contain lypolytic enzymes for if the ground seed is left in contact with the oil, the acidity rises rapidly (Kreis 1918).

The oil is liquid at ordinary temperatures. It is green when recovered by petrolic ether extraction, when expressed it is yellowish green. The chemical and physical constants of the former were found to be:

TABLE II

Chemical and Physical Characteristics of Elm Seed Oil (*Ulmus americana*)

Specific gravity 20°/20°[3]	0.9288
Index of refraction 20°[3]	1.4554
Coefficient of viscosity 20° (centipoises)[3]	0.3381
Surface tension 20° (dynes/cm)[3]	30.72
Titer test °C	14.0
Iodine number (Wijs)	24.10
Saponification number	273.0
Thiocyanogen number	16.18
Hydroxyl number	13.45
Reichert-Meissl number	2.1
Polenske number	33.9
Soluble acids (per cent as butyric)	0.8
Insoluble acids (Hehner number)	82.23
Iodine number of fatty acids	23.08
Thiocyanogen number of fatty acids	15.51
Saponification number of fatty acids	288.7
Unsaponifiable matter (per cent)	1.00

[3] Determined by F. S. Meade.

The presence, in large amount, of fatty acids of intermediate molecular weight as revealed by the high Polenske number, was later verified when, following methyl alcoholysis of the oil itself, approximately 50 per cent of capric acid was indicated. This value, however, must for the present be regarded as tentative for the separation will be repeated.

The calculated percentages of glycerol and the total fatty acids, assuming that this oil is composed of a mixture of triglycerides, are 14.9 and 92.8, respectively. The latter figure closely agrees with that found experimentally, or 92.7. Unsaturated acids are obviously present in small amount. Calculation by the Kaufmann procedure reveals that saturated acids comprise 82.82 per cent of the total acids. Similarly, oleic and linoleic acids comprise 8.83 and 8.36 per cent, respectively, of the whole.

TABLE III

Range of Constants of Elm Seed Oil

Iodine number	15.9 —37.9
Saponification number	26.4 —27.9
Reichert-Meissl number	3.0 — 5.7
Polenske number	33 —41
Hehner number	75.4
Unsaponifiable matter (per cent)	0.92—10.8 (1.35?)
Solidifying point °C	2.0 — 7.1

For purposes of comparison with such of the constants of this oil as have been already reported abroad, there is included here (Table III) a survey of such data. The species in question has not always been indicated by the investigator in question. Included among those reported on are *Ulmus campestris* L., *U. effusa, U. pedunculata,* and *U. scabra,* Mill. Except for the Hehner number, it appears tha the characteristics of the seed oil of *U. americana* fall within the range of those oils secreted by other species of this tree. Probable verification is also noted in the content of capric acid elsewhere reported (Pawlenko, 1912). In this connection it may be timely to recall that this oil is a richer source of this acid than either the milk fats of the cow or the goat, or of coconut and palm kernel oils (4.5 to 10.7 per cent).

In its major aspects, elm seed oil appears to be the counter part of coconut oil growing in the temperate zone. This investigation is being continued.

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Discussion

Mr. Richardson—Are these the first references?

Prof. Schuette—There are about eight references and all of them are of foreign origin. I suspect that this will be the first report on the physical and chemical constants of American elm seed oil.

Dr. Bosart—How much capric acid?

Prof. Schuette—Approximately 50 per cent.

THE ANALYSIS OF SULFONATED (SULFATED) OILS COMMITTEE REPORT

By RALPH HART, Chairman

COMMITTEE REPORT NO. 2—DETERMINATION OF ORGANICALLY COMBINED SULPHURIC ANHYDRIDE

SULFONATED oils are structurally distinguished from soaps by their contents of organically combined sulfuric anhydride, which may be combined with the fatty matter either as a sulfuric acid ester (sulfated oil) or as a sulfonic acid (true sulfonated oil). The SO_3 in the true sulfonated oil is linked directly to a carbon atom, thus, $R\text{-}C\text{-}SO_3Na$, where R represents the fatty radical, whereas in the sulfated oil, it is connected to the fatty matter by an atom of oxygen, thus, $R\text{-}O\text{-}SO_3Na$. The sulfuric acid ester or sulfated oil is readily decomposed upon boiling with mineral acids, liberating free sulfuric acid, whereas the sulfonic compound remains stable under the same conditions.

In the Herbig method (10, 15), the splitting off of SO_3 upon heating the ester with mineral acids, is utilized for its estimation. The sample is hydrolyzed by boiling with hydrochloric acid and the total sulfur in the water layer determined as barium sulfate. Commercial sulfonated oils are contaminated more or less with inorganic sulfate—due to excess acid from the sulfonation process or to the Glauber's salt used in washing the crude sulfonated oil free from excess sulfuric acid. To determine the organically combined SO_3 by the Herbig method, it is therefore necessary to subtract from the total sulfur the SO_3 pres-

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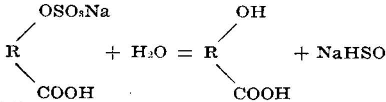
*A PAPER PRESENTED AT THE FALL MEETING, OCTOBER 17-18, 1935 ent as inorganic sulfate. To this end, a fresh sample is dissolved in a solvent or a mixture of solvents, washed with a concentrated solution of sodium chloride until all the inorganic sulfate is removed, and the latter determined in the wash waters with barium chloride. This method obviously applies only to oils that are decomposed by acids (i. e., ester type) and which, when dissolved in a solvent, do not form emulsions with concentrated salt solution. This method is time consuming and has been almost entirely displaced, at least in this country, by the following titration method.

Titration Method

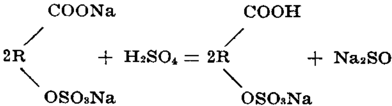
The titration method (8) is the official method of the Bureau of Standards (7), American Leather Chemists' Association (1), the American Association of Textile Chemists and Colorists (A. A. T. C. C.) (9), and has been recommended also by a sub-committee of the Sulfonated Oil Manufacturers' Association. This method has been in use for about 20 years, during which time a number of papers (2, 3, 4, 6, 11, 12, 13, 14) have been published establishing its reliability. It may also be of interest to mention that Biffen and Snell (5) have recently recommended this method also for the analysis of sulfated alcohols.

The titration method depends upon the fact that when a sulfonated oil of the ester type is boiled with excess sulfuric acid hydrolysis or decomposition takes place, producing an increase in the mineral acidity of the mixture that corresponds quantitatively to the amount of organically combined SO_3 liberated. Under the proper conditions the hydrolysis is complete and the subsequent change in acidity may be determined accurately. The change that takes place upon boiling with acid is represented by the following equation:



$$R\begin{cases}OSO_3Na\\COOH\end{cases} + H_2O = R\begin{cases}OH\\COOH\end{cases} + NaHSO_4$$

That is, for every molecule of combined sulfuric anhydride there is liberated one hydrogen equivalent of mineral acid, in the form of $NaHSO_4$. In the presence of soap, some of the acid is lost by neutralization, as follows:



$$2R\begin{cases}COONa\\OSO_3Na\end{cases} + H_2SO_4 = 2R\begin{cases}COOH\\OSO_3Na\end{cases} + Na_2SO_4$$

Hence, to find the total increase in acidity after the boiling, two determinations are necessary: namely, (1) the decrease in acidity due to the neutralization of the acid by the soap—given by the alkalinity of the sample and designated as "A"; and (2) the increase in acidity after boiling the sample with sulfuric acid—designated at "F." Both determinations are made volumetrically in presence of methyl orange indicator.

Cooperative Sample 2 was analyzed by the committee using the titration method, following the A. A. T. C. C. procedure given at the end of this report. The results are listed in Table I, from which it will be noted that the greatest difference was 0.24 per cent and the average deviation 0.06 per cent. It is interesting to note that the A. A. T. C. C. committee, in investigating this method some time ago, reported for a similar sample of a sulfonated oil the greatest difference as 0.29 per cent and the same average deviation, namely, 0.06 per cent. Since the combined results represent the work of about 20 laboratories, it may be concluded that the precision by this procedure is entirely satisfactory for commercial work.

TABLE I

Organically Combined Sulfuric Anhydride in Cooperative Sample 2 by the Titration Method.

	Combined SO_3, Per Cent			
Analyst	I	II	Avg.	Deviation from Mean
Hart	3.45	3.39	3.42	0.04
Irwin	3.30	3.36	3.33*	0.05
Long-Andrews	3.38	3.47	3.42*	0.04
Lauro	3.35	3.45	3.40	0.02
Manning	3.44	3.46	3.45*	0.07
Pingree	3.23	3.28	3.25*	0.13
Sheely	3.42	3.56	3.49	0.11
Tiffany	3.00	3.30	3.28	0.10
Wechsler-Segesseman	3.38	3.40	3.39*	0.01
Mean			3.38	
Greatest difference			0.24	
Average deviation			0.06	

*More than 2 determinations.

Extraction-Titration Method

Unfortunately the titration method is unreliable in the presence of sodium acetate or other compounds that do not give a sharp end point with methyl orange indicator. In such cases, the ash-gravimetric method, described later, is recommended by the A. A. T. C. C. The present committee has investigated a modification of the titration method—namely, extraction-titration method—and finds that it yields entirely reliable results and is simpler to manipulate than the ash-gravimetric method. According to the modified method, the sample is dissolved in a solvent, washed free of the interfering compounds with concentrated salt solution, the solvent evaporated, and the SO_3 determined in the purified residue by the regular titration method. The same cooperative sample was analyzed by the extraction-titration method, details of which will be found below, and the results are given in Table II. Upon comparing this table with the previous one, it will be noted that the mean results differ by only 0.07 per cent, the greatest difference by 0.05 per cent, and the average deviations are identical.

TABLE II

Organically Combined Sulfuric Anhydride in Cooperative Sample 2 by the Extraction-Titration Method.

	Combined SO_3, Per Cent			
Analyst	I	II	Avg.	Deviation from Mean
Hart	3.34	3.36	3.35	0.04
Irwin	3.18	3.39	3.26*	0.05
Long-Andrews	3.33	3.36	3.35*	0.04
Lauro	3.28	3.30	3.29	0.02
Pingree	3.17	3.24	3.20*	0.11
Wechsler-Segesseman	3.39	3.39	3.39	0.08
Mean			3.31	
Greatest difference			0.19	
Average deviation			0.06	

*More than 2 determinations.

Ash-Gravimetric Method

For true sulfonated oils and also for samples containing sodium acetate and other compounds that may interfere with the titration method, the A. A. T. C. C. specifies the ash-gravimetric method, which may also be used as a check on the titration method. This is similar to the extraction-titration method with the exception that the purified residue instead of being decomposed with acid is ashed. The ash of the purified sulfonated oil is pure sodium sulfate and represents half of the organically combined SO_3, in accordance with the following reactions that take place during the ashing:

1) $2R\text{-}O\text{-}SO_3Na = Na_2SO_4 + SO_3 +$ etc.
2) $2R\text{-}SO_3Na = Na_2SO_4 + SO_3 +$ etc.

By means of the ash-gravimetric method and either the titration or extraction-titration method, it is obviously possible to determine separately the SO_3 combined as sulfonic acid and as ester in samples containing both. The application of the ash-gravimetric method is shown by the following work carried out in the writer's laboratory. Samples of Igepon, isobutyl naphthalene sodium sulfate, and sulfonated mineral oil showed by this method combined SO_3 contents of 6.25 per cent, 10.70 per cent, and 14.21 per cent, respectively, whereas by the titration method no combined SO_3 was indicated.

Cooperative Sample 2 was analvzed bv the ash-gravimetric method (see below) and the results are shown in Table III, from which it will be noted that the agreement is of the same order, although not quite so good, as by the other methods. This checks the A. A. T. C. C. committee whose results on a similar sample compared with our work were as follows: Greatest difference, 0.35 per cent and 0.32 per cent, respectively; average deviation, 0.09 per cent for both committees.

TABLE III
Organically Combined Sulfuric Anhydride in Cooperative Sample 2 by the Ash-Gravimetric Method.

Analyst	Combined SO_3, Per Cent I	II	Avg.	Deviation from Mean
Clark	3.71	3.76	3.74	0.08
Hart	3.70	3.77	3.74	0.08
Irwin	3.59	3.75	3.66*	0.00
Long-Andrews	3.76	3.82	3.79*	0.13
Lauro	3.30	3.72	3.47*	0.19
Pingree	3.67	3.80	3.74*	0.08
Sheely	3.48	3.63	3.56*	0.10
Tiffany	3.65	3.68	3.67	0.01
Wechsler-Segesseman	3.54	3.57	3.56	0.10
Mean			3.66	
Greatest difference			0.32	
Average deviation			0.09	

*More than 2 determinations.

Upon comparing, however, the mean by the ash-gravimetric method with those by the other two methods, this committee shows a discrepancy of nearly 0.3 per cent, which is much above the experimental error. On the other hand, the A. A. T. C. C. committee showed practically no difference, namely, 0.02 per cent. Upon investigation it was found that the present cooperative sample contained a small amount of a true sulfonated oil, about equal to the discrepancy, which was not suspected when the samples were distributed to the committee. To determine this SO_3, bound as sulfonic acid, the sample was decomposed as in the titration method and the SO_3 in the decomposed fatty matter determined according to the ash-gravimetric method.

Comments and Suggestions

J. ANDREW CLARK: In the ash-gravimetric method we used a No. 30, 9 mm. ashless paper and dehydrated with sodium sulfate. In the ether used for washing the residue we found it necessary to dehydrate the ether first, as otherwise it seemed to carry small amounts of sodium sulfate through the filter paper.

RALPH HART: In obtaining the end-points in the titration methods, we experienced the same difficulty mentioned by Dr. Manning, namely, vigorous and frequent shakings obscured the end point. This only occurred while soap was still present. The procedure was modified so that there is no vigorous shakings until the solution is definitely on the acid side. Vigorous and frequent shaking at this stage, including the back-titrations, was found not to interfere with the end point. The latter was sharp, accurate to 1 or 2 drops of N/2 solution, and the separations rapid.

M. F. LAURO: The extraction-titration method can be made to yield on the same sample of alkalinity, ammonia and any other salts present in the oil with the exception of the sodium chloride used in washing. In the titration methods, I used a round-bottom flask and a water condenser. With a few beads in the flask, I could leave the process for the entire period of boiling without fear of bumping or loss of liquid. The use of anhydrous sodium sulfate is to be recommended in the dehydration of the ether extract in the ash-gravimetric method, as it reduces to a minimum, if it does not entirely eliminate, the occlusion of sodium chloride. Upon testing the ash of the residue dehydrated with sodium sulfate, I failed to find any sodium chloride.

As a further check on the purity of the extracted fat after washing in the ash-gravimetric method, the ash may be dissolved in a small amount of water and tested for salt (NaCl) and if found present corrected for same using the Volhard or volumetric method for determining chlorides with silver nitrate and chromate as the indicator.

EUGENE R. MANNING: The titration method gave quite satisfactory results except in the presence of sodium acetate and other titratable salts. In the early attempts of this investigation, which formed part of a thesis by students, we had considerable difficulty in titrating the solution because of the presence of fatty matter. This was noticed if the solution was shaken vigorously and frequently. We would also suggest slightly less indicator, i.e., four drops instead of five drops of methyl orange. It was also noticed that it frequently took more than 1½ hours of boiling to get both layers clear.

R. A. PINGREE: I am inclined to believe that the discrepancies in results between the titration and ash-gravimetric methods are due to the presence of true sulfonated oil. If the results by the various members of the committee are in agreement and all show the same discrepancy, I would take it as proof that the difference is due to a small amount of true sulfonated oil.

SEGESSERMAN: There is apparently a small amount of unhydrolisable SO_3 present in the sample. This, of course, we had never suspected, otherwise we would not have chosen this type of oil for the Committee Sample.

Recommendations

In view of these findings, the committee recommends the two A. A. T. C. C. methods—with the minor modification mentioned below—for the determination of organically combined SO_3 in sulfonated oils, viz., the titration and ash-gravimetric methods. In addition the committee recommends a modified titration method, namely, the extraction-titration method, for samples of sulfonated oils of the ester type containing sodium acetate or other interfering compounds. The modifications recommended in the A. A. T. C. C. methods are: A more accurate procedure in determining the end point in the titration method and dehydrating the residue in the ash-gravimetric method with anhydrous sodium sulfate instead of a concentrated solution of the salt.

Proposed Standard Method of Test for Organically Combined Sulfuric Anhydride in Sulfonated (Sulfated) Oils

A—Titration Method

Scope:

1. This method of test determines the organically combined sulfuric anhydride existing in a sample of sulfated oil by boiling the sample with sulfuric acid and titrating the products of reaction. This method applies only to sulfonated oils that split off their combined SO_3 upon boiling with mineral acids and do not contain compounds that cannot be accurately titrated in water solutions with methyl orange as the indicator.

Apparatus

General:

2. The apparatus shall consist of a glass flask provided with a glass stopper and an air condenser. The connection between the flask and the condenser shall be a ground joint. Perforated glass beads shall

be used to prevent bumping.

Flask:

3. The glass flask (Fig. 1) shall be an Erlenmeyer flask (a round bottom flask may also be used) made of Pyrex or other chemically resistant glass, having a capacity of approximately 300 ml.

Condenser:

4. The condenser (Fig. 1) shall consist of a glass tube having a length of 914 mm. (36 in.) and 8 mm. (5/16 in.) outside diameter. The lower end of the tube shall be flared and ground to fit the mouth of the Erlenmeyer flask.

Glass Beads:

5. The glass beads shall be perforated, made of chemically resistant glass, and shall be approximately 4 mm. (5/32 in.) in diameter. Before use, the glass beads shall be boiled throughly in several portions of water or until the wash water reacts neutral to methyl orange indicator.

Reagents

Alkali:

6. The alkali shall be normal sodium hydroxide, the titer of which shall be expressed as mgm. KOH per ml. and standardized according to good practice.

Acids:

7. The acid solutions shall be N/2 sulfuric and approximately normal sulfuric acid; the former standardized according to good practice and its titer expressed as mgm. KOH per ml.

Ether:

8. Ethyl ether U. S. P. shall be used as the solvent.

Sodium Chloride:

9. The sodium chloride used shall be C. P. and neutral to methyl orange.

Methyl Orange:

10. A 0.1 per cent water solution of methyl orange shall be used as the indicator.

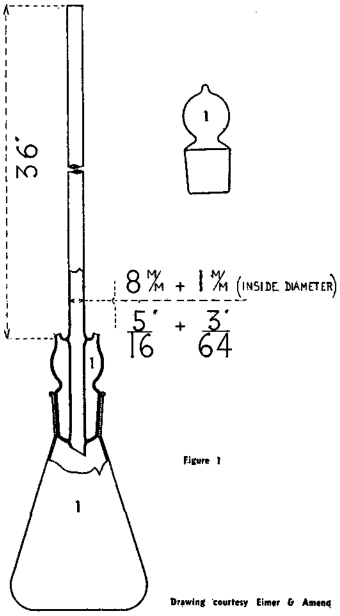
Procedure

General:

11. The procedure shall consist of two determinations, namely, (a) alkalinity of the sample, which shall be designated as "A," and (b) the increase in acidity after boiling the sample with sulfuric acid, which shall be designated as "F."

Reading of Burettes:

12. Burettes shall be drained for 3 min. before taking readings.



Drawing courtesy Eimer & Amend

Figure 1

Alkalinity (A):

13. Ten grams of the sample shall be dissolved in 100 ml. of water contained in the Erlenmeyer flask, warming to obtain solution if necessary. Thirty grams of sodium chloride, 25 ml. of ether, 5 drops of methyl orange, and N/2 sulfuric acid until slightly acid shall be added. The mixture during the titration shall be shaken frequently but gently until excess acid is present, when it shall be shaken vigorously. Normal or half-normal alkali shall now be added, several drops at a time, until the solution is again alkaline. The titration shall finally be completed with the acid, one or two drops at a time, shaking vigorously after each addition.

14. The number of ml. of the acid multipled by its titer, minus the number of ml. of the alkali times its titer, and divided by the weight of the sample shall designate the alkalinity "A," or

$$\text{Total alkalinity (A), mgm. KOH/gm.} = \frac{\text{ml. } H_2SO_4 \times \text{titer minus ml. NaOH} \times \text{titer}}{\text{Weight of Sample}}$$

Increase in Acidity Upon Boiling (F):

15. Ten grams of the sample shall be weighed into the flask and boiled for 1 hour and 30 minutes under the air condenser with excess normal sulfuric acid, using glass beads to prevent bumping. The amount of sulfuric acid shall consist of approximately the amount necessary to neutralize the alkalinity (A) of the sample plus 25 ml. in excess. The heating shall be so regulated that though boiling rather vigorously very little evaporation takes place. The contents shall be frequently agitated during the boiling. At the end of the heating period, the condenser shall be washed with a spray of water from a wash bottle, then disconnected from the flask, and the contents cooled. Thirty grams of sodium chloride, 25 ml. of ether, 50 ml. water, and 5 drops of indicator shall be added, and the mixture titrated with N/1 caustic soda to the same end-point as in the total alkalinity titration. The flask shall be stoppered frequently during the titration and the contents well shaken.

Blank:

16. A blank shall be run simultaneously with the sample, using the same amount and strength of the sulfuric acid, approximately the same weight of glass beads, and heating and titrating under the same conditions as the sample.

Calculation of "F":

17. The ml. of N/1 caustic soda required to titrate the sample minus the ml. required to titrate the blank, multiplied by the titer of the alkali, and divided by the weight of the sample shall designate the increase in the acidity "F," or

$$F = \frac{(\text{ml. NaOH to titrate sample minus ml. NaOH to titrate blank}) \times \text{titer}}{\text{Weight of Sample}}$$

The increase in acidity (F) may be negative, in which case the sign shall be retained in the formula given under "Calculation."

Calculation

Organically Combined Sulfuric Anhydride:

18. The algebraic sum of A plus F, multiplied by the ratio of SO_3/KOH, and multiplied by 1/10 shall be the percentage of organically combined sulfuric anhydride, or

$$\% \text{ Organically Combined } SO_3 = 0.1426\ (A + F)$$

and shall be reported as: ".... % organically combined SO_3 by weight, Titration Method."

Accuracy

Accuracy:

19. The accuracy to be expected with this method is that duplicate determinations of organically combined SO_3 shall not differ from each other by more than 0.05 ml.

of normal solution, or equivalent to about 0.04 per cent of combined SO_3.

Proposed Standard Method of Test for Organically Combined Sulfuric Anhydride in Sulfated Oils

B—Extraction-Titration Method

Scope:

1. This method of test determines the organically combined sulfuric anhydride existing in a sample of sulfated oil by dissolving the sample in a solvent, acidifying, washing with saturated brine, boiling the residue with sulfuric acid after evaporating the solvent, and titrating the products of reaction. This method applies only to sulfated oils that split off their combined SO_3 upon boiling with mineral acids, including samples containing sodium acetate or other compounds that cannot be accurately titrated in water solution with methyl orange as the indicator.

Apparatus:

2. The apparatus shall be the same as in the "Titration Method."

Reagents:

3. The reagents shall be the same as in the "Titration Method" and the "Ash-Gravimetric Method."

Procedure

General:

4. The procedure shall consist of isolating the purified oil and determining the increase in acidity upon boiling it with sulfuric acid. This increase shall be designated as "F."

Reading of Burettes:

5. Burettes shall be drained for 3 min. before taking readings.

Separation of Purified Oil:

6. Ten grams of the sample shall be weighed into 250-ml. pear-shaped separatory funnel containing 50 ml. of sodium chloride solution, some solid salt, 5 drops of methyl orange indicator, and 50 ml. of ether. The mixture shall be shaken and neutralized with approximately normal sulfuric acid until the lower layer is distinctly pink (about 0.2 ml. excess). After settling for at least 5 minutes, the lower layer shall be drawn off into another separatory funnel, and the ether layer washed with 25-ml. portions of salt solution until practically neutral to methyl orange—i.e., until one drop of N/2 caustic soda turns the wash water strongly alkaline. All separations shall be allowed to settle for at least 5 minutes. The water layers shall then be combined, extracted with two 25-ml. portions of ether, and the last two ether extractions combined and washed with salt solution until free from acid, as with the ether layer in the first funnel. All the ether layers shall now be combined in the decomposition flask and the ether evaporated.

Increase in Acidity Upon Boiling (F):

7. The procedure shall be the same as in the "Titration Method."

Blank:

8. The blank shall be run the same as in the "Titration Method."

Calculation of "F":

9. The calculation of "F" shall be the same as in the "Titration Method."

Calculation

Organically Combined Sulfuric Anhydride:

10. The per cent organically combined SO_3 shall be $\frac{F}{10}$ multiplied by the ratio of SO_3/KOH, or

$$\% \text{ Organically Combined } SO_3 = 0.1426 \times F$$

and shall be reported as "....% organically combined SO_3 by weight, Extraction-Titration Method."

Accuracy:

11. The accuracy to be expected with this method is the same as for the "Titration Method."

Proposed Standard Method of Test for Organically Combined Sulfuric Anhydride in Sulfonated Oils

C—Ash-Gravimetric Method

Scope:

1. This method of test determines the organically combined sulfuric anhydride existing in a sample of sulfonated oil by dissolving the sample in a solvent, acidifying, washing with a saturated solution of salt, and ashing the purified extract. This method of test applies to all types of sulfonated oils, including true sulfonic acid oils and those containing sodium acetate or similar titratable compounds.

Reagents

Ether:

2. Ethyl ether U. S. P. shall be used as the solvent.

Sodium Chloride:

3. The sodium chloride shall be C. P. and used in a concentration of 25 per cent.

Sodium Sulfate:

4. The sodium sulfate shall be C. P. and anhydrous.

Methyl Orange:

5. A 0.1 per cent water solution of methyl orange shall be used as the indicator.

Procedure

General:

6. The procedure shall depend upon whether or not the sample contains ammonia.

In the Absence of Ammonia:

7. The procedure shall be the same as under Separation of Purified Oil in the Extraction-Titration Method, with the exception that instead of combining the ether layers in the decomposition flask, they shall be combined in the first funnel. Any water that may settle shall be carefully removed and the ether layer dehydrated as follows: 5 grams of anhydrous sodium sulfate shall be added to the ether layer, shaken vigorously for 5 minutes, and filtered directly into a 150 ml. beaker, placed in a water bath. The flask and filter shall be washed with ether until free from fat (absence of oil stains on filter paper upon drying) and the filtrate added to the beaker. To avoid creeping of oil, the beaker shall at no time be more than one-third full during filtering and washing.

The ether solution shall be evaporated until the volume has been reduced to about 20 ml. and the residue transferred to a tared 50-ml. crucible (high form). The crucible shall be immersed in a 100-ml. beaker filled with warm water, until practically all the ether has evaporated. The beaker shall be rinsed with 2-10 ml. and 3-5 ml. portions of ether, respectively, or until all the oil has been transferred to the crucible, each addition of rinsing-ether shall be made only after the ether of the previous portion has practically evaporated. The residue freed from solvent shall be gently burned and finally ignited to constant weight. To prevent creeping of oil and to hasten evaporation, the solution may be stirred with a glass rod; before the oil is burned, the rod shall be wiped clean with ashless filter paper which shall be added to the crucible. To oxidize traces of carbon or sodium sulfide that might form, the ash shall be moistened with 30% hydrogen peroxide and again carefully ignited to constant weight.

8. The weight of the ash, divided by the weight of the sample, and multiplied by 100 shall be the percentage "extracted" ash in the sample, or

$$\text{Percent ``extracted'' ash} = \frac{\text{Weight ash}}{\text{Weight Sample}} \times 100$$

In the Presence of Ammonia:

Five to eight grams of the sample shall be dissolved in 80 ml. of water in a 300-ml. beaker. Ten ml. of N/1 NaOH shall be added and the solution boiled gently until wet litmus paper no longer indicates ammonia. The solution shall now be cooled and transferred into a 300-ml. pear-shaped separatory funnel and about 35 grams of solid sodium chloride added, or enough to make finally a 25% salt solution. Five drops of methyl orange shall be added and the neutralization, extraction, etc., conducted as under the test "In the Absence of Ammonia."

Calculation

Organically Combined Sulfuric Anhydride:

10. The percent "extracted" ash multiplied by $2SO_3/Na_2SO_4$ shall be the percentage of organically combined sulfuric anhydride in the sample, or

Per cent combined sulfuric anhydride = 1.1267 times per cent "extracted" ash

and shall be reported as: "....% organically combined SO_3 by weight, Ash-Gravimetric Method."

Accuracy

Accuracy:

11. The accuracy with this method is limited to the losses due to manipulation. Practice has shown that the accuracy to be expected is that duplicate determinations of the ash should not differ from each other more than 5 mgms.

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CORRECTION

In Oil & Soap, December, 1935, there was an error in the published formula for calculating Thiocyanogen Number, page 287, column 2, line 24, as follows:

(Blank — Titration) × $Na_2S_2O_3$ factor (I.V.) × 0.2 = Thiocyanogen Value (T.V.).

According to the original article of Martin and Stillman, Oil & Soap, 10, p. 30 (1933), this formula should read:

$$\text{T.V.} = \frac{(\text{Blank Titration}) \times (\text{Normality of } Na_2S_2O_3) \times (12.69)}{\text{Weight of Sample}}$$

or the calculation may be expressed as follows:

$$\text{T.V.} = \frac{(\text{Blank — Titration}) \times Na_2S_2O_3 \text{ factor (I.V.) } (\times 100)}{\text{Weight of Sample}}$$

when the $Na_2S_2O_3$ factor (I.V.) is expressed as grams of I_2/c.c.

THE PROCTER & GAMBLE OIL COLORIMETER

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A GREAT many attempts have been made in the last few years to apply photo-electric devices, spectrophotometers, color analyzers, and similar instruments to the determination of the color of fats and oils. Some of them have been quite successful. Yet in spite of these, the simplest, cheapest and most practical method for evaluating oil colors is the Lovibond system of color glasses, which, with modifications, has been adopted as standard by the American Oil Chemist Society.

The system is not without its faults. One of the chief drawbacks has been the large number of standard glasses required, and the difficulties and errors introduced in handling them. This has led to the development of several instruments in which the glasses are manipulated mechanically. The earliest of these was the colorimeter developed by H. S. Bailey in 1924. More recent instruments have been those introduced by the Precision Scientific Company, The Emil Greiner Company, and The Tintometer Limited, of England, the originators of the Lovibond system. None of these have exactly met the requirements of the Oil Chemists.

The Procter & Gamble oil colorimeter was first developed in 1927. The original instrument built at that time is still in daily use in one of the Ivorydale control laboratories. Recently the instrument was redesigned to meet the specifications embodied in the "Report of the Color Committee for 1933-1934." At the same time a number of mechanical improvements were made.

The colorimeter is essentially a modification of the original Bailey instrument. It consists of two main parts, the light box, and the color glass magazine.

The light box meets the specifications set forth in the "Report on Tintometer Standardization," in the August, 1931, issue of the *Journal of Oil and Fat Industries,* and modified in the "Report of the 1933-34 Color Committee." In this instrument the box is made of cast aluminum. One side is formed by a sliding panel which completely ex-