Association Adopts Chemical Methods

Chemists' Committee Report Embodies New and Revised Analytical Procedure

THE recommendations of the Chemists' Committee of the Interstate Cottonseed Crushers' Association were adopted as submitted by the Memphis Convention of the Association practically without dissent.

The new methods and procedure include rigid rules for the refining loss test and a tentative method for the determination of free fatty acid in oil extracted from seed.

The most radical changes, however, are found in the new Rule 272, Section 7, which dictates that all oil which contains more than 2.50% free fatty acids and less than 4.00% *must* be classed Slightly Off in Flavor, and all oil which contains over 4.00% free fatty acids *must* be classed Off in Flavor, regardless of the actual flavor of the oil.

As the trading rules provide penalties in oil settlements for Slightly Off and Off Flavor, the net result should be to encourage the millers to greater efforts to produce oil of good quality and low free fatty acid content. It is a generally acknowledged fact that in crude oils increased free fatty acid content is nearly always co-incident with deteriorating flavor, so the new rule has a sound basis for its enactment. The indicated relief for the opponents of this rule is the improvement of methods in their seed-handling and pressing operations, so as to produce oil of superior quality.

The Chemists' Committee report as adopted follows:

Recommendations of Chemists' Committee

Insert new Rule 264, Chapter XII, Page 57, as follows:

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	Proceedure
Prime, or better	Over 9%	See par. (b)
Darker than Prime	9% or less	See par. (c)
Darker than Prime	Over 9%	See par. (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% 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 necessary 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 270-A

Rule 270-A, Page 59—Add new Section 3, as follows:

Rule 270-A, Sec. 3—Tentative Method for:

Determination of Free Fatty Acid in Extracted Oil From Seed

At least 100 Grams of the well mixed sample of seed is heated from 30-45 minutes at from 100-105 Degrees Centigrade and cooled. The meats are then separated by any laboratory huller or mill that will approximate factory conditions and ground sufficiently to pass a 1½ MM sieve. Not less than 10 Grams of the thoroughly mixed meats are extracted by cold percolation with gasoline boiling below 70 degrees—the gasoline evaporated off and the oil weighed. 30 cc of neutralized denatured alcohol are added and the Free Fatty Acid of the oil is titrated with a Standard Caustic using Alkali Blue as an indicator. The Free Fatty Acid is calculated by the formula:

$$\frac{28.2 \times \text{normality of Alkali} \times \text{cc used}}{\text{Weight of Oil}} = \% \text{ F.F.A.}$$

Notes

The gasoline percolation should be continued sufficient time to give at least 2 Grams of oil.

The addition of a small amount of gasoline to the flask after the alcohol has been added before titrating makes the end point shaper.

Tenth Normal Caustic Soda is preferable for low Fatty Acid oils but for oils above 5 per cent, quarter or fifth Normal is preferable.

In case it is desired to make the determination where an analytical balance is not available, extract a larger quantity of meats and, after evaporating all the gasoline from the oil, pipette 7.05 Grams and titrate with quarter Normal Caustic. The reading in this case is percentage directly.

Rule 270-B—Change Section 2 to read as follows:

Section 2. Oil. Place 5 grams of the Hulls, free from Meats and Seed, in a suitable extraction apparatus and extract for three hours with petrolic ether (See Specifications Rule 271). Evaporate the ex-

tract, at the temperature of boiling water, to constant weight and report as per cent of Oil.

Rule 271, Section 3.

Eliminate "boiling below 70 degrees C," in third line and substitute (See Specifications Below):

Whenever words quoted above appear in rules, substitute (See Specifications, Rule 271, Sec. 3).

Add second paragraph to Section 3, as follows:

Specifications for petrolic ether:
Distilling between 30-70.....90%
Initial B. P. .....30-40 Deg. C.
End Point not over.....85 Deg. C.
N. V. M. (on Steam Bath) . .003%
Specific Gravity ..... .630-.650

Rewrite Rule 272, as follows: (Secs. 1, 2, 3, 4 and 5)

Rule 272—CRUDE OILS (Note —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 procelain 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—Weigh 7.05 gr. of well mixed oil into a 4 oz. bottle or 250 CC flask, add 50 CC of denatured alcohol (Formula 30) containing .05% of phenolphthalein and a sufficient amount of NaOH solution to give the alcohol a faint pink color. The alcohol must be made faintly pink before adding to the oil in the bottle or flask. Titrate with .25 N NaOH solution until a permanent faint pink color is produced, which cannot be removed by violent shaking of the mixture. Report as per cent free fatty acid oleic, the number of cubic centimeters of .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 plus or minus 10 R. P. M. and the speed of paddles in the hot water bath must be 70 plus or minus 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 maintain 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 measurable amounts 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 by specific gravity tests. The important point is to know the exact content of sodium hydroxide rather than the exact specific gravity or Beaumé. Solutions of only the following strengths shall be used and they must be within the limits shown.

Sodium Hydroxide Table

Nominal Degrees Beaumé 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:

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

Per Cent F. F. A. Oil	Hydraulic	Expeller Oil
1.5 or less	10° and 14° B'me.	12° and 18° B'me.
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°

For hydraulic or hot pressed oils:

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

For expeller or cold pressed oils:

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

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 above shows the maximum precentage 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+or—5 R.P.M. for eight minutes. Temperature of oil must

For Hydraulic or Hot Pressed Oils Per Cent Lye of Varying Beaume at 15° C Containing Maximum NaOH								For Expeller or Cold Pressed Oils Per Cent Lye of Varying Beaume 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.

% FFA	Max. NaOH	10°	Max. NaOH	12°	18°
1.5	.83	12.6	1.11	13.9	8.8

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 temperature 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° plus or minus 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 substract 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.

Rule 272, Section 6. Same as present.

Rule 272, Section 7. Entire section eliminated. Substitute following:

Rule 272, Section 7. Flavor. Oil which is not decidedly rancid, musty, sour or bitter, and does not have a flavor foreign to cottonseed oil made from sound seed, shall be graded as Prime in flavor if free fatty acids does not exceed 2.50%.

Slightly Off. Oil which is not decidedly rancid, musty, sour or bitter and exceeds 2.50% free fatty acids, but does not exceed 4.00% free fatty acids, shall be graded Slightly Off in flavor.

Off. Oil which is decidedly rancid, musty, sour or bitter and/or oil which exceeds 4.00% in free fatty acids, shall be graded Off in flavor.

Rule 277, Section 2, Page 75, Five lines from bottom—Eliminate boiling below 70 degrees C., and substitute:

(See specifications Rule 271.)

Personal Comment, cont. from p. 248

ness for some weeks, he made a special trip to Memphis to attend the Convention, although this necessitated his returning to California to complete his business there before going back to his home at Cincinnati. Mrs. Cluff accompanied Mr. Cluff on his California trip.

The croix-de-guerre with all the palms should be awarded G. Owen Daniel as the most entertaining host of Memphis. Mr. and Mrs. Daniel, assisted by their daughter and son-in-law, Mr. and Mrs. Sidney Allen, kept open house for their friends all during the convention and contributed largely to the success of all social activities.

J. P. "Jack" Harris, of Chicago Chemical Engineer and Carbon Specialist, was present with his smile ready to expound the merits of "Black" to all comers.