Report of Refining Committee for Year 1932-1933

By C. B. CLUFF, Chairman

THE Refining Committee reports as follows on the various problems studied during the past year:

Coconut Oil—

Tests were undertaken to improve the present tentative method, particularly by reducing the amount of caustic and time of agitation. The results indicate that the following changes could advantageously be made.

Reduce the amount of 20° lye from 1.25 times the free fatty acid to 1.10 times the free fatty acid.

Change the amount of salt from 1 per cent as now specified for all cases to an amount equal to .1 per cent for each 1.0 per cent free fatty acid.

Change the time and temperature of agitation from 15 minutes cold and 12 minutes hot as at present, to 5 minutes at 30-35° C. and 5 minutes in bath at 50-53° C. Loss and color were found to be at least equally good as by the present method, and there was practically no separation of excess water solution or lye.

While further experience is desired before recommending a change in the rule, we recommend that the new procedure be tried out by all interested in coconut oil.

Soya Bean Oil—

It was thought that longer agitation would be beneficial. Three lots of soya bean oil were obtained as follows:

1. From A. E. Staley Company—Free fatty acid .5%
2. Buckeye Cotton Oil Co...Free fatty acid..... .7%
3. Allied Mills—Free fatty acid..... 1.1%

Numerous variations in time of agitation, both cold and hot, were tried but without much improvement in any case, but it was found that the three oils were of very different characteristics, and it appears very difficult to make any single change that would apply satisfactorily to all types of soya bean oil.

We accordingly recommended no change in the existing method.

Stronger Lye for Cold Pressed Oil—

It has been suggested that 22° lye gives better results than 20° lye on some cold pressed oils. One lot of oil tested confirmed this point, but it was impossible to obtain other lots for investigation.

We accordingly can make no recommendation for a change in the present rule.

Reduced Caustic for Cottonseed Oils with 1.5 Per Cent F. F. A. or Less—

The committee was asked to determine whether satisfactory refinings for settlement purposes might not be obtained with lower losses by using less caustic than the 80 per cent of the formula now specified for such oils. One hundred and seventy-two samples of crude cottonseed oil with low free fatty acid were tested and reported by five members of the committee, using on each oil four different amounts of caustic, that is, 60 per cent, 70 per cent, 80 per cent and 100 per cent of that calculated from the formula. Only 80 per cent is specified in the present rule for oils of this character.

The results showed that only a small proportion of oils refined satisfactorily with 60 per cent of the formula. We found that with 70 per cent of the formula, 47 per cent of the samples gave nearly the same color as obtained with the full 100 per cent of the formula, and furthermore, that 80 per cent of the samples gave nearly as good color as with the present requirement, using 80 per cent of the formula. Our criterion of a satisfactory refining in all these cases was that the color must be within 10 per cent of that obtained with the standard.

On the question of reducing the required lye from 80 per cent to 70 per cent of the formula for refining these oils, or possibly retaining the present 80 per cent and making an additional test with 70 per cent, the committee was divided. Two members favor continuing the present requirements of 80 per cent of the formula and adding an additional test with 70 per cent of the formula. Three members of the committee, constituting a majority, oppose the use of less than the present requirement of 80 per cent. Consequently, the committee is not recommending a change in the present rule.

Filter Paper for Refined Oils—

It has frequently been found that the color of a freshly refined oil will differ according to the kind or grade of filter paper used for filtering same. This matter was investigated partially several years ago. Further work was done during the past year. It was found that in some cases differences as great as 1.5 red were obtained on the same oil when filtered through the standard Whatman's No. 40 filter paper or through some other paper. Differences as great as .5 red were very common. These differences are especially great on cold pressed oils or on hydraulic pressed oils which are not refined with the maximum amount of lye. Apparently, the difference is due to a small amount of colloidal matter which is retained by the highest grade filter papers but passes through the cheaper grades. Differences of 30 red occurred on some cold pressed oils.

Examination of some eighteen different brands and grades of filter paper showed that about half of these, especially the cheaper grades, showed numerous minute holes when the paper was held up to the light. A second group of papers showed but few of these holes, while a third group showed none.

Further tests showed that by using a small amount of filter cel the colloidal matter was absorbed from any of the oils, so that they would then show the same color after filtering through any of the ordinary grades of paper as was obtained by filtering through the Whatman No. 40 paper.

The final results of this work was that two grades of paper were found to be very nearly equal to Whatman No. 40, but very much cheaper than this, and at the same time they are vastly superior to some of the cheaper grades that have heretofore been in common use. Secondly, the use of filter cel on refined oil from cold pressed crudes is the only safe way to assure complete removal of the colloidal matter and thus obtain the true color of the refined oil.

The committee feels that it is important to remove this variable condition from our refining procedure and require all laboratories to use the same standard paper or the same standard procedure, and accordingly we recommend that the refining method be changed to require the refined oil to be filtered through either Eaton & Dikeman No. 617 or Reeve-Angel No. 230 paper. Other grades not to be used until and unless approved by the American Oil Chemists' Society. Also in the case of cold pressed oils, .5 gram filter cel is to be added to the refined oil and agitated for 5 minutes before filtering.

Summary—

Our recommendations on these points are as follows:

Coconut Oil—No change, but the details outlined

should be tried out by members interested during the coming year.

Soya Bean Oil—No change.

Cold Pressed Oil—No change.

Reduced Caustic for Cottonseed Oils with 1.5 per cent F. F. A. or Less—No change.

Filter Paper—The refining method to be changed as follows, referring to the method as published in N. C. P. A. Rules 1932-33, page 148, under Refining Procedure, line 24. Eliminate the sentence beginning, "This oil is to be filtered" and substitute the following two sentences:

In case of expeller oils only, add 0.5 gram filtered (obtained from Secretary of the A. O. C. S.) and agitate in bleaching machine for 5 minutes at 250 r.p.m. at room temperature, to absorb colloidal matter. This oil is to be filtered through white filter paper of an approved brand (see below) and used for determination of grade.

Also, add the following note after the present note at the bottom of page 148:

Note: Approved brands of filter paper are
Eaton & Dikeman No. 617
Reeve-Angel No. 230.

Other brands not to be used unless first approved by the American Oil Chemists' Society.

C. B. CLUFF, *Chairman.*
E. R. BARROW,
R. H. FASH,
J. J. GANUCHEAU,
R. C. HATTER.

Report of Committee on Revision of Methods

The changes and additions in the methods made at the Fall meeting, 1931, and the Spring meeting, 1932, made necessary the reprinting of about 18 pages of our methods at a total cost of $69.00.

It was decided not to attempt to print the methods of the Soap Section until the Committees of that Section have had an opporunity to review the methods and get them in shape for publication. It is hoped that these methods will be ready by the time the next revisions are made, in July.

The committee wishes to urge upon the chemists interested in oils, fats and soaps, to place their names on the permanent list for the revisions as they are printed each year in order that there may be no confusion in the various laboratories due to the use of methods which are obsolete. The cost of these revisions does not exceed 50c.

W. H. IRWIN, *Chairman.*

Duty-Free Exportation Decreed for Animal Oils, Carbonic Acid Gas and Pastilles of Chicle

A Mexican presidential decree, published and effective February 10, exempts from export duty animal oils in any container (export tariff item 48, formerly dutiable at 0.01 peso per gross kilo), and creates two new items in the export tariff exempting from export duty carbonic acid gas (export tariff item 264-A) and pastilles of chicle, even when containing medicinal substances (export tariff item 282-A).

Research on Bleaching of Palm Oil With Benzoyl Peroxide*

By W. H. Dickhart

PALM oil was chosen for the subject because of its high color content. A sample of palm oil having a free fatty acid of 10.28 per cent (Palmitic acid) and a color reading of 35 yellow-33.3 red (one inch Lovibond cell) was heated to 130° C. and 6 per cent benzoyl peroxide added. The mixture was stirred at this temperature for 5 minutes after which the oil was filtered through filter paper and the color determined in the same manner as the original sample of oil. The color, after bleaching read 35 yellow 1.3 red. Comparing these results with 3 other bleaching agents using the same method we have 35 yellow 8.3 red, 35 yellow 9.9 red, and 35 yellow 11.5 red.

It is commonly known that certain palm oils will bleach by passing air through the oil at an elevated temperature. Having devised a convenient laboratory apparatus similar to the Phosgene gas boards that we had in the gas defense laboratories during the war, except I placed an oil jacket made of pyrex glass around each test tube. They were connected together with glass tubing and rubber hose in the normal manner, so that air could be drawn through each test tube at the same time by means of a vacuum pump. I made a four-tube board marking the tubes A, B, C and D. Into (A) tube I placed 50 grams of the original oil containing 6 per cent benzoyl peroxide and in each of the remaining three tubes the same amount of original palm oil containing 6 per cent other bleaching agents. The four samples were heated to 130° C. and held at this temperature for 30 minutes while hot air was being bubbled through the oils at the same time. After the time expired the bleached samples of palm oil were removed, filtered through filtered paper and the colors determined on each, as in the previous method. The colors of the bleached oils gave with 6 per cent benzoyl peroxide the heated air treatment (A) 35 yellow-1.9 red, while the other bleached samples gave (B) 35 yellow-5.2 red, (C) 35 yellow-7.1 red, (D) 35 yellow-3.2 red. Compiling the results we have:

	COLOR	
Original Oil	35 Y-33.3 R	(I inch L. C.)

Sample (A) (6% B. P.) 35 Y-1.3 R (5 min. at 130° C.) (1 in L. C.)
Sample (B) (6% other B. A.) 35 Y-8.3 R (5 min. at 130° C.) (1 inch L. C.)
Sample (C) (6% other B. A.) 35 Y-9.9 R (5 min. at 130° C.) (1 inch L. C.)
Sample (D) (6% other B. A.) 35 Y-11.5 R (5 min. at 130° C.) (1 inch L. C.)

Tube (A) (6% B. P. & H. A.) 35 Y-1.9 R (30 min. at 130° C.) (1 inch L. C.)
Tube (B) (6% other B. A.) 35 Y-5.2 R (30 min. at 130° C.) (1 inch L. C.)
Tube (C) (6% other B. A.) 35 Y-7.1 R (30 min. at 130° C.) (1 inch L. C.)
Tube (D) (6% other B. A.) 35 Y-3.2 R (30 min. at 130° C.) (1 inch L. C.)

*Merck's Index (4th edition) page 112.
B. A. (bleaching agents).