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BUREAU OF CHEMISTRY—Circular No. 77.

H. W. WILEY, Chief of Bureau.

THE DETERMINATION OF CAMPHOR.

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The extensive use of camphor in medicine and the fact that the Pharmacopœia includes preparations which must contain definite quantities of camphor make it imperative that there should be a reliable method of assay. There have been in vogue for some time procedures depending on the rotation of an alcoholic, benzol, or oil solution and on the loss by evaporation, but they are open to objection, and in certain instances the results might easily be misinterpreted. Artificial camphor is without rotatory power, natural camphor might contain a portion of the levo body, the rotation varies with the strength of the solvent, and fixed oils themselves on heating often undergo loss or gain in weight. These are a few of the reasons which call for a method based on a more substantial foundation.

Camphor, being of ketonic character, forms with hydroxylamin a well defined oxim $C_{10}H_{16}NOH$, and advantage has been taken of this property in assaying camphor preparations, the procedure being based on Walther's¹ carvone estimation and on the work of Nelson,² who determined in essential oils by the hydroxylamin method a number of ketones including camphor. The procedure is simple and may be applied directly to spirits of camphor. Of the sample 25 cc are measured into an Erlenmeyer flask of 100 cc capacity, 2 grams of sodium bicarbonate are added, and then, accurately, from a burette, 35 cc of a hydroxylamin solution (20 grams $NH_2OH.HCl + 30$ cc $H_2O + 125$ cc absolute alcohol aldehyde free). The flask is connected with a reflux condenser, and heated to gentle boiling for two hours; it is then cooled to $25^{\circ} C.$, treated with a mixture of 6 cc hydrochloric acid (1.12 specific gravity + 6 cc water) transferred to a 500 cc volumetric flask, rinsing out the condenser and flask with water, and finally made up to volume; 50 cc portions are filtered off and titrated as follows: Methyl orange is added and the mineral acid neutralized with normal alkali, then phenolphthalein is added and the hydroxylamin hydrochlorid titrated with tenth-normal alkali. A blank must be run using the same amount of hydroxylamin solution and 25 cc of alcohol to correspond with the spirits of camphor, the difference in titrations representing the hydroxylamin converted into camphor oxim. Each cubic centimeter of tenth-normal sodium hydroxid is equivalent to 0.01509 gram of camphor.

¹ Pharm. Centralhalle, 1900, 41: 613.

² U. S. Dept. Agr., Bureau of Chemistry Bul. 137, p. 186.

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