

labor required for the preparation, as well as the loss of ether, is much reduced by the following procedure.

To 3 l. of U.S.P. ether is added 450 g. of technical flake sodium hydroxide, and the mixture is allowed to stand at room temperature (25–30°) for two weeks with occasional shaking. After the first day the liquid becomes yellow and the sodium hydroxide appears somewhat powdery. After a week the color has nearly disappeared from the ether, but the sodium hydroxide has become yellow or brown. In about two weeks the ether is colorless and may be used directly for most purposes which require absolute ether, such as Grignard reactions. Since the non-volatile residue is very small (5 cc of the ether thus prepared left <0.01 mg of residue dried at 40°, or <0.032%), distillation can ordinarily be omitted. The ether can be decanted and stored over sodium with very slight evolution of hydrogen.

The sodium hydroxide can not profitably be used for a second lot of ether without purification. Smaller proportions of hydroxide to ether result in lengthened time and eventually incomplete decolorization. Other processes using sodium or potassium hydroxide for drying ether are described in patents (Hammond, U. S. 1,466,435 and 1,466,436 (1923) and others). The ether is best stored over a small amount of sodium in bottles at least three quarters full to minimize "breathing" with change in temperature. Under these conditions, no peroxide formation has been observed. The cost of absolute ether made by this method is much less than the current price, and the quality, judged by its behavior both toward sodium and toward dilute permanganate in strongly alkaline solution,² is better than that of commercial grades.

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THE USE OF CARBON DIOXIDE IN THE PREPARATION OF SILICIC ACID JELLIES

In studies on the growth of hydrogen-oxidizing bacteria on silicic acid jellies certain difficulties in the preparation of the jellies have been overcome by neutralizing the silicate with carbon dioxide. To 16 ml of nutrient solution in 6- or 12-oz. glass bottles is added 2 ml of a potassium silicate solution which has been made normal with respect to titratable alkalinity. A sufficient quantity of a mixture of normal hydrochloric, phosphoric and sulfuric acids is then added to give a reaction of approximately pH 8.0. After the bottles

² G. S. Forbes and A. S. Coolidge, *Jour. Am. Chem. Soc.*, 41: 152, 1919. Commercial U. S. P. ether, absolute ether and the product above gave the following reaction times: 3, 12 and 20 seconds, respectively.

containing this liquid medium have been evacuated to a pressure of about 7.5 cm of mercury, a gas mixture consisting of 60 per cent. hydrogen, 20 per cent. oxygen and 20 per cent. carbon dioxide is run in to equalize the atmospheric pressure. The bottles are placed in a horizontal position, and within twenty or thirty minutes the carbon dioxide has been absorbed and the silicate has set to a firm transparent medium with a reaction of approximately pH 7.0.

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