late the sum to percentage retained on the No. 40 sieve.

3. Add the weight of the residue retained on the No. 12 and No. 40 sieves to the weight retained on the No. 100 sieve and calculate the sum to percentage of residue retained on the No. 100 sieve.

CONCLUSIONS AND RECOMMENDATIONS

Considering the difficulties of sampling and the friable characteristics in general of all soap powder products, it is believed that the screen tests as reported are in fairly good agreement. Most collaborators seemed to favor method B, or as an alternative method, the Ro-Tap procedure. Inasmuch as the present official methods do not include a method for screen tests, it is felt desirable to recommend B as a tentative hand screening method and method C as an alternative method for use where a large number of samples are to be examined.

GENERAL RECOMMENDATIONS

The present set of methods of soap analysis has been tentative for a period of two years. It is now recommended that all methods, with the exception of the screen and naphtha determinations above recommended for tentative adoption, be made official methods of the Society.

DETERMINATION OF VOLATILE HYDROCARBONS IN SOAPS

A CONTRIBUTION FROM THE LABORATORIES OF PROCTER AND GAMBLE

THE method for determining volatile hydrocarbons in soaps which is given in detail below is one that has been in use in the laboratories of Procter and Gamble for the last 15 years. In its development a considerable number of men have participated, so it cannot be credited to any one or two chemists, but can only be designated as a company method.

The method is not only applicable to volatile hydrocarbons which are lighter than water, but can be applied with simple modifications to any constituent in soaps and other materials, such as paints, that is volatile with steam, immiscible with water, and liquid at temperatures of condensing water. The volatile constituent may be heavier than water and collected in a measuring tube of the Bidwell Stirling type.

Method for Volatile Hydrocarbons

(Developed in the Laboratories of the Procter & Gamble Co.)

This method requires a source of dry, oil-free steam which is passed through the sample treated with acid, sufficient to liberate the fatty acids from the soap. The steam is next passed through strong caustic solution to scrub out any volatile fatty acids while the volatile hydrocarbons are condensed with the steam in a suitable arrangement which allows the excess water to flow away leaving the volatile hydrocarbon in the measuring burette. The method may be applied to samples containing substances immiscible with water and volatile with steam. For solvents heavier than water a Bidwell Stirling tube should be used.

Apparatus

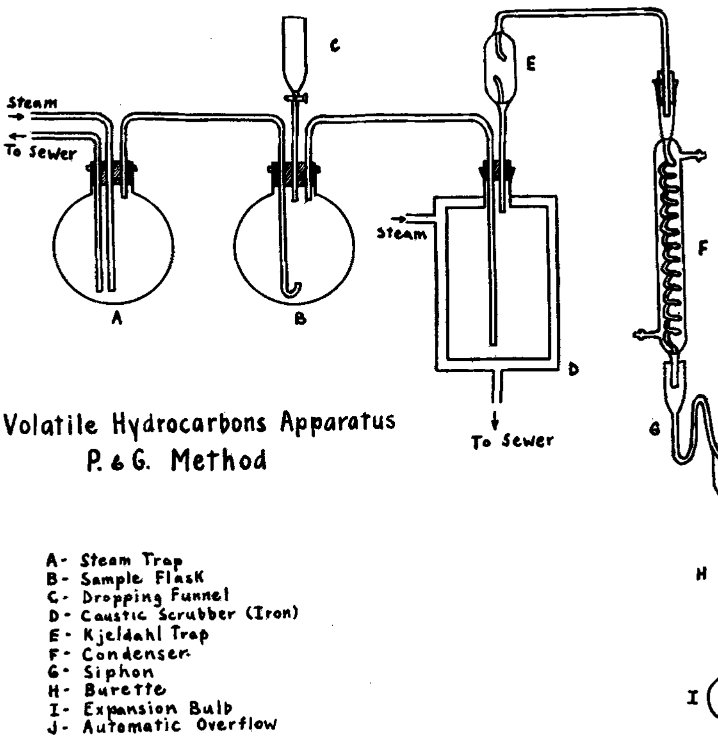
The apparatus and its arrangement are shown in the sketch. The following are the important items, lettered to correspond to the lettering of the sketch:

Steam Trap, A, a litre round bottom ring neck flask equipped with a siphon tube to the drain from the bottom of the flask and provided with a means of regulating the steam flow from the flask.

Evolution or sample flask, B, a litre round bottom ring neck flask. In case large samples are desirable the size of this flask may be increased.

Caustic scrubber flask, D, a steam jacketed metal flask is preferred, but a liter Florence flask provided with a steam coil of ⅛-in. copper tubing around the upper half may be used. If the glass flask is used it should be provided with a safety bucket below it and should be renewed frequently since the strong caustic dissolves the glass rather rapidly. This flask should be connected to the condenser by a Kjeldahl connecting tube, E, or similar safety device.

The inlet tubes for the steam into



Volatile Hydrocarbons Apparatus
P. & G. Method

A- Steam Trap
B- Sample Flask
C- Dropping Funnel
D- Caustic Scrubber (Iron)
E- Kjeldahl Trap
F- Condenser
G- Siphon
H- Burette
I- Expansion Bulb
J- Automatic Overflow

the evolution and scrubber flasks should extend nearly to the bottom of the flasks and be bent at right angles and parallel to the sides of the flask.

Condenser, F, a 12-in. or longer spiral condenser of sufficient bore so the condensate will not readily close it.

Measuring Burette, H, a 10 c.c. burette calibrated to 0.1 c.c. and carrying a bulb, I, approximately 100 c.c. capacity, at the lower end.

The stoppers used should be of a good grade of rubber and should have been thoroughly cleaned free from any surface sulfur and should be given a steam distillation in position for several hours before use on a sample.

Insulating the flasks and tubing to reduce condensation aids distillation and its control.

Determination:

Place 150 c.c. NaOH solution (about 46° Be') and several sticks of solid NaOH, to provide against dilution in the scrubber flask. Rinse out the condenser and burette with acetone. Attach a rubber tubing to the lower end of the burette, fill the burette and tubing with water and raise the outer end of the tubing so that the water level in the burette is near the top of the scale when the water is flowing to the drain from the automatic overflow, J. Be sure the connections are tight and that the tubing contains no air bubbles. Place the condenser in position so the lower end extends directly into the upper end of the burette just above the water level or connect to an adapter siphon, G, which discharges into the burette. The cooling water should be 15.5° C. or colder. Ice water may be desirable for low boiling hydrocarbons.

Weigh 100 ± 0.5 grams of the soap (cut into cubes of about 1 cm. edges) or 50 ± 0.3 grams of soap powder and transfer to the evolution flask. Add about 10 grams of gum arabic (commercial) and 100 c.c. of distilled water. Place the flask in position with 100 c.c. of 1:3 H_2SO_4 in a dropping funnel, C, carried in the stopper. Connect the steam, evolution, and wash flasks and condenser into position, making sure that the stoppers are tightly fitting and held in place by wiring. Rubber connections in the lines between the evolution flask and condenser should be avoided.

Add the acid to the sample slowly to avoid excessive frothing. While adding the acid, turn on the steam cautiously, so adjusting the pressure by a bleeder valve that just enough steam flows to prevent any liquid backing into the steam trap flask.

When all the acid has been added, turn on enough steam to cause brisk distillation, taking care that no liquid is carried over from the evolution and wash flasks and that the condenser water does not become warm.

Continue the distillation until there is no increase in the volume of the upper layer for 45 minutes or no small droplets can be noted in the condensate.

When distillation is completed, shut off and drain condenser water and allow the steam to heat up the condenser to drive out the last traces of volatile hydrocarbon. Shut off the steam as soon as vapor begins to issue from the lower end of the condenser. Immediately open the stopcock of the dropping funel to prevent caustic being drawn into the evolution flask.

Stopper the burette and allow its contents to come to room temperature or bring them to a definite temperature by placing the burette in a water bath held at 25° C. for one to two hours.

Read the volume of the upper layer to the nearest 0.01 c.c. The volume times the specific gravity equals the weight of the volatile hydrocarbon. The specific gravity should be determined at the temperature at which the volume is read. A small Sprengel tube made of 3 mm. glass tubing is convenient for this purpose.

Calculation:

$$\frac{\text{c.c. of volatile hydrocarbon} \times \text{specific gravity} \times 100}{\text{Wt. of sample}} = \% \text{ of volatile hydrocarbon}$$

For some samples the volatile hydrocarbon content may be so low that a larger sample than 50 or 100 grams is desirable. The size of the evolution flask may need to be increased if larger samples are used. The amount of water in the evolution flask and acid used should also be correspondingly increased.

THE COMPOSITION OF OITICICA OIL*

By R. S. McKINNEY
and
G. S. JAMIESON

IN the past there has been considerable confusion concerning the identity of the tree, the seeds of which yield oiticica oil, but it has been definitely established that the tree is Licania rigida of the family Rosaceae, which, as is well known, grows in northeastern Brazil. Circular No. 470 (1934) of the National Paint, Varnish and Lacquer Association, by Henry A. Gardner, contains much information in regard to the tree and discusses the commercial production of the oil, its properties and utilization in the manufacture of paint and varnish.

Until recently it was believed that the principal unsaturated constituent of this oil, known as couepic acid, was an isomer of elaeostearic acid. However, W. B. Brown and E. H. Farmer (Biochem. J. 29, p. 631, 1935), have shown that it is a gamma keto $\Delta^{9,11,13}$ octadecatrienoic acid ($C_{18}H_{28}O_3$) which melts at 74-75° C. They have suggested that from now on it be known as licanic acid because the oil is from the seeds of Licania rigida instead of from the tree Couepia grandiflora as formerly believed.

The present investigation was made on oils furnished by Dr. Gardner. One sample was raw oil

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*Contribution from the Oil and Fat and Wax Laboratory, Bureau of Chemistry and Soils, United States Department of Agriculture.