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Micro-Gasometric Determination of Water in Hydrated Salts.

By

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With 1 Figure.

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Notevarp\(^1\), Rosenbaum and Walton\(^2\) have used calcium hydride to determine the water content of organic liquids. Perryman\(^3\) has used anhydrous dioxane as a water extractant and suspending medium in conjunction with calcium hydride for a microdetermination of water in several liquids and powders.

A gasometric method is described by Elitsur\(^4\), \(^5\), \(^6\), \(^7\), \(^8\) in which powdered calcium hydride is used in conjunction with either anhydrous ethanol, pyridine, or dioxane as an intermediary liquid for the determination of water in hydrated salts and blood serum.

The method of Elitsur has been modified by the adaption of a manometer-buret assembly from the Soltys\(^9\) apparatus for the determination of active hydrogen and by the design of a special reaction vessel with a steam-heated jacket.

The method is based upon the extraction of water from hydrated salts by anhydrous pyridine and quantitative interaction of the water and calcium hydride.

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\text{CaH}_2 + 2 \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 + 2 \text{H}_2.
\]

To prevent the formation of stable pyridine hydrate, \(\text{C}_5\text{H}_5\text{N} \cdot 3 \text{H}_2\text{O}\) (b. p. 93\(^\circ\) C) the reaction mixture is heated. Calcium hydride is available in a state of high purity and only 1.1683 mg of it is stoichiometrically required for 1 mg of water, but for the actual analysis it is preferable to use five or six times as much.

The stoichiometric equivalent is one millimeter of hydrogen at S. T. P. per 0.80376 mg of water.

Apparatus.

The apparatus, Fig. 1, consists of a manometer-buret assembly with a rack and pinion device for the leveling bulb. The buret (B) is graduated in 0.02-ml intervals from 0 to 7 ml. The manometer (A) has graduations corresponding exactly in position to those on the buret. The apparatus is filled with dry mercury.

The buret is connected with the atmosphere and side arm leading to the reaction vessel through the three-way T-bore stopcock. The reaction vessel (D) of approximately 30 ml capacity is connected to the side arm of the buret by a 14/20 ground-glass joint which is secured by two steel springs. The reaction vessel is surrounded by a steam jacket which is connected to a steam line or steam generator.

The stopper (E) and side arm of the reaction vessel are equipped with a 10/12 ground-glass joint which is secured by a steel spring. The stopper extends into the reaction vessel and has a groove near the end for the wire handle of the sample cup.

The glass cup (F) for the sample is provided with a tight-fitting ground-glass stopper which prevents loss or gain of water during weighing. The cup has an inner diameter of 5 mm and a height of 15 mm. The cup fits into a Nichrome belt (G) with a Nichrome wire handle, so that it may be suspended on stopper (E).

Stirring is performed by a glass-covered steel rod of about 5 mm diameter and 18 to 20 mm length, which is made to spin by a commercially available rotary type magnetic stirrer.

Procedure.

The ground-glass joints of the apparatus are thoroughly lubricated, and the upper outlet of the buret is connected to a calcium chloride guard-tube for the exclusion of water vapor from the interior of the apparatus. The three-way stopcock is turned to connect the buret and the reaction vessel to the atmosphere. The mercury level in the buret is raised as close to the stopcock as possible.