Determination of Total Fatty Acids

*Method for Soap Using Modified Stokes Flask**

By A. K. CHURCH†

THIS method is novel in that a specially designed extraction flask, a modified Stokes flask, suggested by members of our laboratory staff, is employed in extracting the fatty acids in place of the usual separatory funnel, thereby materially reducing the time required. When ether is mentioned ethyl ether is meant, unless otherwise specifically stated. When a Stokes flask is mentioned the specially designed modified Stokes flask is indicated. Prepare the sample in an acceptable manner—for example, by the A.C.S. standard method.

Separation of the Fatty Matter Containing the Fatty Acids

FIVE grams of the sample are weighed on the analytical balance, using an aluminum pan counterpoised with a brass weight except in the case of liquid soaps when approximately 5 grams are weighed from a weighing bottle. Transfer to a 100 cc beaker. Soaps containing perfume, or other volatile matter soluble in ether, must be dried overnight in a 105° C oven to drive off all such volatile matter before dissolving the soap in water.

Now dissolve in about 20 cc hot water and wash into the special Stokes extraction flask with a little hot water. Powdered soaps may be washed directly into the flask through a small funnel with about 50 cc cold water. 1 C additional water is added to bring the total volume of water in the extraction flask to about 75 cc. The flask is now placed on an asbestos mat on a hot plate and heated until complete solution (or disintegration) of the soap takes place when 10 cc 1: 1 hydrochloric acid is added to precipitate the fatty matter. Three glass beads are dropped into the flask, an air condenser (30″ long) attached, and the contents allowed to boil gently on an asbestos mat on the hot plate until the fatty matter forms a clear layer.

Extraction of the Fatty Matter Containing the Fatty Acids

COOL the flask by placing it in cold water, wash the condenser down with a little ether, remove the condenser and rinse it with ether. The amount of ether for these operations need not exceed 30 cc. The contents of the flask are well mixed to insure complete solution of the fatty matter in the ether. A 10% solution of sodium chloride (saturated with ether) is now added until the junction of the ether and water layers is just above the shoulder of the lower part of the flask. Insert the siphon in the flask so that the turned up end of the tube projects about ⅛″ above the surface of the water layer. Place the forefinger of one hand over the end of the short tube and hold the upper part of the flask with the other hand. This will cause the ether vapor to expand, and the ether solution of the fatty matter will siphon off into the weighed 150 cc flask. Add 10 cc ether to the extraction flask, shake gently and again siphon off the top layer. Now remove the siphon tube, add 15 cc ether, insert a wet rubber stopper in the extraction flask and invert the whole three or four times. Violent shaking is unnecessary. Allow the ether layer to separate, siphon off and repeat the extraction twice, each time using 15 cc ether. The total volume in the weighed flask will not exceed 90 cc of ether.

Evaporation of the Ether

PLACE the weighed flask containing the ether extract on a water bath and evaporate the ether, controlling the bath so that an excess of water vapor does not fill the hood,

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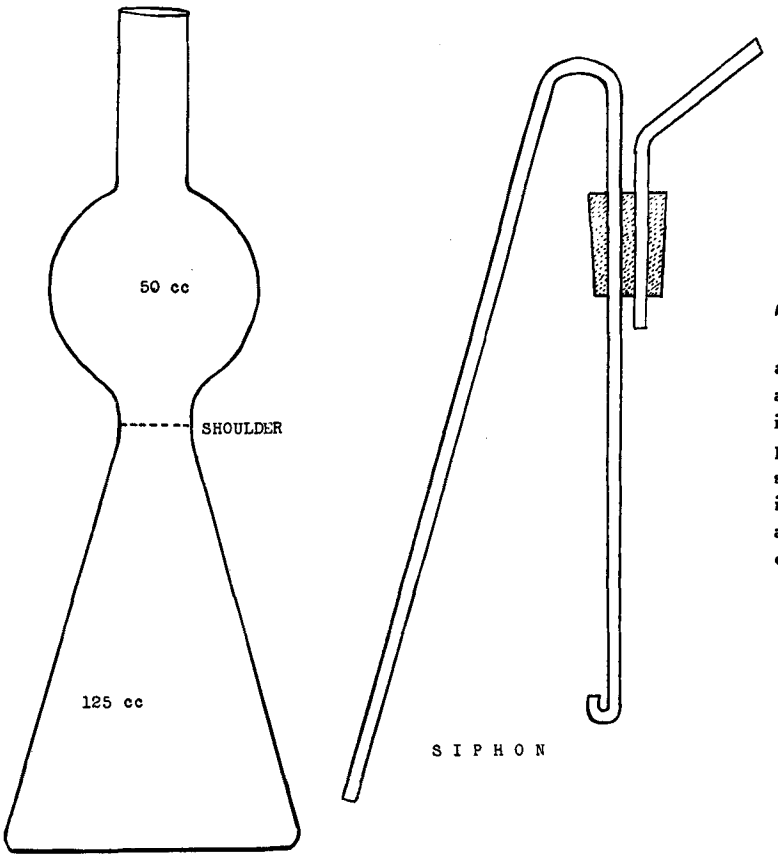

THE Modified Stokes Flask is eight inches high over all. It is three inches in diameter at the bottom and two inches in diameter at the widest point of the bulb above the shoulder. Below the shoulder, it has a capacity of 125 cc. and above the shoulder a capacity of 50 cc.

thus guarding against condensed water vapor getting into the flask. The last traces of ether vapor are blown out of the flask with a slow current of air and, if the manipulation has been carefully carried out, there should be no visible water in the bottom of the flask. When there is visible water present, the fatty matter cannot be weighed directly but may be weighed as a sodium soap.

Weighing Fatty Matter Directly

THE weighed flask containing the fatty matter is now heated in the 105°C air oven for just five minutes, no longer, cooled in a desiccator and weighed. Dry in 105°C oven for an additional 5 minutes to obtain check weight. The total fatty matter includes in addition to the fatty acids any unsaponifiable oil, neutral fat or other ether soluble matter present which must be determined and deducted to obtain the fatty acids. The percent of fatty acids times .97 gives the fatty anhydrides nearly enough.

Total Soap and Combined Alkali

AFTER evaporating off the ether as directed above, add 100 cc of neutralized alcohol free from carbon dioxide, add phenolphthalein indicator, and titrate to exact neutrality with standard semi-normal sodium hydroxide solution. Evaporate off the alcohol and water and dry at a temperature not exceeding 105°C to constant weight. At this stage the soap includes any unsaponifiable oil and neutral fat present, as well as the neutral salts from the sodium hydroxide and alcohol, all of which are determined separately and deducted from the total weight to obtain the true soap. Calculate the combined sodium oxide (Na_2O) and deduct from the weight of the true soap to obtain the fatty anhydrides. If the original soap is found to be wholly or partly potash soap, proper calculation must be made to reduce to potassium oxide (K_2O), or a mixture of K_2O and Na_2O, as the case requires. In case of the presence of free fatty or rosin acids in the soap, there must be determined and a proper correction made. If the soap contains rosin, determine the rosin

acids and substract the anhydrous rosin soap from the total anhydrous soap to obtain the soap from the fatty acids.

Unsaponifiable Matter and Neutral Fat

Results checking with the A.C.S. method are secured employing the Stokes flask and dissolving the soap in 50% alcohol and extracting with petroleum ether.

Rosin—Wolff's Method

THIS is the standard A.C.S. method except that the Stokes flash is used and a blank of .25 cc N/1 NaOH for each 3 grams of total fatty and rosin acids is subtracted from the titration. This blank was arrived at by running a test on pure soap known to contain no rosin. Dissolve enough of the soap sample to give close to 3 g of fatty and rosin acids in about 100 cc of hot water in the Stokes flask, add a slight excess of dilute sulphuric acid (1:4), heat until the fatty and rosin acids collect in a clear layer, cool to room temperature and extract with ether, following the procedure given under "The extraction of fatty matter." Siphon the ether layer into another Stokes flask. Evaporate off the ether and dry the fatty and rosin acids one hour at 105°C, cool and dissolve in 20 cc absolute alcohol. Then add 10 cc of a solution of one volume of concentrated sulphuric acid (Sp. Gr. 1.84) and four volumes of absolute alcohol and boil on the steam bath for four minutes under a reflux condenser. Remove from steam bath, cool and add about 30 cc ether. A 7 to 10% sodium chloride solution (saturated with ether) is now added until the junction of the ether and water layer is just above the shoulder of the lower part of the flask. Extract the ether layer as in the extraction of fatty matter, putting the extract into a 150 cc flask. Add 30 cc neutral alcohol, and titrate the rosin acids with standard sodium hydroxide solution, using phenolphthalein as indicator. For 3 g of fatty and rosin acids subtract .25 cc from the total NaOH required and calculate to rosin or rosin soda soap as desired (1 cc normal alkali equals 0.346 g rosin or 0.377 g rosin soda soap).

When the constants of the fatty and rosin acids are to be obtained, the rosin may be determined by using 3 grams of the fatty matter prepared for this purpose.

IT is recommended that for soaps made from all, or largely, cocoanut oil the fatty matter be weighed as a sodium soap.

Some Results Obtained

Soap made from	% Fatty Anhydride weighing fatty acids directly and multiplying by .97	% Fatty Anhydride modified A. C. S. method using Stokes flask and weighing the soap.	% Fatty Anhydride std. A.C.S method using separatory funnels & weighing the soap.
100% Tallow	83.63	83.65	83.65
25% Coco, 75% Tallow	82.90	82.94	82.94
35% Rosin, 65% Tallow	67.41	67.39	67.43
100% Cocoanut Oil	73.82	74.15	74.16
20% Coco, 20% Corn, 60% Tallow	74.13	74.16	74.16

Olive Oil Tariff

The United States Tariff Commission has issued Notices, pursuant to Section 336 of the Tariff Act of 1930, that a public hearing in Investigation No. 25 will be held at the office of the United States Tariff Commission in Washington, D. C., at 10:00 o'clock a. m. on the 27th day of January, 1931, at which time and place all parties interested will be given opportunity to be present, and to be heard with regard to the differences in cost of production and all other facts and conditions enumerated in Section 336 Of the Tariff Act of 1930 with respect to OLIVE OIL.

Early reports from the whaling vessels in the Antarctic fields were very favorable, provided the supply of whales holds out, the yield of whale oil this year should be the largest on record and a probable increase of several hundred thousand barrels over last year. Most of the Norwegian companies as previously reported have already sold next season's yield at a price of £25 per ton for the 0/1 grade. Based on the capacity of the factories these sales represent a total production of 1,400,000 barrels of whale oil.