Refining Test Methods Are Revised

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The following revised method for refining tests has been approved by the Refining Committee of the American Oil Chemists Society.

Rule 264: Crude Cottonseed Oil Chemists Reports

(See Rule 272 for Method of Analysis)

Determination of Color and Excess Loss. Refining tests for settlements, shall be subject to the following provisions:—

(a) At least three refinings shall always be made, using different strengths or amounts of sodium hydroxide, endeavoring to obtain a prime color with loss not over 9 per cent; the maximum amount permitted in rule 272 must be used on at least one of these refinings when the F.F.A. of the oil do not exceed 3 per cent, and on at least two of the refinings when the F.F.A. exceed 3 per cent. If a prime color with loss not over 9 per cent is not obtained, proceed as follows:

Color	Loss	Procedure
Prime or better	Over 9%	See paragraph (b)
Darker than Prime	9% or less	See paragraph (c)
Darker than Prime	Over 9%	See paragraph (d)

(b) If a prime color is obtained with loss over 9 per cent, additional refinings with less sodium hydroxide must be tried to obtain a prime color with the lowest possible loss.

(c) If the color is darker than prime, with loss under 9 per cent, additional sodium hydroxide over the normal maximum of rule No. 272 may be used, and if this gives a prime color with loss not over 9 per cent, this result is to be reported, but otherwise the results obtained with the normal maximum lye of rule No. 272 shall govern.

(d) If a prime color cannot be obtained without exceeding 9 per cent loss even as provided in paragraph (c) of this section, or if the color is darker than prime with loss over 9 per cent when using the maximum sodium hydroxide of rule No. 272, then of two or more tests showing varying results in color, that test which produces the best color must be reported, unless another of the tests produces an oil which is darker in color reading than this lighter oil by not more than 10 per cent, with a reduction in loss greater than the numerical difference in color reading; in which case the latter result is the one to be reported.

Illustration:

1—loss 12% Color, Red 10
2—loss 10.8% Color, Red 11
REPORT

(e) Chemists' reports for use in settlements must show the following data:

Free fatty acids, to tenths of one per cent.

Color of refined oil, in terms of 35 yellow and necesary red to produce best match.

Flavor of refined oil.

Amount and strength of lye used in refining.

Refining loss, to tenths of one per cent.

Rule 272: Crude Oils

(See Rule 264 for reporting refining tests for settlement purposes)

Section 1. Mixing Sample: Shake thoroughly with the can in

an inverted position, before removing any of the oil for analysis. If oil is cold, it must be warmed to 70°F, before attempting to mix.

Section 2. *Moisture or Volatile Matter*: Place in a tared metal or porcelain dish 10 grams of the well mixed sample, heat gently over a direct flame with a rotating or rocking motion, until oil barely smokes. Cool and report loss in weight as Moisture. Reserve for Meal Determination.

Section 3. *Meal or Impurities:* Wash the residue from the moisture determination into a beaker with kerosene and warm to dissolve the Oil content. Filter the resulting mixture on a tared Gooch crucible and wash the residue with petrolic ether until free from Oil. Dry to constant weight, divide by 0.8 and report as per cent Meal or Impurities.

Section 4. *Free Fatty Acids:* Weight 7.05 grams of the well mixed crude oil into a four-ounce bottle or 250 cc. flask. Add 50 cc. of freshly neutralized, denatured alcohol, or freshly neutralized saturated salt solution, and about 2 cc. of 1 per cent phenophthalien solution or alkali blue indicator. Titrate with 0.25 N NaOH solution, until upon the addition of one drop and violent shaking of the mixture a permanent change in color is produced. Report as per cent free fatty acids, oleic, the number of cubic centimeters of 0.25 N NaOH used in the titration.

Section 5. *Refining Crude Cottonseed Oil:*

(A) *Apparatus:*

Scales, 1000 grams capacity, sensitive to ½ gram. Weights, 500 grams to ½ gram.

Refining cups. Seamless or enameled iron cups, 4 to 4½ inches in diameter and 4 to 4½ inches deep, with a total capacity of about 900 cc.

Refining apparatus, a mechanical stirrer having T-shaped paddles with flat (not twisted) blades 1 inch wide and 3½ inches over all, set at right angles to the shaft of the paddle. Each paddle must be driven by a gear, or with other positive device, which will give the required speed without slipping even under maximum load. (Note: It is recommended that after September 1, 1928 only gear drives be permitted.) Speed of paddles in the cold water bath must be 250 ± 10 R.P.M. and the speed of paddles in the hot water bath must be 70 ± 5 R.P.M. The motor and driving belt or chain must be of sufficient capacity to drive all the paddles simultaneously at the specified speeds under full load.

Two water baths with thermometers, suitably arranged to hold cups while stirring; one bath to be maintained at 18° to 22°C, using ice to cool it to this temperature, if necessary, and the other maintained at 63° to 67°C. Level of water in the baths must be as high as the level of oil and lye in the cups, and the water must be continuously agitated to mantain uniform temperatures.

(B) *Sodium Hydroxide Solutions:*

These solutions must be of accurately known sodium hydroxide content, free from carbonate and other impurities and must be prepared in the following manner:

A super-saturated solution is first prepared by adding to each kilogram of pure, dry solid sodium hydroxide broken up into small pieces; ¾ of a kilogram of distilled water. Heat on the steam bath with occasional stirring, for at least three hours. Then allow

to settle and cool for 24 to 48 hours, keeping the vessel covered, to exclude air as far as possible. During the cooling a portion of the sodium hydroxide which had dissolved in the hot solution will crystallize out, and under these conditions the solution will then contain no measureable amount of carbonate. If such crystals do not separate out, the solution was not super-saturated.

Decant the solution from the residue, and if not perfectly clear filter through filter paper or asbestos. Dilute to the various concentrations required with distilled water which has been previously boiled and cooled. The final strength of the diluted solutions must be adjusted by actual titration, and not be specific gravity tests. The important point is to know the exact content of sodium hydroxide rather than the exact specific gravity or Beaume. Solutions of only the following strengths shall be used and they must be within the limits shown.

SODIUM HYDROXIDE TABLE

Nominal Degrees Beaume at 15° C.	Actual NaOH Content	Allowable Variations	
		Minimum	Maximum
10	6.57%	6.44%	6.70%
12	8.00	7.84	8.16
14	9.50	9.31	9.69
16	11.06	10.84	11.28
18	12.68	12.43	12.93
20	14.36	14.07	14.65
22	16.09	15.77	16.41
24	17.87	17.51	18.23
26	19.70	19.31	20.09
28	21.58	21.15	22.01
30	23.50	23.03	23.97

(C) *Choice of Lye.*

The maximum amount of sodium hydroxide allowable for refining shall be calculated from the following formulae:

For hydraulic or hot pressed oils:

$$\frac{\text{F.F.A.}}{5.2} \quad .54 = \text{Maximum NaOH}$$

For expeller or cold pressed oils:

$$\frac{\text{F.F.A.}}{4.365} + .77 = \text{Maximum NaOH}$$

The strengths of lye (expressed in Beaume degrees) to be used for refining oils of various F.F.A., shall be as follows:

Per Cent F. A. A.	Hydraulic Oil	Expeller Oil
1.5% or less	10° and 14° Beaumé	12° and 18° Beaumé
1.6-3.0	12° and 16°	16° and 20°
3.1-4.0	14° and 18°	16° and 20°
4.1-5.0	16° and 20°	16° and 20°
5.1-7.5	18° and 22°	20° and 26°
7.6-10.0	20° and 24°	20° and 26°
10.1-15.0	20° and 26°	20° and 30°
Over 15.0	22° and 28°	20° and 30°

Note: Lyes of intermediate strength between the two required by the above table may also be used, but results from same are only to be used when better than can be obtained with either of the strengths specified in the table.

The table on the page following shows the maximum percentage of each strength of lye allowable.

(D) *Refining Process:*

Place 500 grams of the thoroughly mixed sample of crude oil in a tared refining cup. Adjust the temperature of the oil and of the water bath to 18-22°C. Agitator must run at the rate of 250 R.P.M. plus or minus 10. Add the proper amount of NaOH solution to the oil, as quickly as possible, with the agitator running, and stir exactly 10 minutes (30 minutes to 60 minutes for expeller or cold pressed oil) from the time the lye is added. Then immediately transfer to the 65°C hot water bath, and stir at 70 = 5 R.P.M. for eight minutes. Temperature of oil must then be 55 to 60°C, adjusting the temperature of the water bath, if necessary, within the limits specified, to obtain this final oil temperature. Stop agitator and allow to settle in the water bath at 65°C for one hour. Cool by setting in the cold bath 30 minutes and allow to remain at normal room tempera-

	For Hydraulic or Hot Pressed Oils — Per Cent Lye of Varying Beaumé at 15° C. Containing Maximum NaOH										For Expeller or Cold Pressed Oils — Per Cent Lye of Varying Beaumé at 15° C. Containing Maximum NaOH				
% FFA	Max. NaOH	12°	14°	16°	18°	20°	22°	24°	26°	28°	Max. NaOH	16°	20°	26°	30°
1.5	.83	...	8.7	...	...	...	...	...	...	...	1.11	...	...	...	...
2.0	.93	11.7	...	8.4	...	...	...	...	...	...	1.23	11.1	8.5	...	...
2.5	1.03	12.9	...	9.3	...	...	...	...	...	...	1.34	12.1	9.3	...	...
3.0	1.12	14.0	11.8	10.1	8.8	...	...	...	...	...	1.45	13.1	10.1	...	...
3.5	1.22	...	12.8	...	9.6	...	...	...	...	...	1.56	14.1	10.9	...	...
4.0	1.31*	...	13.8	...	10.4	9.1	...	...	...	...	1.68	15.2	11.7	...	...
4.5	1.41	...	...	12.7	...	9.8	...	...	...	...	1.80	16.3	12.5	...	...
5.0	1.50	...	...	13.6	11.8	10.4	9.3	...	...	...	1.92	17.4	13.3	9.8	...
5.5	1.60	...	...	...	12.6	...	9.9	...	...	...	2.03	...	14.1	10.3	...
6.0	1.70	...	...	...	13.4	...	10.5	...	...	...	2.14	...	14.9	10.9	...
6.5	1.79	...	...	...	14.1	...	11.1	...	...	...	2.25	...	15.7	11.4	...
7.0	1.89	...	...	...	14.9	...	11.7	...	...	...	2.37	...	16.5	12.0	...
7.5	1.99	...	...	...	15.7	13.9	12.4	11.1	...	...	2.49	...	17.3	13.6	...
8.0	2.08	...	...	...	...	14.5	...	11.6	...	...	2.60	...	18.1	13.2	...
8.5	2.18	...	...	...	...	15.2	...	12.2	...	...	2.72	...	18.9	13.8	...
9.0	2.27	...	...	...	...	15.8	...	12.7	...	...	2.83	...	19.7	14.4	...
9.5	2.37	...	...	...	...	16.5	...	13.3	...	...	2.94	...	20.5	14.9	...
10.0	2.46	...	...	...	...	17.1	...	13.8	12.5	...	3.06	...	21.3	15.5	13.0
10.5	2.56	...	...	...	...	17.8	...	...	13.0	...	3.17	...	22.1	...	13.5
11.0	2.65	...	...	...	...	18.4	...	...	13.4	...	3.29	...	22.9	...	14.0
11.5	2.75	...	...	...	...	19.1	...	...	13.9	...	3.40	...	23.7	...	14.5
12.0	2.84	...	...	...	...	19.8	...	...	14.4	...	3.52	...	24.5	...	15.0
12.5	2.94	...	...	...	...	20.5	...	...	14.9	...	3.63	...	25.3	...	15.5
13.0	3.03	...	...	...	...	21.1	...	...	15.4	...	3.75	...	26.1	...	16.0
13.5	3.13	...	...	...	...	21.8	...	...	15.9	...	3.86	...	26.9	...	16.4
14.0	3.23	...	...	...	...	22.5	...	...	16.4	...	3.98	...	27.7	...	16.9
14.5	3.32	...	...	...	...	23.1	...	...	16.9	...	4.09	...	28.5	...	17.4
15.0	3.42	...	...	...	...	23.8	21.3	...	17.4	15.9	4.20	...	29.3	...	17.9
15.5	3.52	...	...	...	...	...	21.9	...	...	16.3	4.32	...	30.1	...	18.4
16.0	3.62	...	...	...	...	...	22.5	...	...	16.8	4.44	...	30.9	...	18.9
16.5	3.71	...	...	...	...	...	23.1	...	...	17.2	4.55	...	31.7	...	19.4
17.0	3.81	...	...	...	...	...	23.7	...	...	17.7	4.67	...	32.5	...	19.9
17.5	3.90	...	...	...	...	...	24.3	...	...	18.1	4.78	...	33.3	...	20.4
18.0	4.00	...	...	...	...	...	24.9	...	...	18.5	4.90	...	34.1	...	20.9
18.5	4.10	...	...	...	...	...	25.5	...	...	19.0	5.01	...	34.9	...	21.3
19.0	4.19	...	...	...	...	...	26.1	...	...	19.4	5.13	...	35.7	...	21.8
19.5	4.29	...	...	...	...	...	26.7	...	...	19.9	5.24	...	36.5	...	22.3
20.0	4.38	...	...	...	...	...	27.2	...	...	20.3	5.35	...	37.2	...	22.8

Part of table which could not be included above

F.F.A.	Max. NaOH	10°	Max. NaOH	12°	18°
1.5	.83	12.6	1.11	13.9	8.8

ture until the soap stock becomes hard and firm. Weigh the refining cup and contents, and deduct this weight from the total weight at beginning of test to obtain loss by evaporation. Decant the refined oil into a tared refining cup and drain the soap stock for 30 minutes. This oil is to be used for determination of grade. Weigh both oil and soap stock cups and contents. Melt the soap stock by setting it in water bath at 75° ± 2°C; without stirring, for 30 minutes; decant and weigh separately any additional oil thus recovered. Add this weight to the weight of refined oil first obtained, and subtract it from the soap stock weight. Repeat the remelting, if necessary, for periods of 30 minutes each until the recovered oil from the last remelting amounts to not over 2.5 grams.

Calculations: Determine refining loss by two methods of calculation, the results of which should check within one-quarter of one per cent.

Method No. 1: Weight of Crude Oil minus weight of Refined Oil gives refining loss.

Method No. 2: Weight of Soap Stock plus loss in evaporation, minus weight of NaOH solution used gives refining loss.