

TABLE NO. 11

Agreement of Plant Operator's Rapid Moisture Tests with Laboratory Tests Made in Freas Oven, 1.5 Hours at 105-110° C.

Remarks: 10 gram samples in 3" moisture dish were used on thin asbestos pad. The moisture values ranged between 10 and 13%. The apparatus and method is capable of much better accuracy than these figures show, as indicated by the results given in Table I. The plant operator who obtained these results was not of the type to take pains; his use of the interval timer was even rather inaccurate.

Date.....	First few days of training 14-15 min.	May 10 14 min.	May 11 13 min.	May 12 13 min.	May 14 13 min.	May 23 13 min.
Testing time.....	14-15 min.	14 min.	13 min.	13 min.	13 min.	13 min.
Difference in % moisture reported. + indicates plant result was high..						
	+ .65	+ .22	— .01	+ .55	+ .06	+ .21
	+ .30	+ .60	— .23	+ .22	+ .08	+ .38
	+ .22	+ .65	+ .29	— .01	+ .80	+ .35
	+ .35	+ .28	— .15	+ .38	+ .22	— .06
	+ .54	+ .29	+ .30	— .07	...	+ .43
	+ .30	...	— .13	+ .36	...	+ .38
	+ .35	...	+ .05
	— .04	...	+ .18
	— .03	...	— .10
	— .02
	— .02
	— .05
	— .28
	+ .32
	+ 1.00*
	— .40
	+ .20
	+ .35
	+ .40

the taking of some of the samples, with the consequence that there is no control at all in the real sense of the word—only periodic checking of values; whereas by going to a little trouble to find a suitable method of testing, many oil mill and refinery operations, especially those of a continuous nature, can be conducted with enhanced efficiency, freeing the laboratory at the same time from a rather annoying burden of testing.

Notes on Conditions—Moisture in Oil

The apparatus was designed with the additional view of providing a hot-plate of definite controlled temperature for use in making moisture determinations on oil. The A.O.C.S. official hot-plate method specifies a temperature of 130° C. for this test but gives no hint of how this temperature might be measured. The use of a thermometer in the oil being tested is impractical because

of weighing complications. The table given below shows one condition under which the test might be held at 130° C., using the apparatus described. Alternately, the boiling liquid in the tester might be replaced with oil and the temperature regulated at any desired value by use of a burner, the temperature being indicated by a thermometer placed in the bath oil.

The writer has seen unpublished data, which he has confirmed, showing that the requirements of our hot-plate method of bringing the oil to incipient smoking may cause high results to be obtained on Soybean Oil. The cause of accuracy would apparently be better served by changing the method so as to provide a definite maximum temperature to which the oil should be heated, even if momentarily; but should this be done, some such hot-plate of controlled and indicated temperature, as described, would be required.

RELATION OF VARIOUS TEMPERATURES

Boiling Liquid	194° F.	382° F.
Vapor	182	360
Air over Test	134	273
Oil in Dish on Bare Heater		
Outside of housing.....	154	310
Inside of housing.....	175	348
Oil in Dish on Asbestos Pad		
Outside of housing.....	130	266
Inside of housing.....	164	327

REPORT OF THE UNIFORM METHODS AND PLANNING COMMITTEE*

COLOR COMMITTEE:

The Color Committee have undertaken some very interesting work in the past year, which has not as yet been entirely completed. It is the recommendation of the Uniform Methods and Planning Committee that this work be continued.

COLOR GLASS DEVELOPMENT COMMITTEE:

This Committee had no recommendations to make, but the Uniform Methods and Planning Committee noticed that our methods call for the testing of standard glasses only by the Bureau of Standards. We, therefore, recommend that on page 16-e of our methods the statement be made that the Lovibond Color Glasses should be standardized either by the Bureau of Standards or the Electric Testing Laboratory.

*Reported at New Orleans, La., May 13, 1938.

CRUDE MILL OPERATIONS COMMITTEE:

This Committee made no formal report, but it was suggested by the chairman that a committee of this nature works under considerable difficulties, owing to the fact that developments in the crude mills are usually of a confidential nature. The Uniform Methods and Planning Committee, therefore, recommend that this committee be discontinued for the coming year.

FATTY ACID SOAP STOCK COMMITTEE:

This Committee made the following recommendations:

1. That the methods for analyzing soap stock and acidulated soap stock, except from copra or palm kernel oils, as outlined, be suggested to replace the ones now given in the Methods of the American Oil Chemists' Society;

2. that the methods be designated as 'Dry Extraction Method for Total Fatty Acids of All Soap Stock and Acidulated Soap Stock, except from Copra or palm Kernel Oils,' and 'Wet Extraction Method for Total Fatty Acids of All Soap Stock and Acidulated Soap Stock, except from Copra or Palm Kernel Oils';

3. that the term 'petroleum ether' be adopted throughout all methods of the Society for the solvent variously referred to as petroleum and petrolic ether."

The Uniform Methods and Planning Committee approve these recommendations and suggest that the methods be included during the coming year as tentative.

COMMITTEE ON INDICATORS:

This Committee made the following recommendations:

"The majority of the members of this committee feel that the purchase of a tested lot of indicator, to be dispensed by the Secretary, would be a satisfactory solution to the problem of an alternate indicator for dark oils. If that should prove to be the will of the Society, we recommend the selection of a Committee for that purpose.

"We are of the further opinion that if the indicator designated must be uniform, regardless of 'lot' or source of supply, that the value of continuing this work another year is doubtful. The Committee has no further recommendations."

The Uniform Methods and Planning Committee recommend that the Committee be continued. Also, that they purchase a lot of Aniline Blue and study its keeping quality.

We also recommend that the brine and petroleum method for Free Fatty Acids (applying only to dark oils where our present Alcohol Method is not applicable) be inserted as a tentative method.

It is also recommended that the new Committee study the A.S.T.M. Method for determining free fatty acids.

JOURNAL COMMITTEE:

This Committee made no recommendations, but brought out the fact that they had obtained a new contract with the publishers which would make the Society some money under very definite conditions. We feel that this Committee did a very fine piece of work in this direction.

OLIVE OIL COMMITTEE:

This Committee made no recommendations. The Uniform Methods and Planning Committee feel that this Committee is doing a very fine piece of work. We also recommend that the standards for Olive Oil and Olive Oil Foots, developed by this Committee in the past, should be placed in the Lefax method book under a new section. When data on other oils are collected these should also be included therein.

REFINING COMMITTEE:

The Refining Committee made the following recommendations:

"The Refining Committee recommends that the tentative soybean procedure as given on page 16D-16E of the AOCS methods be continued as tentative but

changed and augmented as follows:

"Insert new section after first paragraph to cover mixing:

"Heat the sample in hot water in original container to a temperature not to exceed 50°C. and *mix well*. Settlings must be thoroughly incorporated with the rest of the sample by examining the bottom of the container."

"Change the last two paragraphs to read as follows:

"At the end of the slow agitation period of refining the expeller, hydraulic and extracted soybean oils shall be allowed to settle in the 65° bath for one hour. Cool by settling in a cold water bath at 12-15°C. for one hour. The oil shall then be allowed to set overnight before pouring off. After settling overnight, the oil should again be chilled in a bath at 12-15°C. for 30 minutes before pouring off, unless the temperature has been maintained within this range.

"The soap stock must be hardened by chilling in water at a temperature of 12-15°C. or lower, to permit satisfactory draining of the oil."

"The Committee further recommends that the method developed this year for Extracted Soybean Oil be adopted as tentative and included in the method following the paragraph covering Hydraulic Soybean Oil with proper mention of caustic strength and amount in the first paragraph. Additions to be made are as follows:

Paragraph No. 1:

"Strength of lye shall be 14 Bé for Extracted Oil. Two tests shall be made on each oil using 7/8 of the maximum of sodium hydroxide as calculated from the formula FFA .54 and

$$\frac{\quad}{5.2} +$$

2/3 of this maximum quantity of sodium hydroxide."

"To follow paragraph covering handling of Hydraulic Oil:

"Extracted Soybean Oil shall be agitated at 20-24°C. for 90 minutes from the time the sodium hydroxide solution is added with the agitator running 250 R.P.M. \pm 10. It shall then be immediately transferred to the

65°C. bath and stirred at 70 \pm 5 R.P.M. for exactly 20 minutes. 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."

The Uniform Methods and Planning Committee approve these methods for adoption as tentative.

COMMITTEE ON REVIEW OF SCIENTIFIC LITERATURE:

This Committee has continued to do a very excellent piece of work. The Uniform Methods and Planning Committee recommends that a vote of thanks be given to Swift and Company for the work of this Committee and the special efforts they have given to the work of the Committee on the Refining of Soybean Oils.

SEED ANALYSIS COMMITTEE:

This Committee had no recommendations and the Uniform Methods and Planning Committee suggest that further work be done along the lines suggested by the newly-appointed chairman.

MOISTURE COMMITTEE:

The Committee made the following recommendations:

"The Committee recommends, therefore, that the following oven, described in terms of functional performance, be substituted for the now recommended Freas oven No. 601-233; 'A forced circulation oven designed to produce temperature of circulative air which can be controlled between the limits of 100-105°C. The sensitivity of the thermostat shall be such that the maximum variations of the temperature at the position of the thermo-regulator in the oven shall be \pm 1°C. at any one setting. However, no oven is approved for use with a greater number of seed or meal samples than the number of empty containers of approved type which can be placed in the oven without causing a variation of more than 4°C. within the usable space of the oven in the range 100-105°C.

"The rate and direction of the flow of air shall be such that proper drying will be obtained without danger of finely divided materials blowing from the sample container.

"The Committee recommends that a drying interval of three hours be required for meal sam-

ples, and a drying interval of four hours for cottonseed samples.

Correction statement received April 19, 1938.

"On page 2, the second paragraph of this report now states:

"The Society's methods now require that a 5-hour drying interval be used in both the standard jacketed glycerin oven and in the Freas forced-circulation oven."

This is to be changed to read as follows:

"The Society's methods now require that a 5-hour drying interval be used for cottonseed, and a 3-hour interval for cottonseed meal, in both the standard jacketed glycerin oven and in the Freas forced-circulation oven."

The Uniform Methods and Planning Committee suggest that the Committee continue its work and study the minimum time required on both seed and meal in the forced draft circulation oven, which will enable them to obtain values equivalent to the standard oven. They are also to investigate the variation of the temperature at different points of this oven.

SOYBEAN ANALYSIS COMMITTEE:

The report of the Soybean Analysis Committee contained the following recommendation:

"It is recommended that many collaborative analyses be made following the tentative method adopted last year, with a view of making these methods official."

This was approved by the Uniform Methods and Planning Committee and if actively followed should result in considerable information for the coming year.

SAMPLING COMMITTEE:

There was no report received from this Committee.

OIL CHARACTERISTICS COMMITTEE:

This Committee presented some data on several lots of cottonseed oil. The Uniform Methods and Planning Committee are of the opinion that the work of this Committee is quite valuable and should be continued. The results should be published as stated under comments on the Olive Oil Committee, in a separate section in the Lefax Methods.

STABILITY COMMITTEE:

There was no report from the Stability Committee and the Uni-

form Methods and Planning Committee recommend that this Committee be continued, as there is no doubt that there is a place for this type of evaluation of oils and fats.

FAT ANALYSIS COMMITTEE:

This Committee had several recommendations to make, the following of which were approved by the Uniform Methods and Planning Committee:

"Wiley Melting Point: The Committee recommends that the specifications for the beaker and test tube be changed to the following:

Beaker: Height, 200 mm.

Diameter, 85 mm.

(This corresponds to the common Griffin tall form-1000 ml.)

Test Tube:

Overall length, 300 mm.

Inside diameter, 35-38 mm.

Moistures: The Committee recommends that the wording of the method for the hot-plate moisture determination be changed slightly so as to avoid the possibility of misinterpretation. We suggest the method read as follows:

Hot-Plate Method — Determination:

Weigh out 5- to 20-gram portions of the prepared sample into a glass beaker or casserole and heat on a heavy asbestos board over burner or hot plate, taking care that the temperature of the sample does not at any time go above 130° C. until the very end of the test (see below). During the heating rotate the vessel gently by hand to avoid spattering or too rapid evolution of moisture. The approach of the end point may be judged by the absence of rising bubbles of steam and by the absence of foam at the last. At this point the heating should momentarily be carried on to incipient smoking (Caution!). Cool in desiccator and weigh.

"Limitations. This method is applicable to all the ordinary fats and oils, including emulsions such as butter and oleomargarine, and high acid coconut oil. It is not applicable, however, to certain abnormal samples such as naphtha extraction greases which contain, in addition to moisture, solvents of fairly high boiling point which are driven off with difficulty. In handling such samples it is possible to obtain satisfactory results by using the Kingman distillation method for actual moisture and steam distillation of the solvents. In difficult cases it may be advis-

able to determine the actual saponifiable matter present."

REFEREE BOARD:

In the report of the Referee Board it was suggested that several phrases in the wording of the Refining Test were somewhat ambiguous. The following were specifically mentioned; the word "concordant" and "two or more closely agreeing tests." This matter was discussed by the Uniform Methods and Planning Committee and the recommendation made that this Committee, together with the Chemists Committee of the National Cotton Products Association, try and work out a more specific description to replace the above.

SULPHONATED OILS COMMITTEE:

This Committee had no report to present.

SOAP IN OIL COMMITTEE:

This Committee made the following recommendation, which was approved by the Uniform Methods and Planning Committee:

"The Committee does not recommend the adoption of any of the methods tried as official or tentative methods of the Society, but does recommend that the cooperative work be continued for at least another year, particularly on the Durst HCL extraction method, the Durst method as modified by R. C. Stillman, and the alcohol extraction method."

The Uniform Methods and Planning Committee recommend that the following tentative methods be adopted as official:

1. Free Fatty Acid on p. 3.
2. Wiley Melting Point on p. 44.
3. Thiocyanogen Method on p. 44-b.
4. Twitchell Method on p. 44-d.
5. Methods of Analysis for Sulfonated (Sulfated) Oils on pp. B-1 to B-9 incl.

All of these have been tentative since 1935.

All of the above recommendations made by the Uniform Methods and Planning Committee were considered separately by the Convention and approved unanimously.

- J. T. R. Andrews,
- E. Freyer,
- R. C. Hatter,
- C. P. Long,
- H. P. Trevithick,
- J. J. Vollertsen, Chairman.