

Refining Test Committee Report

Year's Work Covered Investigation of Filter Paper,
Color Differences and Recovery of Oil from Soapstock

By C. B. CLUFF, *Chairman*

THE Committee's work during the past year consisted mainly of three points: First, quality of filter paper used in filtering refined oil. Second, differences in color between the first and last portions of oil filtered. Third, more detailed instructions regarding cooling and draining foots after remelting.

First, as to quality of filter paper. The following brands of filter paper were tried out by different members and compared with Whatman's No. 40 paper as a standard or some paper equivalent to this as a standard.

Reeve Angel No. 202
Reeve Angel No. 211
Reeve Angel No. 230
Eimer & Amend White
Eaton-Dikeman No. 613
Eaton-Dikeman No. 615
Whitall Tatum
Whatman No. 50
Sargent No. 500

The difference in color reading when filtered through these various papers was, in most cases, so small as to be negligible when compared with our standard. Hence we can only recommend at this time that a good quality paper be specified without definitely excluding any of the commonly used brands.

Second, with regard to color of first and last portions of oil filtered. Experiments were made by reading the color of the first oil through the paper, then a portion after 50 cc. had been filtered, and thirdly the last portion of oil through the paper. The differences were in all cases very small and, in most cases, negligible. Inasmuch as our present rule requires that the first 50 cc. be returned to the filter, we feel that this is a sufficient precaution and that oil filtered after the first 50 cc. will show no measurable change. We therefore do not recommend any change in the existing rule.

Third, with regard to cooling and draining foots on remelting. Our present rule is not specific enough, as was pointed out by Mr. Fash. Some comparative tests show different results, depending on the time of cooling and draining. Hence, for the sake of uniformity,

we recommend to melt for 30 minutes, cool 15 minutes or until thoroughly chilled, and then drain 15 minutes.

In addition to the above points, a fourth point was given consideration relating to the time of agitation on slow breaking oils. A sample furnished by Mr. Fash to the Chemists' Committee, representing a very slow breaking oil, was found to give best results with 45 minutes agitation in the cold and 20 minutes hot. While no additional experimental work has been done on this point by the Refining Committee itself, the various members have had sufficient experience with this type of oil so that they confirm the conclusions of the Chemists' Committee on this point.

Recommendations

OUR recommendations are therefore as follows:

First, add to the note at the end of Rule 273, section 5(d) on Refining Procedure, the following paragraph:

"The filter paper used must be a white paper of good quality so that the filtered oil will have a color substantially equivalent to that filtered thru Whatman's No. 40 paper."

Second, in the same section (d) on Refining Procedure, insert the following, beginning in the seventh line from the bottom, replacing the clause beginning with the words "cool and decant." The new clause will read as follows:

"Cool in the cold water bath for 15 minutes or until thoroughly chilled. Then decant into a weighed beaker any additional oil thus recovered, and drain for 15 minutes."

Third, in the same section (d) on Refining Procedure, in the ninth line, change the parentheses to read as follows:

"45 minutes for oils designated as expeller or cold pressed type oil, or slow breaking oil."

The personnel of the committee included R. H. Fash, E. R. Barrow, R. C. Hatter, J. J. Ganuchau and C. B. Cluff, *Chairman*.

Revised Refining Procedure Including
Recommendations of the Refining
Committee, May, 1931

Rule 273 Section 5 (d)

REFINING Procedure. Place 500 grams of the thoroughly mixed sample of crude oil in a tared refining cup and settle to allow air to escape if necessary. Adjust the temperature of the oil and of the water bath to 20°-24° C., using ice to cool to this temperature if necessary. Agitator must run at the rate of 250 R.P.M. \pm 10. Add the proper amount of NaOH solution to the oil as quickly as possible, with the agitator running, and stir exactly fifteen minutes (forty-five minutes for oils designated as "expeller or cold pressed type" oil, or "slow breaking" oil) from the time the lye is added. Then immediately transfer to the 65° C. hot water bath and stir at 70 \pm 5 R.P.M. for twelve minutes (twenty minutes for designated slow breaking oils). Temperature of oil must then be 60°-65° 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 a water bath at 65° C. for one hour. Cool by setting in a cold water bath at 20°-24° C. 30 minutes and hold at this temperature for at least one hour additional, preferably over night. 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 filtered through white filter paper and used for determination of grade. The first 50 cc. of oil through the filter must be returned to the filter before taking sample for color reading. Weigh the oil and also weigh the soap stock cup with its contents. Melt the soap stock by setting it in a water bath maintained at 75 \pm 2° C., without stirring, for 30 minutes; cool in the cold water bath for 15 minutes or until thoroughly chilled. Then decant into a weighed beaker any additional oil thus recovered, and drain for 15 minutes. Weigh this oil separately. 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, if necessary, until the recovered oil from the last remelting amounts to not over 2.5 grams.

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 foots. The filter paper used must be a white paper of good quality so that the filtered oil will have a color substantially equivalent to that filtered thru Whatman's No. 40 paper.

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

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.

Niger Co., Ltd., controlled by Unilever, Ltd., and Nilever, N. Y., report for period of eighteen months ended December 31, 1930, net profit of £583,943. After payment of debenture charges and deducting sums written off for discount and expenses of the debenture interest there was a balance of £51,200 left. This with balance of £67,818 brought forward from previous year makes total of £108,018 carried forward.

In determining the solidifying points of vegetable oils good results were obtained with 6 to 7 grams of oil in a modified Mohr method. Solidification curves of some glycerides change upon adding other fats to them, while some retain original characteristics. *Kiserlet. Kozlemen.* 31,321-31 (1931), *Chem. Abstr.* 25,3188 (1931).

The hexabromide value of soy bean oil is said to be lowered by heating in vacuo to 225° C., the change being increased by the presence of infusorial earth and active nickel. The acid value increases under similar conditions. Elaidic acid is relatively stable toward active nickel on a support, resembling oleic acid in this respect. *Chem. Weekblad* 27,146-7.

Reduced freight rates on liquid caustic soda in tank cars from Solvay and Syracuse, New York, to numerous destinations in the state of New York have been approved by the New York Public Service Commission. The reductions averaged three cents per cwt. and went into effect early in July.