products, with a somewhat higher formation of tarry products.

UNIVERSITY, VIRGINIA RECEIVED AUGUST 9, 1935

The Preparation of Ethylene Imine from Monoethanolamine

BY HENRY WENKER

In view of the readiness with which compounds containing a β-aminoethyl group can be obtained from ethylene imine, a method may be of interest by which 80 g. of this base can be prepared conveniently in the laboratory in one day from inexpensive commercial material. The process consists, firstly, in preparing β-aminoethyl sulfuric acid[1,2] by thermic dehydration of monoethanolamine acid sulfate according to the equation

$$OH{-}C_2H_4{-}NH_2{\cdot}H_2SO_4 \longrightarrow \begin{matrix} CH_2CH_2NH \\ | \quad\quad | \\ O{-}SO_2{-}O \end{matrix} + H_2O$$

secondly, in distilling this compound with alkali.

An equimolar mixture of monoethanolamine and sulfuric acid is heated rapidly over a free flame until, at a temperature of about 250°, charring begins, necessitating the end of the operation. Only 75% of the mole of water indicated by the formula can be expelled since, irrespective of the rate of heating, the product begins to turn brown rapidly when this degree of dehydration has been reached. As much as 10 moles—610 g., of monoethanolamine and 1020 g. of 96% sulfuric acid—may be used for one operation. On cooling, the thin, clear brown liquid solidifies to a hard, white crystalline cake. It is ground in a mortar with one-half its weight of 60% ethanol, filtered by suction and washed with ethanol; yield, 100 g. per mole of starting material or 71%. It is convenient to dilute both components with their own weight of water previous to mixing, and then to boil off the excess of water.

282 grams of β-aminoethyl sulfuric acid is distilled with 880 g. of 40% caustic soda solution from a 3000-cc. flask. Shortly before the boiling point is reached, a reaction occurs which keeps the mixture boiling for several minutes; during this time, heating must be discontinued. When the reaction ceases, heating is resumed and a total of 240 cc. is distilled over. One distillation requires about forty-five minutes. On addition of potassium hydroxide to the distillate, 65 cc. of base separates; this is dried repeatedly with potassium hydroxide, then with sodium, and finally fractionated. The use of an efficient distilling column is recommended since the crude base contains higher boiling by-products; yield, 23 g. of ethylene imine boiling at 55–56.5° or 26.5%.

(1) Gabriel, *Ber.*, **21**, 1056, 2667 (1888).

(2) Fraenkel and Cornelius, *ibid.*, **51**, 1660 (1918).

ELIZABETH, N. J. RECEIVED JULY 29, 1935

An All-Glass Valve

BY JOHN WILLARD

An all-glass valve for use in systems where stopcocks are objectionable is illustrated in Fig. 1. It is easily constructed and may be opened and closed by merely heating a quartz tube which surrounds a fine Pyrex capillary. It is particularly useful for admitting a corrosive but thermally stable gas from a reservoir to a reaction.

The tube D is of quartz, about 6 mm. in diameter. Part C is a quartz-to-Pyrex graded seal and the portion of the device above C is of Pyrex. E is a fine Pyrex capillary. The outlet A is connected to a gas reservoir and the outlet B to an evacuated system to be filled from the reservoir.

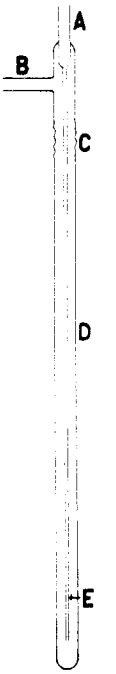

Fig. 1.

A small flame from a hand torch applied to the quartz near the tip of the capillary, when there is only a few mm. greater pressure on the A side than on the B side, will cause the capillary to blow out without affecting the quartz. When pressure has been equalized the capillary may be sealed again by similar application of heat. Repeated openings and closings of the valve may be carried out without difficulty if the capillary is long and fairly small even though it may stick to the side of the quartz tube. During an investigation in this Laboratory this device has been in constant use for several months. As many as twenty fillings have been made with it before it was necessary to seal in a new Pyrex capillary.

Alyea[1] has developed a valve which is similar in purpose to the one here described but which is made completely of Pyrex glass. It is opened by the use of a magnetic pellet and closed by sealing off the outer tube and the capillary together. Other types of greaseless valves are described by

(1) Alyea, THIS JOURNAL, **52**, 1937 (1930).