THE PREPARATION OF AMMONIUM HYDROXIDE FOR LABORATORY USE

LLOYD E. WEST and ARTHUR WILSON *Oregon State College, Corvallis, Oregon*

KOBE AND MARKOV[1] have described a process for the preparation of ammonium hydroxide for laboratory use in which ammonia gas is bubbled through water. Due to the exothermic nature of the reaction the solution becomes heated above room temperature and the resulting decreased solubility necessitates a series of absorption bottles to prevent the loss of ammonia to the air. In this paper a simpler apparatus is described in which cooling arrangement shortens the time of preparation and avoids the use of a series of absorption bottles.

[1] KOBE AND MARKOV, J. CHEM. EDUC., **18**, 29 (1941).

APPARATUS

A tank of liquid ammonia is connected by rubber

tubing to a 40-liter carboy containing 25 liters of water. The 12-mm. glass tubing through which the ammonia is passed into the water is curved to somewhat conform with the curvature of the carboy, Figure 1. The end of the tube is about one inch from the bottom and one inch from the side of the carboy. The exit tube from the carboy is led into a tall hydrometer jar filled with water to which 5–10 drops of phenolphthalein have been added.

The cooling device is a hollow iron ring, such as a circular gas burner, which has numerous small holes around its circumference. The ring is placed over the neck of the carboy and cold tap water run through it so the water sprays evenly over the shoulders of the carboy. A few towels are wrapped around the carboy to further increase the cooling efficiency.

PREPARATION

The apparatus is arranged as shown in Figure 1. The carboy is set in a sink or near a drain and tap water run through the cooling ring continuously. The valve on the ammonia tank is opened wide or just less than to where "bumping" in the carboy is objectionable. If the ammonia valve is left open continuously for many hours a layer of frost collects on the outside of the tank, thus reducing the rate of flow of gas. In such case the rate may be increased again by warming the tank with water. The authors usually found it more convenient to operate the absorption for only three or four hours at a time, thus avoiding excessive cooling of the tank.

After about 16 hours of operation the volume of solution in the carboy has increased from 25 to approximately 40 liters. At about this time enough ammonia has passed into the hydrometer jar or tall cylinder, under a head of about 12 inches of water, to turn the phenolphthalein pink. The ammonia gas is then shut off and the specific gravity of the solution in the carboy is measured. A portion may be removed from the carboy, warmed to room temperature, and tested with a hydrometer. By rearranging the connections on the carboy the pressure from the ammonia cylinder may be used to start a siphon action.

If the hydroxide does not give the specific gravity usually desired, 0.9, the absorption is continued. During the final stages, the rate of flow of ammonia is reduced to minimize the loss of the gas from the carboy.

As a safety measure a water trap may be inserted between the tank and the carboy to prevent water from sucking back into the tank. However the authors prefer to partially close the tank valve and pull off the rubber tubing at the tank simultaneously with shutting the valve on the nozzle.

DISCUSSION

The mixing of the solution in the carboy is well done by the ammonia being led into the water in a horizontal direction near the edge of the carboy. The rising of the undissolved ammonia further aids the mixing.

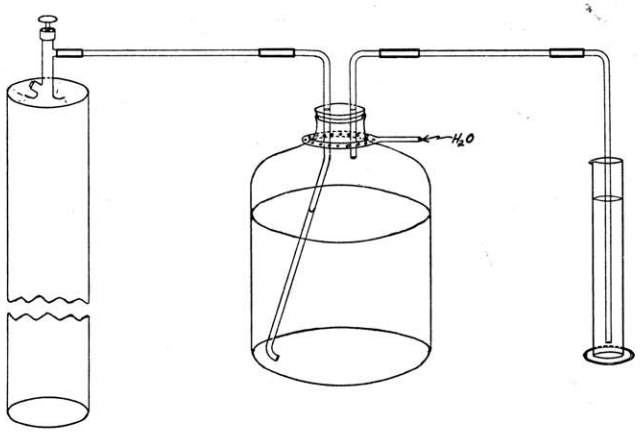

FIGURE 1.—APPARATUS FOR THE PREPARATION OF AMMONIUM HYDROXIDE

The cooling ring, the most unique part of the preparation, was an old burner designed for a small still. Similar burners may be obtained from any chemical supply house at a nominal cost.

During one preparation the hydrometer jar was replaced by a four-liter flask of water. About 40 liters of solution, specific gravity 0.9, were prepared by passing ammonia for 17–18 hours. The ammonia dissolved in the flask was found by titration to be 17.6 g. This value represented a loss of 0.17 per cent of the 10,680 g. of ammonia dissolved in the carboy. The cooling tap water was 12°C.

Some laboratories prepare ammonium hydroxide from ammonia in the winter months and store some for summer when it is impossible for them to prepare it by their present methods. Using the arrangement described in this paper such laboratories could release many carboys. A national shortage of carboys has recently been reported.