were run as indicated in Table II. The flavor of all samples was "Prime" except where noted.

A peculiar condition arose in the last two refinings for specimens C & D. A gelatinous mass having a darker red color than the oil formed in the refined oil which disappeared upon warming. The warmed oil was passed through a single filter paper, but the resulting oil appeared so cloudy in the color tube that a true reading could not be obtained. Upon filtering the oil at room temperature through two papers the gel was retained, whereupon the clear filtrate was read at 35 Yellow, 7.8 Red. The iodine number of the gel thus obtained was 107.0 as compared to 110.0 of the filtered oil. From the appearance of the gel and its lower iodine number we believe it to be a polymerized body probably resulting from a catalytic action of the copper on the oil. The gelatinous mass, however, did not appear in the filtered bleached oil, which of course was filtered hot. Whether this material was rendered more fluid again or whether the earth prevented its passing through the filter, we could not determine.

The interior of both containers C & D was well coated with a dark green copper soap insoluble in the crude but readily soluble in both chloroform and gasoline. This, no doubt, explains the reduction of the free fatty acid in these two samples. From the work of King, Roschen and Irwin (1) we attribute the early development of rancidity of both these oils to their contact with the copper. A decided rancidity was later found in the sample stored in container "F" which is composed of about 30 per cent copper. The only other case of rancidity was in the oil stored in container "A," although this alloy is supposedly free of copper. The remainder of the samples, while not quite as sweet as at the beginning, still in our opinion would be classed as "Prime" under the refining rules. Examination of the containers after the tests revealed no corrosive action except in containers C & D.

From the series of tests run no definite conclusions can be drawn except that in the case of copper and copper bearing alloys, neither should be used in contact with crude cotton seed oil.

In the use of Hastelloy "A" a slight increase of color over the control was noted with an attending increase in rancidity, but no decided increase in refining loss was detected.

Catalysis is suspected in the formation of a gel in the use of Deoxidized copper and Everdur "A" metals, the gel having the appearance and test of a polymerized oil.

In conclusion we express our sincere appreciation to the following manufacturers who have made possible these tests by furnishing and fabricating the necessary containers:

The Allegheny Steel Co., Brackenridge, Pa.

The Aluminum Cooking Utensil Co., New Kensington, Pa.

The American Brass Co., Waterbury, Conn.

The American Rolling Mill Co., Middletown, Ohio.

Haynes Stellite Co., Kokomo, Ind.

The International Nickel Co., Inc., New York, N. Y.

Wilson & Bennett Mfg. Co., Chicago, Ill.

Wyatt Metal & Boiler Works, Houston, Texas.

References:

(1) "The Accelerating Effect of Metals on the Development of Peroxides in Oils & Fats," by A. E. King, H. L. Roschen and W. H. Irwin; Oil & Soap, Vol. X, No. 11, p. 204.

REPORT OF

THE REFINING COMMITTEE

FOR THE SEASON OF 1934-1935

THE Refining Committee for the season 1934-1935 begs to submit the following report:

Tentative procedures for refining Peanut, Cocoanut and Corn Oil have been outlined and in use for a number of years. Your Committee recommends that the methods for refining these oils be now adopted as official methods of this Society. The methods are now designated as "Tentative" and are printed as part of our official methods.

There is a correction to be made in the printed method for Cocoanut Oil. In line with the results obtained and reported by Mr. Cluff on some fifty samples of crude cocoanut oil the amount of salt to be used shall be 0.1% for each 1.0% F.F.A. and the amount of lye reduced from 1.25 times the F.F.A. to 1.10 times the F.F.A. Temperature conditions have also been changed

The attention of the Committee has been directed to the variation in the characteristics of different lots of crude soya bean oil, some of which do not refine satisfactorily by the proposed method. We recommend that the work on methods for refining crude soya bean oil be continued next year.

There was a suggestion made a few years ago that on some cold pressed cottonseed oils better results were obtained with 22° Be. lye than with 20° Be. The Committee has been unable to obtain more than one sample of this type and we, therefore, recommend that since such types of cold pressed oil are so rare, they do not warrant an investigation by the Refining Committee.

Your Committee has not tested any additional brands of filter papers during the past season, but still believes that development of specifications for a suitable paper which will make it unnecessary to mention manufacturers' names or brands in the official methods is desirable.

Your Committee recommended to President Hutchins that the investigation pending of a method for determining traces of soap in refined cocoanut oil should be broadened to include all refined oils and a special committee be appointed to undertake this investigation. President Hutchins acted favorably on this suggestion and we believe a report on this subject will be submitted to

the Society in the very near future.

Your Committee undertook to rewrite and confine refining procedures for all oils in one method rather than having separate procedures for the different oils. A copy of these procedures is attached and we recommend the adoption of this in place of the now existing one.

We are attaching a tabulation of refining conditions for all oils which we recommend be printed as part of the refining methods. From this table the required conditions for each oil will be more easily found than by reading through the written descriptions.

While this Committee has primarily been interested in domestic oils, new problems have been confronting the oil chemist in handling various other oils from time to time. Your Committee suggests that the membership consider the advisability of developing laboratory procedures for handling the various foreign oils now available in this country as well as some of the lesser known domestic vegetable and marine animal oils.

C. B. CLUFF,
Chairman
A. W. PUTLAND,
Vice-Chairman
R. H. FASH
R. C. HATTER
J. J. GANUCHEAU
R. R. BARROW
A. GUDHEIM
W. R. STRYKER
B. H. THURMAN.

PROCEDURES

CRUDE OILS—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 20°C. before attempting to mix. In the case of cocoanut oil, immerse the entire can in a water bath maintained at about 38°C. for several hours, or until the oil is completely melted. After shaking, examine the bottom of the can for sediment by feeling with a glass rod. If any is found, it must be completely removed (cutting the can open if necessary), and thoroughly incorporated with the oil before proceeding with the analysis.

Section 5: *Refining Crude Oils:*

(a) Apparatus:

Scales — Capacity 1000 grams, sensitive to ½ gram.

Weights—½ to 500 grams.

Refining Cups—(Same).

Agitator and Water Baths — (Same as now written).

(b) Sodium Hydroxide Solutions—(Same as now written).

(c) Refining Procedure: Place 500 grams of the thoroughly mixed sample of crude oil in a tared refining cup, and settle, if necessary, to allow air to escape. Adjust the temperature of the crude oil and water bath to 20°-24°C. (or in the case of Cocoanut Oil to 30°-35°C.); using ice to cool the water if necessary. With the agitator running 250 R.P.M. ± 10, add, as quickly as possible, the proper amount of sodium hydroxide solution to the oil. (In the case of Cocoanut Oil, add 0.1% dry table salt for each 1.0% F.F.A. present in the crude oil before the addition of the caustic solution.)

Continue stirring for exactly fifteen minutes from the time the sodium hydroxide solution was added (forty-five minutes for cottonseed oil designated as cold pressed, expeller or whole pressed, or slow breaking types, thirty minutes for peanut oil and five minutes for cocoanut oil).

Then immediately transfer to the 65°C. bath (50°-53°C. for cocoanut oil) and stir at 70 ± 5 R.P.M. for exactly twelve minutes (twenty minutes for designated slow breaking oils and five minutes for cocoanut oil). Temperature of oil must then be 60°-65°C. (48°-52°C. for cocoanut oil), adjusting the temperature of the water bath, if necessary, within the limits specified to obtain this final oil temperature.

Allow to settle in a water bath at 65°C. (50°-53°C. for cocoanut oil) for one hour. Cool by setting in a cold water bath at 20°-24°C. (26°-30°C. for cocoanut oil in order to keep the oil liquid) 30 minutes and hold at this temperature for at least one hour additional, preferably overnight.

Weigh the refining cup and contents and deduct this weight from the total weight of crude oil and cup to obtain loss by evaporation. Decant the refined oil into a tared refining cup and drain the soap stock for 30 minutes. Weigh the refined oil and filter through white filter paper (Eaton & Dykeman No. 617, Reeve-Angel No. 230, or such other brand as is approved).

In the case of cold pressed and/or whole pressed oils, add 0.5 gram of Filter-Cel (may be obtained from Secretary of A.O.C.S.) to the decanted refined oil and agitate for five minutes in bleaching machine at 250 R.P.M. at room temperature to absorb colloidal matter before filtering. This filtered oil is to be used for determination of grade.

Weigh the soap stock cup with its contents. Then melt the soap stock by setting the cup in a water bath maintained at 75 ± 2°C., without stirring, for thirty minutes; cool in a cold water bath for fifteen minutes or until thoroughly chilled, then decant into a weighed container any additional oil thus recovered, draining fifteen minutes. In the case of Soya Bean Oil, the soap stock may be hardened by chilling in ice water, if necessary, to permit draining the oil from the soap stock. Weigh the oil recovered from melting the soap stock separately and add this weight to the weight of refined oil first obtained and subtract it from the weight of soap stock first obtained. Repeat the remelting, cooling, and decanting as above outlined, until the recovered oil from the last remelting does not amount to more than 1.5 grams. In the cases where the soap stock does not solidify, the last small portion of oil can best be removed from the surface of the foots by means of a pipette.

Note: Any foots floating on the surface of the oil or decanted with the oil must be recovered and added to the main body of foots before weighing. This can conveniently be accomplished by decanting the oil through a common tea strainer with a fine mesh screen which will retain the floating foots so that it can be returned to the main body of soap stock.

(d) Calculations: Determine the refining loss by two methods of calculations, the results of which should check within one-quarter of one per cent. Report the average of the two methods of calculations: Method No. 1: Weight of crude oil minus weight of refined oil gives the refining loss. Method No. 2: Weight of soap stock plus loss in evaporation, minus weight of sodium hydroxide solution (include weight of salt used in the case of cocoanut oil) used gives refining loss.

Section 6: Strength and Amount of Sodium Hydroxide Solutions:

(a) Crude Cottonseed oil—The maximum amount of sodium hy-

REFINING CONDITIONS

	Hydraulic	C. S. Cold Pr.	C. S. Slow break	Peanut	Soya Bean*	Corn	Cocoanut
Refining:							
Lye	*11A	*11A	*11A	*16D	*16D	*16D	*16D
Tempt. start	20–24° C.	20–24° C.	20–24° C.	20–24° C.	20–24° C.	20–24° C.	30–35° C.
Speed	250±10	250±10	250±10	250±10	250±10	250±10	250±10
Time cold	15′	45′	45′	30′	15′	15′	5′
Temp. hot bath	63–67° C.	63–67° C.	6°–67° C.	65–67° C.	63–67° C.	63–67° C.	50–53° C.
Time hot	12′	12′	20′	12′	12′	12′	5′
Final oil temp.	60–65° C.	60–65° C.	60–65° C.	60–65° C.	60–65° C.	60–65° C.	45–50° C.
Temp. cooling bath	20–24° C.	20–24° C.	20–24° C.	20–24° C.	20–24° C.	20–24° C.	26–30° C.
Time cooling in bath	30′	30′	30′	30′	30′	30′	30′
Addn'l cooling and settling	1–16 hrs.	1–16 hrs.	1–16 hrs.	1–16 hrs.	1–16 hrs.	1–16 hrs.	1–16 hrs.
Drain soap stock	30′	30′	30′	30′	30′	30′	30′
Clarification:							
Filter-Cel	0	.5 gm.	0	0	0	0	0
Temp.	0	Room	0	0	0	0	0
Speed	0	250±10	0	0	0	0	0
Remelting:							
Temp.	75±2	75±2	75±2	75±2	75±2	75±2	50±2
Agitation	none	none	none	none	none	none	none
Time	"0"	30′	30′	30′	30′	30′	30′
Temp. cooling bath	20–24° C.	20–24° C.	20–24° C.	20–24° C.	ice water	ice water	26–30° C.
Time	15′ or till solid	15′ or till solid	15′ or till solid	15′ or till solid	15′ or till solid	15′ or till solid	15′ or till solid
Drain	15′	15′	15′	15′	15′	15′	15′

*Tentative. **For Cocoanut Oil, add 0.1% table salt for each 1.0% F.F.A. before adding lye.
*Lefax Methods.

droxide allowable for refining shall be calculated from the following formula for hydraulic or hot pressed oils with F.F.A. above 1.5%; and for hydraulic or hot pressed oils with F.F.A. less than 1.5%, only 80% of the amount calculated from this formula shall be used.

$$\frac{\text{F.F.A.}}{5.2} + .54 = \text{maximum sodium hydroxide}$$

The maximum amount of sodium hydroxide allowable for refining expeller, cold pressed, or whole pressed oil shall be calculated from the following formula:

$$\frac{\text{F.F.A.}}{4.365} + .77 = \text{maximum sodium hydroxide}$$

The strengths of sodium hydroxide solutions (expressed in Baume degrees) to be used for refining crude cottonseed oils of various F.F.A. shall be as follows:

Per Cent F.F.A.	Hydraulic or Hot Pressed Oils	Cold Pressed, Expeller, or Whole Pressed Oils
1.5% or less	12° and 14°	16° and 20°
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 20°	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°

Tables follow which show the required percentages of each strength of lye to be used.

(b) Crude Peanut Oil: On crude peanut oils three refinings shall be made, using the following strengths and amounts of sodium hydroxide solution:

Crude Peanut Oils with F.F.A. not exceeding 3%—

12° sodium hydroxide solution using 60% of the maximum amount calculated from the formula given for hydraulic pressed crude cottonseed oil.

16° sodium hydroxide solution, making one test with 60% and one test with 80% of the maximum amount calculated from the formula given for hydraulic pressed crude cottonseed oil.

On Crude Peanut Oils with F.F.A. exceeding 3%—

16° sodium hydroxide solution using 60% of the maximum amount calculated from the formula given for hydraulic pressed crude cottonseed oil.

One test using 16° and one test using 20° sodium hydroxide solution, using in both tests 80% of the maximum amount calculated from the formula given for hydraulic pressed crude cottonseed oil.

Tables follow which show the required percentages of each strength of lye to be used:

(c) Crude Cocoanut Oil: The strength of sodium hydroxide solution to be used in refining crude oils shall be 20° in all cases. The amount to be used shall be calculated from the following formula:

F.F.A. (as Oleic) × 1.10 = % of 20° sodium hydroxide solution.

(d) Crude Corn Oil: The strength of sodium hydroxide solution to be used in refining crude corn oils shall be 16° in all cases. Two tests shall be made, using respectively one-half and two-thirds of the maximum amount calculated from the formula given for hydraulic pressed crude cottonseed oil.

MISCELLANEOUS OILS:

Section 1: Tentative method for refining Crude Soya Bean Oil: The apparatus and general procedure shall be the same as prescribed for hydraulic pressed crude cottonseed oil, with the following exceptions: Strength of lye shall be 14° in all cases, and two tests shall be made, using respectively one-half and two-thirds of the maximum amount permitted for hydraulic crude cottonseed oil having the same F.F.A. The soap stock may be hardened by chilling in ice water, if necessary, to permit draining the oil from the soap stock.

SECRETARY-TREASURER'S ANNUAL REPORT

May 1st, 1934 Through April 30th, 1935

FINANCIAL STATEMENT

RECEIPTS

Office of the Secretary-Treasurer.

Cash balance May 1, 1934—		
Checks on hand	$	1.00
In Petty Cash Drawer		1.50
In Savings Acc't, Hibernia Nat'l Bk. (Liquid)		50.93
In Savings Acc't, National Bk. of Commerce in N. O. (Liquid)		51.00
In Checking Acc't, National Bk. of Commerce in N. O. (Liquid)		100.26
In Savings Acc't, Whitney Nat'l Bk. (Liquid)		539.85
In Checking Acc't, Whitney Nat'l Bk. (Liquid)		717.14
		$1,461.68