

ings is due to the adjustment of the eye which always takes time.

I have had the opportunity to see a demonstration by a physician in the use of the fleuroscope in which readings of the heart, lungs, etc., are made by the X-ray and I find that physicians who do this work allow themselves to remain in a dark room for a period of from ten to twenty minutes before taking their readings in order to allow time for the adjustment of the pupils of the eyes through dilation. The importance of waiting in a dark room in making clear and definite readings in this case was clearly shown by passing the X-ray through a thick layer of blank paper at the bottom of which were letters and figures which could not be at first read, but which later were clearly revealed. I do not intend to infer from this experiment that we should change our method to take color readings under the same conditions (although it probably would increase accuracy), but I mention this to show something of the eye-adjustment process which varies with different individuals, and here is where the personal equation comes in.

As information from the standpoint of the instrument and the non-symmetry of the eyes in the individual, I have a letter which was kindly written by Mr. Roger S. Estey of the Photometric Department of Electrical Testing Laboratories which I would like to quote:

"In my opinion, the principal conditions affecting variations in Lovibond readings (assuming the sample and the matching glasses to be identical in all cases) are:

"(a) Variations in the color of

the light source. These can be reduced by agreeing to use a standard source such as, for example, a standard lamp operating in connection with a standard blue filter.

"(b) Non-symmetry in the instrument. The instrument may contain reflecting or transmitting surfaces which, due to yellowing with age or accumulation of dirt, oil films, etc., have slightly modified the color of one beam with respect to the other. This may easily amount to a few tenths red on the Lovibond scale.

"(c) Temperature of the oil sample. I do not know what the temperature co-efficient of these samples is but presume that in the most careful interlaboratory comparisons a uniform oil temperature would have to be selected and maintained.

"(d) Non-symmetry of the eye. It is well known that even perfectly normal observers do not have exactly the same color sensitivity in each of their two eyes and, furthermore, that even one eye has a color sensitivity which is non-uniform across the retinal field. This effect and the effect described under (b) above can be eliminated by grading oils with the sample on the left and repeating with the sample on the right. The grade representing the average of these two measurements would eliminate any lack of color symmetry in the instrument or in the observer. (e) Unsymmetrical reflectance in the instrument.

"(f) Brightness differences in the photometric field. These elements produce a lack of symmetry in the measuring procedure which would undoubtedly contribute to a greater

or less degree to uncertainties in the values of oil samples measured.

"It is important in the interests of accuracy to have the illumination through the tintometer as bright as the observer finds comfortable. Lower illuminations will lead to discomfort and inaccuracy. As regards the use of a dark room, it is naturally important that the eyes of the observer should not have to undergo large changes in adaptation when he starts to use the instrument. The eyepiece should be shielded from stray light which would produce confusing and tiring stimulation of the margins of the visual field. I do not see how these difficulties would affect the readings directly but undoubtedly the observer would quickly become tired and confused."

In conclusion, I feel that the main differences caused by chemists are two in number: (1) The reading of samples in the midst of the eye-adjustment process, and (2) the reading of samples that are too dark in color for comparisons to be properly made.

I believe that correction in large measure for the eye-adjustment process would be brought about by having a suitably constructed and standardized hood and requiring that chemists look into the dark spaces of the hood for a definite period before taking their readings.

Finally concordant results may be obtained in the reading of dark oils of more than 25 red by taking half the usual column, namely, $2\frac{5}{8}$ inches, and whatever yellow glass the Color Committee might decide upon, and then multiply the reading of red by two.

REPORT OF THE SMALLEY FOUNDATION COMMITTEE*

WE are presenting herewith the 18th report of the Smalley Foundation Committee of the American Oil Chemists' Society. During these past eighteen years considerable progress has been made in the accuracy of the determination of Oil and Ammonia on cottonseed meal. According to our rules the cup, which represents the best results in both Oil and Ammonia determinations, must be won by a collaborator three times before it becomes his permanent posses-

sion. This has occurred on two occasions, the first cup having been won by Dr. H. B. Battle. This cup was presented by the Industrial Chemical Sales Corporation. The second cup, which was presented by Dr. Battle, was won by Dr. W. F. Hand, and he immediately replaced the cup by a third, which now stands as the trophy for the second year.

As usual, thirty samples of cottonseed meal were distributed to the collaborators. The results, as a whole, are on the same high plane

as those of the preceding years, the differences in percentage of perfection being so small as to be almost negligible. During the year it was decided to send out one meal sample which differed from the rest. This was sample No. 10, which had a higher oil content than the others and was passed through a 20 mesh screen when it was prepared. We felt that this was not unlike samples that come into the laboratory of a great majority of the members who participated in this cottonseed work

*As presented at the Spring Meeting, A. O. C. S., New Orleans, May 28-29, 1936.

and for this reason thought it would be a good idea to see how well the members checked on a sample of this nature. Owing to the fact that the results were not in as close an agreement as the regular samples, it was decided to omit them from the general average. Therefore, the tables which accompany this report are based on the average of twenty-nine samples, rather than the full set of thirty which were received by the collaborators.

There are attached to this report four tables, indicating the standing in percentage of the members taking part. Table No. I gives the

TABLE NO. I
Determination of Oil.

| Analyst No. | Points off | Per cent Efficiency |
|-------------|------------|---------------------|
| 47 | 14 | 99.931 |
| 13 | 15 | 99.925 |
| 20 | 22 | 99.890 |
| 36 | 23 | 99.886 |
| 6 | 27 | 99.866 |
| 27 | 27 | 99.866 |
| 66 | 30 | 99.851 |
| 64 | 31 | 99.846 |
| 2 | 33 | 99.835 |
| 10 | 34 | 99.831 |
| 12 | 35 | 99.825 |
| 16 | 35 | 99.825 |
| 69 | 39 | 99.805 |
| 26 | 42 | 99.791 |
| 65 | 42 | 99.791 |
| 22 | 43 | 99.786 |
| 23 | 43 | 99.786 |
| 8 | 48 | 99.760 |
| 40 | 50 | 99.752 |
| 41 | 50 | 99.752 |
| 46 | 50 | 99.752 |
| 14 | 51 | 99.746 |
| 53 | 54 | 99.731 |
| 49 | 56 | 99.721 |
| 21 | 57 | 99.716 |
| 3 | 58 | 99.711 |
| 45 | 58 | 99.711 |
| 11 | 60 | 99.701 |
| 71 | 60 | 99.701 |
| 55 | 62 | 99.691 |
| 32 | 64 | 99.681 |
| 7 | 66 | 99.671 |
| 37 | 66 | 99.671 |
| 28 | 69 | 99.656 |
| 1 | 70 | 99.652 |
| 57 | 70 | 99.652 |
| 38 | 72 | 99.642 |
| 15 | 78 | 99.612 |
| 5 | 80 | 99.602 |
| 48 | 95 | 99.527 |
| 56 | 96 | 99.522 |
| 52 | 107 | 99.467 |
| 51 | 108 | 99.462 |
| 4 | 112 | 99.443 |
| 68 | 114 | 99.433 |
| 19 | 119 | 99.408 |
| 18 | 149 | 99.258 |

standing of 47 collaborators who reported Oil determinations on all samples. Table No. II gives the

TABLE II
Determination of Ammonia.

| Analyst No. | Points off | Per cent Efficiency |
|-------------|------------|---------------------|
| 24 | 3 | 99.988 |
| 13 | 5 | 99.980 |
| 14 | 6 | 99.975 |
| 20 | 6 | 99.975 |
| 5 | 7 | 99.971 |
| 6 | 9 | 99.963 |
| 15 | 10 | 99.959 |
| 10 | 11 | 99.955 |
| 27 | 11 | 99.955 |
| 64 | 11 | 99.955 |
| 16 | 12 | 99.946 |
| 21 | 13 | 99.946 |
| 69 | 13 | 99.946 |
| 2 | 14 | 99.943 |
| 41 | 15 | 99.938 |
| 33 | 16 | 99.935 |
| 28 | 17 | 99.930 |
| 47 | 17 | 99.930 |
| 62 | 19 | 99.923 |
| 55 | 20 | 99.918 |
| 68 | 20 | 99.918 |
| 71 | 20 | 99.918 |

| | | |
|----|-----|--------|
| 22 | 24 | 99.901 |
| 46 | 24 | 99.901 |
| 36 | 25 | 99.898 |
| 26 | 26 | 99.893 |
| 4 | 29 | 99.881 |
| 40 | 30 | 99.877 |
| 18 | 31 | 99.873 |
| 23 | 32 | 99.869 |
| 34 | 32 | 99.869 |
| 3 | 33 | 99.864 |
| 19 | 34 | 99.861 |
| 53 | 34 | 99.861 |
| 7 | 37 | 99.848 |
| 49 | 37 | 99.848 |
| 11 | 40 | 99.836 |
| 45 | 41 | 99.832 |
| 57 | 42 | 99.827 |
| 37 | 43 | 99.824 |
| 29 | 44 | 99.819 |
| 51 | 44 | 99.819 |
| 54 | 45 | 99.815 |
| 66 | 49 | 99.799 |
| 8 | 53 | 99.782 |
| 12 | 53 | 99.782 |
| 48 | 54 | 99.779 |
| 32 | 63 | 99.742 |
| 1 | 69 | 99.717 |
| 60 | 83 | 99.659 |
| 56 | 97 | 99.602 |
| 66 | 98 | 99.598 |
| 38 | 114 | 99.532 |
| 52 | 130 | 99.466 |

standing of 55 collaborators who reported Ammonia results on all samples. Table No. III gives the

TABLE NO. III
Determination of Oil and Ammonia.

| Analyst No. | Per cent Efficiency |
|-------------|---------------------|
| 13 | 99.953 |
| 20 | 99.933 |
| 47 | 99.931 |
| 7 | 99.915 |
| 26 | 99.911 |
| 64 | 99.901 |
| 10 | 99.893 |
| 3 | 99.892 |
| 2 | 99.889 |
| 16 | 99.886 |
| 69 | 99.876 |
| 14 | 99.861 |
| 41 | 99.845 |
| 22 | 99.844 |
| 26 | 99.842 |
| 21 | 99.831 |
| 23 | 99.828 |
| 46 | 99.827 |
| 40 | 99.815 |
| 71 | 99.810 |
| 33 | 99.808 |
| 55 | 99.805 |
| 12 | 99.804 |
| 53 | 99.796 |
| 65 | 99.795 |
| 28 | 99.793 |
| 3 | 99.788 |
| 5 | 99.787 |
| 15 | 99.786 |
| 49 | 99.785 |
| 45 | 99.772 |
| 8 | 99.771 |
| 11 | 99.769 |
| 7 | 99.760 |
| 37 | 99.748 |
| 57 | 99.740 |
| 66 | 99.725 |
| 1 | 99.685 |
| 68 | 99.676 |
| 4 | 99.662 |
| 48 | 99.653 |
| 51 | 99.641 |
| 19 | 99.635 |
| 38 | 99.587 |
| 18 | 99.566 |
| 56 | 99.562 |
| 52 | 99.467 |

standing of 47 collaborators who reported Oil and Ammonia on all samples. In these tables we have taken into consideration the results of those reports which were received within the time specified in our original announcement of the Smalley Foundation work. In table No. IV we have given the standing of those collaborators who reported on all samples, but some of whose reports were received too late to be included under the rules.

TABLE NO. IV
Special Table.

| Analyst No. | Points off | Per cent Efficiency |
|----------------------------------|------------|---------------------|
| Determination of Oil | | |
| 9 | 51 | 99.746 |
| 59 | 62 | 99.691 |
| 70 | 74 | 99.632 |
| Determination of Ammonia | | |
| 9 | 18 | 99.926 |
| 70 | 32 | 99.869 |
| 59 | 98 | 99.598 |
| 67 | 107 | 99.561 |
| Determination of Oil and Ammonia | | |
| 9 | | 99.836 |
| 70 | | 99.751 |
| 59 | | 99.645 |

The winning collaborators are as follows:

The "Mississippi State Chemical Laboratory Cup" for the highest efficiency in the determination of both Oil and Ammonia on all samples is awarded to Analyst No. 13, Mr. N. C. Hamner, Southwestern Laboratories, Dallas, Texas, with an average of 99.953 per cent. The average efficiency is lower than that of last year, which was 99.977 per cent. The certificate for second place goes to analyst No. 20, Mr. J. N. Pless, Royal-Stafolife Mills, Memphis, Tenn., with an efficiency of 99.933 per cent, as compared with 99.947 for last year.

The certificate for the highest efficiency in determination of the oil only is awarded to Analyst No. 47, Mr. T. L. Rettger, Buckeye Cotton Oil Company, Memphis, Tenn., with an average of 99.931, as compared with 99.974 for last year. The certificate for second place goes to Analyst No. 13, N. C. Hamner, Southwestern Laboratories, Dallas, Texas, with an efficiency of 99.925, as compared with 99.926 for last year.

The certificate for the highest efficiency in the determination of ammonia is awarded to Analyst No. 24, Mr. George K. Redding, Larowe Milling Company, Rossford, Ohio, with an average of 99.988, as compared with the same average for last year. The certificate for second place goes to Analyst No. 13, N. C. Hamner, Southwestern Laboratories, Dallas, Texas, with an average of 99.980, as compared with 99.984 for last year.

During the past year some difficulty has been encountered by the Chairman in tabulating and reporting the analyses because in certain instances reports have been received after the averages had been compiled and just before the tabulation has been sent to the printer. In some cases it has been found that these late reports have been received from the same individuals and it would appear that this late reporting in such cases has become a

matter of habit. We would recommend that during the next year's work only such reports as are in the hands of the chairman not later than Tuesday noon of the week in which the report is to be prepared, shall be considered in the final compilation.

We have often wondered how many of the members of the Society ever give any thought to the labor and care which is required in

the preparation and distribution of these check meal samples. It is very easy for all of us to take this as a matter of course and to find fault with any little discrepancies which we think make the analysis of the sample more difficult. We feel that the Society, and particularly those members who take part in the check meal work, are greatly indebted to Mr. T. C. Law for his willingness to assume this burden year by year.

Personnel of Committee:

- THOS. C. LAW,
- E. H. TENNENT,
- B. L. CALDWELL,
- T. B. CALDWELL,
- F. PAQUIN,
- J. N. PLESS,
- W. C. MOOR,
- M. E. WHITTEN,
- J. J. VOLLERTSEN,

Chairman.

REPORT OF COMMITTEE ON SOAP IN REFINED OILS*

THE determination of small quantities of alkali soap in refined oils has been the problem of nearly each individual plant chemist who is in direct contact with edible oil refining. Since there is no standard method for this analysis, each chemist devised his own procedure with the result that there are now quite a few methods available. Because of the difficulty involved in obtaining concordant results by different methods, it is quite apparent that a standard analysis for the quantitative determination of soap in oil is important.

The amount of soap that may be found in a freshly neutralized oil will vary from 0.05% to 0.15%, depending upon the oil and the method of neutralization. A dried, bleached, and filtered oil will contain less than 0.005% of soap. A good edible oil should not contain any soap.

When a dried and filtered oil, liquid at ordinary temperature, is allowed to remain at 90° to 100° F. for some time, any soap present will separate in the form of a cloud. There may be other substances present in an oil that will tend to cloud it, however, the experienced chemist can probably recognize an alkali soap.

The oldest and probably the most common method employed is that of ashing the oil or fat, dissolving the ash in water, titrating with a standard acid solution and calculating the equivalent to sodium oleate or stearate.

This method is quite satisfactory when employed on oils containing high amounts of soap, such as 0.1% or greater. However, when the soap content varies from 0.001% to 0.01%, the amount of oil to be ashed so as to obtain a fair quan-

tity of ash for titration with an acid would be so great as to make this method almost prohibitive and certainly time consuming.

From the standpoint of the oil plant chemist, it is more important to be able to determine accurately the smaller than the larger quantities of soap in oil. Your committee reasoned that if it were possible to devise a method that will accurately determine small quantities of soap in oil, the larger quantities will present no problem.

Following is the method used in the preparation of samples that were distributed to the members of this committee: A neutralized oil was washed with water several times to remove the soap. The oil was then dried, bleached, filtered, and allowed to remain at a temperature of 90° F. for three days and filtered twice through filter paper at ordinary temperature. This oil was then analyzed by both of the below outlined methods and found to be absolutely soap free. A definite amount of sodium oleate was then incorporated and samples mailed to the members for analysis. The sodium oleate was analyzed previous to incorporation and found to be 100% pure.

Following is the procedure for the first method of analysis:

Weigh 50.0 grams of the fat to be tested into a 250 ml. separatory funnel. Add 50 ml. distilled

water heated previously to about 150° F. and shake for about two minutes. Add 5 ml. N/2 HCl and shake vigorously for five minutes. Allow to settle and draw off the water. Wash the oil remaining in the separatory funnel with 50 ml. portions of hot water until the wash water is neutral. Three or four washes are usually sufficient. Draw off the washed oil into a 250 ml. beaker and place in a hot water bath at about 160° F. for ten minutes to settle the water. Filter the oil to remove any remaining moisture and determine the F. F. A. as oleic acid using N/50 NaOH. Also determine the F. F. A. of the original oil with the same standard N/50 NaOH solution. Using a 28.2 gram sample.

$$\% \text{ F. F. A. as Oleic} = \text{ml. N/50 NaOH} \times .02$$

$$(\text{F. F. A. treated oil} - \text{F. F. A. original oil}) \times 1.08 = \% \text{ soap as sodium oleate}$$

For accuracy it is best to determine the free fatty acid of the treated and untreated oils at the same time, using the same alcohol that was previously neutralized with NaOH solution and employing phenalthalein as indicator; the end point being when the mixture just turns pink.

The results obtained are shown in Table 1.

TABLE 1.

| Kind of Oil | Sample A Cottonseed | Sample B Cottonseed | Sample C Cocoonut |
|---|------------------------|------------------------|----------------------|
| Per cent sodium oleate actually incorporated..... | 0.0040 | 0.0100 | 0.0050 |
| Laboratory No. 1 | 0.0043 | 0.0108 | 0.0054 |
| Laboratory No. 2 | 0.0048 | 0.0111 | 0.0064 |
| Laboratory No. 3..... | 0.0045 | 0.0045 | 0.0014 |
| Laboratory No. 4 | 0.0089 | 0.0157 | 0.0093 |
| Laboratory No. 4 | 0.0042 | 0.0032 | 0.0005 |
| Average of all laboratories | 0.0053 | 0.0090 | 0.0046 |

*As presented at Spring Meeting, A. O. C. S., New Orleans, May 28 and 29, 1936.