	(108)	1.2	Foots OK
2661	270	0.19	"	0.58	(234)	ŝ	(108)	1.14	Foots OK.
2661	Swift	0.19	"	0.64	(.234)	š	61085	11	Well grnd
					·····	•	(,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	***	foots.
2661	Armour	0.19	"	0.61	(.234)	5	(.108)	0.61	Foots firm.
268	271	0.21	"	0.98	(.396)	(7.71)	0.2	3.61	Foots sl.
260	272	0.21	"	0.09	( 206)	17 41			scummy.
200	414	V.41		0.98	(.390)	(7.71)	0.2	3,76	Foots si.
268	273	0.21	**	0.64	(.257)	5	(.119)		Foots too
	071	0.04							scummy.
208	2/4	0.21	••	U.64	(.257)	5	(.119)	3.66	Foots scummy.

CENTRIFUGAL SEPARATION OF FOOTS IN REFINING TEST

A number of tests were made in the Swift Research

Laboratories to explore the possibility of obtaining a better and quicker separation of foots by use of a centrifuge than is now obtained by settling. The machine used for this work was a special unit built for colloidal solid separation in certain types of water. It was powered by a half-horse motor which gave an R.P.M. of 1725. Pear shaped glass containers of 100 cc. volume with the lower end drawn out into a graduated cylinder holding about 3 cc. were used to carry the oil-foots mixture. Most of the tests made were qualitative in nature, designed to observe the compactness of the separated foots. The soybean oil was first refined in accordance with the tentative method and a 50 cc. volume of the oil-foots mixture was quickly poured into the pear shaped centrifuge containers which, after various methods of treatment, were whirled for 5 minutes. The oil was then drained off and the foots reheated at 75° C. which was followed by a second centrifugal treatment. There was practically no further oil separation in this second whirling.

october.

1941

The preliminary work with the centrifuge indicates that a firm foots with a minimum amount of oil results from such treatment.

## RECOMMENDATIONS FOR NEXT YEAR'S REFINING COMMITTEE ACTIVITIES

I. The refining method recommended by the Regional Soybean Laboratory as a result of the extensive investigational program carried out there shows sufficient promise to be studied cooperatively by the Refining Committee.

II. The use of a centrifuge for quick and more efficient separation of foots may well be given further consideration.

III. It is hoped that the Regional Laboratory will again be in a position to assist in the Investigational Program in cooperation with the Refining Committee. Such cooperation is essential for a reasonably quick and satisfactory solution to the extracted soybean oil refining problem.

Refining committee:

E R Barrow	Lamar Kishlar
C. B. Cluff	N. F. Kruse
C. A. Coffey	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	D. H. Wheeler
H. S. Mite	chell, Chairman