REPORT OF REFINING COMMITTEE FOR 1925-1926

This year's work is a continuation of last year's study of results obtained by limiting the discretion of the operator, as far as possible, in making refinings. Procedure was limited the same as described in last year's report.

Three samples of crude oil were tested, but results are not as good as those obtained last year, which was probably due to the poorer quality of oil handled this year, which was more difficult to refine. The following points, however, may be noted:

1. F.F.A. tests by different operators do not check as closely as we feel they should.

2. Colors by the revised method average practically the same as by the official method, but in all cases color readings vary much more than when made by an individual observer. This is probably due to differences in Lovibond glasses, nature of illumination and personal factors. No two operators used the same illumination. The lamps were reported to be 75, 100, 150, and 300 watts and one was not given.

3. Refining losses by the new procedure average distinctly lower than by the present official method, but they are not consistent.

Our conclusions and recommendations are as follows:

a. We feel that any improvement in results will be obtained only by a further limitation of variables still existing among different operators, in carrying out the refining operation, but do not feel that results so far obtained justify recommendations for radical changes in the present rule.

b. We feel that one improvement greatly to be desired is to restrict the choice of lye so that only two strengths can be used for any oil of a given F.F.A., and recommend the adoption of the following table:

F. F. A. %	Hydraulic Pressed Oils—Lye	Expeller Oils —Lye	F. F. A. %	Hydraulic Pressed Oils—Lye	Expeller Oils —Lye
3.1 or less	12° and 16°	16° and 20°	7.1-10.0	20° and 24°	24° and 28°
3.1- 5.0	16° and 20°	20° and 24°	10.1-15.0	22° and 26°	26° and 30°
5.1- 7.0	18° and 22°	22° and 26°	15.1 and up	24° and 28°	26° and 30°

c. We recommend that a lamp of 100 watts be specified for use in tintometer.

d. We recommend that further work on improvement of refining procedure be carried out and that the committee should have at least one meeting at a central point, where all members will refine a sample of crude oil simultaneously, in the endeavor to locate differences in procedure, which cannot be brought out by correspondence.

e. We recommend a re-writing of rule No. 272, beginning at Section 5, in order to consolidate sections 5 and 6 and to conform to the recommendations of the Chemists Committee of the Interstate Association, at its special meeting in Memphis, Dec. 28, 1925, as per copy herewith.

NOTE—R. H. Fash, a member of the Refining Committee, desires to withhold his approval of recommendations b. c and e in the above report.

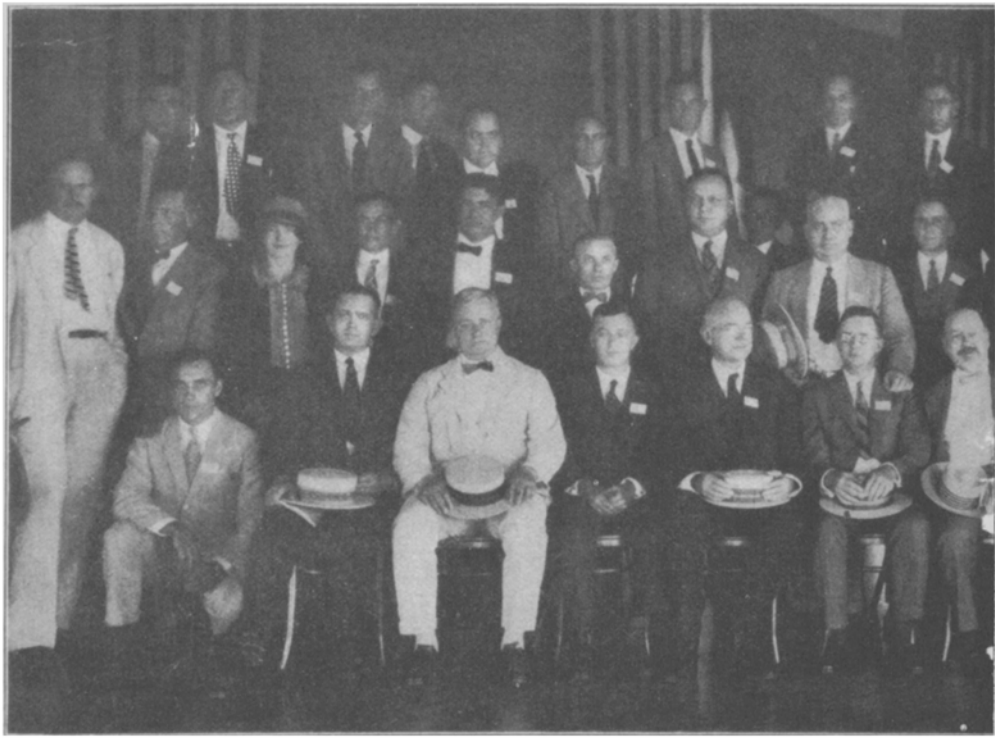
C. B. CLUFF, Chairman.

PROPOSED REVISION OF RULE 272, SECTIONS 5, 6, 7, 8

Section 5—REFINING LOSS

(a) General Directions

All oils with 2 per cent F.F.A. or less may be refined with any amount of sodium hydroxide solution necessary to neutralize the F.F.A. plus a reasonable excess, provided the total sodium hydroxide does not exceed 926 per cent for hydraulic pressed oils or 1.208 per cent for cold pressed or expeller oils. On all oils, three refinings shall be made, using different strengths or amounts of sodium hydroxide solution, provided the maximum amount equal to $\frac{\text{F.F.A.}}{5.7} + 0.575$ of sodium hydroxide is not exceeded, and that one of the tests be made with this maximum amount. On any cold pressed or expeller oil, the same procedure shall be carried out, allowing, however, a maximum amount equal to $\frac{\text{F.F.A.}}{4.365} + 0.75$ in per cent of sodium hydroxide, and making at least one test with 20° Be sodium hydroxide solution.



A Group of Delegates and Guests at the Seventeenth Annual Convention of

(b) Choice of Lye

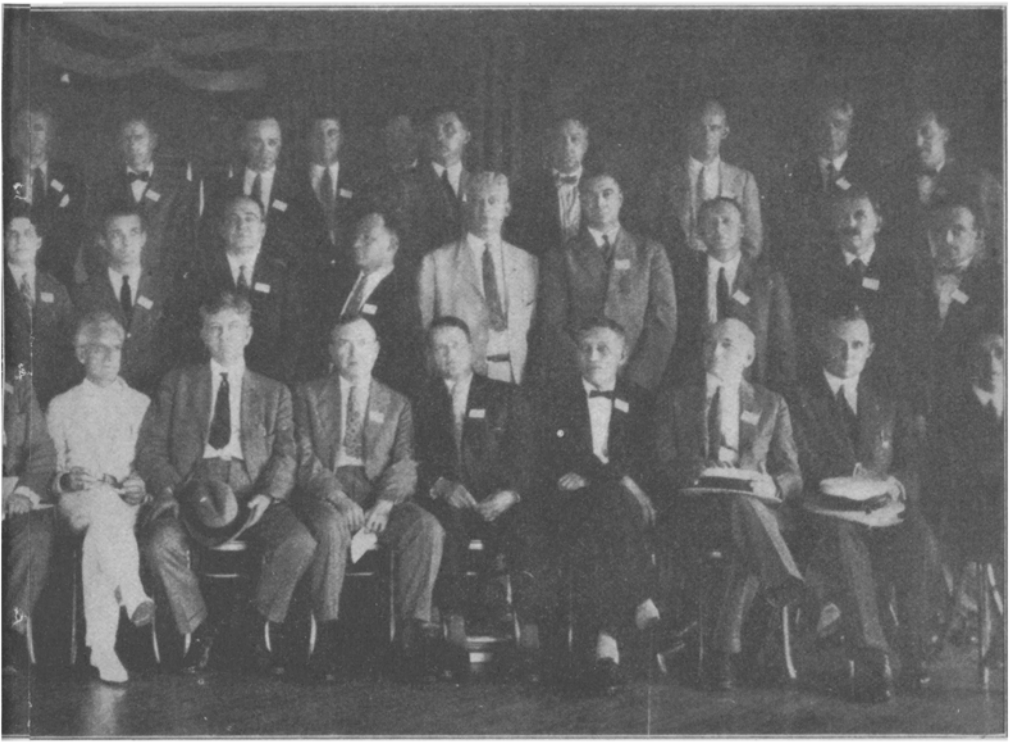
The strengths of lye to be used on oils and various F.F.A. are:

F. F. A. %	Hydraulic Pressed Oils—Lye	Expeller Oils —Lye	F. F. A. %	Hydraulic Pressed Oils—Lye	Expeller Oils —Lye
3.1 or less	12° and 16°	16° and 20°	7.1-10.0	20° and 24°	24° and 28°
3.1- 5.0	16° and 20°	20° and 24°	10.1-15.0	22° and 26°	26° and 30°
5.1- 7.0	18° and 22°	22° and 26°	15.1 and up	24° and 28°	26° and 30°

Tables showing maximum percentage of lye are appended for ready reference. (Tables to be same as present tables on pages 69 and 70, but with addition of amounts of lye for 2.0 and 2.5% F.F.A., and columns for 26° and 28° lye for hydraulic oils, and 16° lye for expeller oils.)

(c) Apparatus

Scales, 1000 grams capacity and sensitive to ½ gram. Weights, 500 grams to ½ gram. Refining cups—seamless or enameled iron cups, 4½ inches in diameter and four inches deep. Refining apparatus, a mechanical stirrer having flat (not twisted) T-shaped paddles ½ inch wide, with crossarm 3½ inches over all, set at right angles to the shaft of the paddle. Water bath with thermometer, suitably arranged to hold cups used in tests.



the American Oil Chemists Society, Hotel Roosevelt, New Orleans, May, 1926.

(d) Preparation of Sodium Hydroxide Solution

Sodium hydroxide solutions of 12, 16, 18, 20, 22, 24, 26, 28 and 30° Be[1] at 20°C shall be prepared from a saturated solution of the pure material (77° per cent Na_2O).

"Sodium Hydroxide Table"

(Table to be same as present table on page 67 of rules, 1925 edition)

(e) Refining Process

Place 500 grams of the thoroughly mixed sample of crude oil in a tared refining cup. Adjust the temperature of the oil to 24-27 degrees C., being sure that the oil contains no air bubbles or foam. Stir continuously at the rate of 250 r.p.m. plus or minus 10. Add the proper amount of NaOH solution to the oil and stir exactly five minutes for oils with F.F.A. not over 3.0 per cent, and 15 minutes for oils with F.F.A. over 3.0 per cent, (in the case of any cold pressed oil, stirring should not be less than thirty minutes) as soon as the lye has been added. Then begin heating the oil at a uniform rate and continue heating until a temperature of 60°C is reached. The rate of heating should be such that this temperature is reached in as nearly as possible fifteen minutes. Stir constantly and continue stirring until a complete separation of oil and soap stock results. After heating is begun, the rate of stirring may be reduced, if desired. At times, to effect this separation, it will be necessary to attain a temperature of 65°C. Place in a water bath at 45°C. for three hours. Allow to stand over night or cool thoroughly so that the soap stock will become hard and firm. Decant the refined oil into a tared refining cup and drain the soap stock for twenty minutes. Weigh both oil and soap stock cups and contents. If necessary, melt the soap stock on the steam bath and correct for any oil recovered.

(f) 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.

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

Note: It is the intent of these rules to so refine the oil, using the maximum permitted amount of sodium hydroxide solution, as to produce a prime color when possible to do so.

In case the above limit of Sodium Hydroxide produces worse than a prime color with a loss less than 9 per cent, then a greater amount of Sodium Hydroxide can be used in order to produce a prime color if possible, provided the refining loss does not exceed 9 per cent, and in such case if a prime color is produced, the oil shall be graded a prime as to color.

No oil shall be rejected unless treated with three or more sodium

hydroxide solutions of the above Beaume; nor shall any claim for excessive refining loss be established if it can be proven that the color can be made with a smaller loss than produced with the maximum amount of sodium hydroxide allowed; nor shall any claim for deficiency in color be established if it can be proven that the color can be obtained without exceeding 9 per cent refining loss with maximum amount of caustic soda allowed.

When it is not possible to produce a prime color with the maximum sodium hydroxide solution specified, and when two or more tests on the same oil show varying results in color, that test which produces the best color must be reported, provided losses are the same, or approximately so.

Illustrations: (a) (1) Loss 12 per cent Color Red 10.
(2) Loss 12.3 per cent. Color Red 9.0 Report

However, when two or more tests are made on the same oil, the one producing the best color with maximum sodium hydroxide solution specified shall be used as the basis of comparison, and when another test produces an oil which is not more than 10 per cent darker in color than this basis test, and with a reduction in loss equal to, or greater than the numerical difference in color reading, then the latter result is the one to be reported.

Illustration: (b) (1) Loss 12 per cent. Color Red 10.
(2) Loss 10.8 per cent. Color Red 11 Report.

Section 6—COLOR

(a) Oil refined as above must be clarified before reading the color, by filtering through white filter paper at a temperature of 20° to 24°C without the application of heat or addition of any material to facilitate filration.

Report color of refined oil in terms of Lovibond's equivalent color scale using yellow and red glasses.

In every case where color standards are mentioned only the two standard glasses specified in the rule must be used to determine the grade.

(b) The color examination shall be made as follows: Place the oil in a white polished-bottomed bottle or cylinder of such depth that a column of oil 5¼ inches can be accurately measured therein. Such bottle or cylinder to be of not less than ¾ inch internal diameter. The reading shall be made by artificial light in a form of tintometer approved by the Chemist Committee, and at a temperature of 21 degrees C. If the oil is of deeper color than the combined standard glasses 35 yellow, 7.6 red, it shall not be classed as prime.

(c) Color Readings—

1. In reading color of 12 red or over, a tolerance of plus or minus 5 per cent of the actual color reading will be allowed *in establishing a grade of oil as defined in the rules.*

Illustration: A reading of 11.4 to 12.6 to be reported as 12 Red, 15.2 to 16.8 as 16 Red; and 19 to 21 as 20 Red.

2. No colors shall be read or reported darker than 50 Red. Any color apparently darker than 50 Red shall be reported "too dark to read," or "50 plus."

Section 7—FLAVOR

Oil which is not rancid, musty, sour or bitter, and does not have a flavor foreign to Cotton Seed Oil made from sound seed shall be graded as Prime in flaver. Only three designations of flavor will be recognized, Prime, Slightly Off and Off.

C. B. CLUFF, Chairman