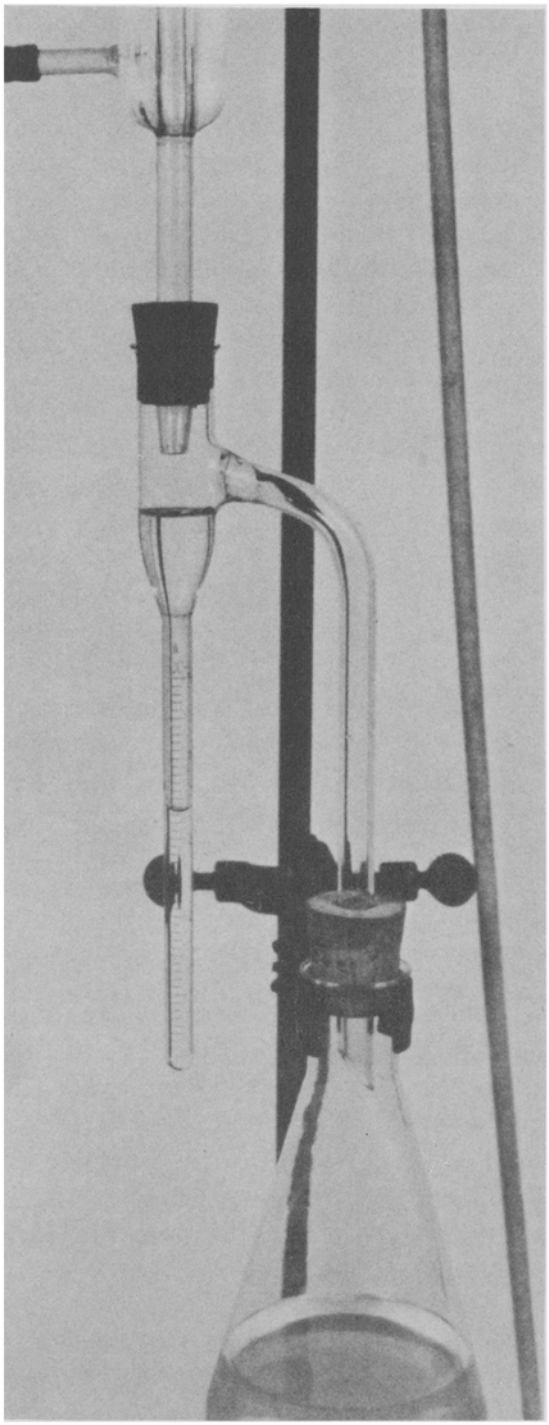
Determination of Moisture In Essential Oils

By R. M. REED

Procter & Gamble Co., Ivorydale, Ohio



Graduated Bidwell-Sterling Moisture Tube

THE Bidwell-Sterling toluene distillation method for the direct determination of moisture[1] has been successfully applied to a wide variety of materials. Hoyt and Clark[2] adapted this method to the determination of water in glycerol, while Church and Wilson[3] employed a similar method for the determination of moisture in soap. The experience of our laboratories confirms the value of the method as applied to both soap and glycerin. In addition, the Bidwell-Sterling method has been applied by the writer to a number of samples of essential oils and was found to be satisfactory for the determination of the moisture in such materials. Thus the method promises to be of wide application in laboratories concerned with the problems of soap manufacture.

Apparatus

The apparatus used is shown in the accompanying photographs. The Bidwell-Sterling moisture receiving tube is graduated in tenths up to five cc. and quantities of water may be estimated to the nearest 0.01 cc. with the aid of a lens. A 500 cc. Pyrex Erlenmeyer flask and a straight Liebig condenser are used. Cork stoppers, which are not affected by toluene vapors, are used in the apparatus.

Method

In carrying out the determination, 100 grams of oil are weighed into the flask and about 150 cc. of toluene added. The apparatus is assembled and the receiving tube filled with toluene, which is poured in through the reflex condenser. The flask is then heated until its contents distil into the moisture tube at a rate of about two drops per second. After the greater part of the water has been distilled into the receiving tube, the distillation rate may be increased somewhat and distillation continued until no more water is collected. The volume of the water in the receiving tube is

read after it reaches room temperature and the water content of the oil is then calculated.

Determinations which were made on various samples, both on the oils as received and after they had been saturated with water, are recorded in the accompanying table.

Moisture in the samples as received varies from 0.1 to 2.5 per cent. The moisture content of the saturated samples in most cases varies directly with the amount of free alcohols ($C_{10}H_{17}OH$) present. Rosemary samples (free alcohols 9.1 to 19.5 per cent) contain the least moisture, while a sample of Brazilian bois de rose oil, with a free alcohol content of 83.8 per cent, shows the highest percentage of moisture found in the series.

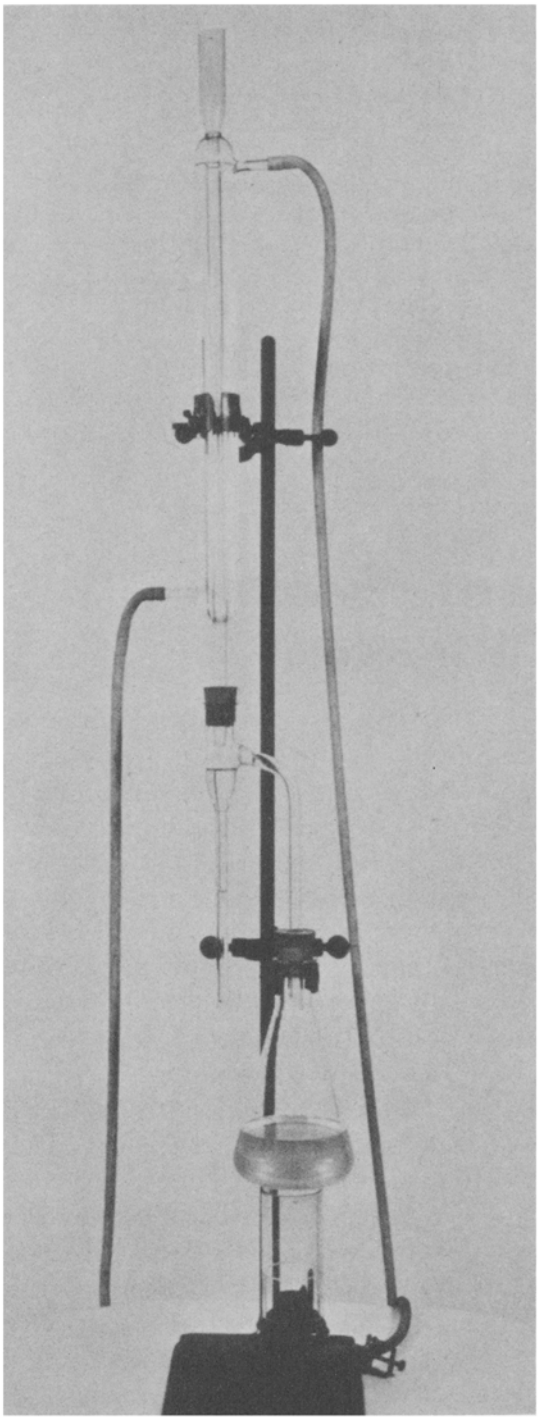
That the relatively high percentage of free alcohol shown by the citronella samples is partly due to acetylizable aldehydes explains the rather low moisture contents of those samples.

The results below are quite similar to those obtained by Glichitch[4]. He used xylene instead of toluene, and employed a somewhat more complicated distillation apparatus in carrying out the determination. Toluene is considered preferable to xylene, since its lower boiling point gives it the advantage of reducing the danger of decomposition of the oil.

This method is also similar to that used by Percy May in the determination of moisture in cloves[5]. The Bidwell-Sterling method is a modification of that of Dean and Stark, which is the one employed by May, who used petroleum distillate with a boiling range of 165-195°C. for distilling the water out of the cloves.

Bibliography

1. Bidwell and Sterling, Ind. Eng. Chem. 17, 147 (1925).
2. Hoyt and Clark, Oil and Fat Ind. 8, 2 (1931).
3. Church and Wilson, Soap 7, No. 11, 35 (1931). (Presented before American Oil Chemists' Society, Chicago, October, 1931.)
4. Glichitch, Les Parfums de France (1925), 351.
5. May, Perf. & Ess. Oil Record 17, 65 (1926).



Apparatus for determining moisture in essential oils

Sample	Free Alcohol Content, Per Cent	Moisture Content. As Received, Per Cent	Moisture Content. Saturated, Per Cent
1. Oil lavender, French	40.4	1.1	1.4
2. Oil lavender, French	....	1.0	1.4
3. Oil lavender, French	....	0.7	1.1
4. Oil lavandin, French	....	1.3	1.6
5. Oil rosemary, Spanish	19.5	0.5	0.6
6. Oil rosemary, Spanish	12.8	0.2	0.2
7. Oil rosemary, Spanish	12.3	0.3	0.5

(OVER)

8.	Oil rosemary, Spanish	9.1	0.3	0.4
9.	Oil rosemary, Spanish	14.4	0.4	0.5
10.	Oil bois de rose, Brazilian	83.8	2.5	3.2
11.	Oil citronella, Ceylon	58.5	0.8	1.4
12.	Oil citronella, Ceylon	57.4	0.9	1.1
13.	Oil citronella, Ceylon	56.9	0.9	1.1
14.	Oil citronella, Estate	63.9	0.6	1.1
15.	Oil citronella, Estate	59.0	0.7	1.2
16.	Citronellal	....	0.1	1.1
17.	Oil spike, French	41.7	1.8	1.8
18.	Oil spike, French	36.8	1.6	1.9
19.	Oil spike, French	31.9	0.8	1.7
20.	Oil spike, French	39.2	1.6	1.9
21.	Oil spike, Spanish	18.2	0.5	0.7
22.	Oil spike, Spanish	42.1	1.0	1.8
23.	Oil spike, Spanish	42.1	1.6	1.7

Acid Soaps

By H. BENNETT*

ALTHOUGH a few specialty soaps contain amines instead of alkalies, practically all commercial soaps consist of the alkali or alkaline earth salts of fatty acids, and are alkaline in reaction. The pH value of the amine type is lower than that of soaps made with alkalies.

Recently there have been introduced two new commercial soaps which not only are not alkaline but actually are faintly acid, having pH value of about 6.2. These acid soaps are diglycol oleate and diglycol stearate.

Diglycol oleate is an oily brown liquid of marked color, soluble in alcohol, esters and hydrocarbons, but insoluble in water. It is useful as a softening agent for rubber, resins and varnish gums. A permanently non-alkaline liquid soap, it will not affect colors or fibers. Because of its solubility in naphtha and other dry-cleaning solvents, it is available for the production of dry-cleaners' soaps. As an emulsifying agent it gives water-in-oil emulsions which are of advantage for automobile and furniture polishes. These emulsions can be inverted to the oil-in-water type by the addition of small amounts of alkali.

Diglycol stearate is an almost white waxy solid, melting at 58° C., somewhat soluble in cold alcohol and hydrocarbons, its solubility increasing rapidly with increase of temperature. Being free from alkalies and amines, it finds many applications as an emulsifying agent. In water heated to 60° C. or higher, diglycol stearate disperses readily. A 3 per cent dispersion gives a viscous milky stable fluid, while a soft white cream or paste is the product of a 10 per cent dispersion. Emulsions produced with these diglycol esters may be used for lubrication and to increase flexibility and lustre, on wool, cotton, rayon, leather, and paper.

The addition of four to six per cent of diglycol stearate to ordinary soaps for industrial washing is said to increase the detergent power of the soaps, producing lather of creamier consistency having small bubbles and greater stability. In toilet soaps this product promotes a smoothing effect upon the skin. When used as a superfatting agent in soaps, diglycol stearate tends to lighten the color of the soap, rather than darken it, as is so often the case with superfatting agents.

To increase the smoothness, flexibility and strength of wool during the various manufacturing processes that it undergoes, the wool is oiled, large quantities of oil emulsions being employed for this purpose. Where small quantities of a diglycol ester is incorporated with the oiling emulsion, many of the difficulties previously attributed to the oiling process are said to have been eliminated. The penetration of the oiling emulsion is increased by the addition

*Glyco Products Company, Inc.