[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF COLORADO]

The Preparation of Pure Triethanolamine (β, β', β''-Trihydroxytriethylamine)

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Very pure hydrochloride of triethanolamine can be prepared from technical triethanolamine as follows. The triethanolamine, with or without preliminary fractional distillation, is carefully neutralized with concentrated hydrochloric acid at 10 to 15° and a very slight excess of acid, as shown by litmus paper, added. In an hour the pure white crystals that have settled are filtered off with suction, thoroughly washed with 95% ethyl alcohol and dried for an hour at 110°; yield, 70%, based on the amount of triethanolamine present in the original mixture; m. p. 177–178°; Knorr gives 177°. Calculated percentage of chlorine, 19.10; found, 19.05.

The conversion of the hydrochloride into the free base with silver oxide or alcoholic solutions of sodium or barium hydroxide is difficult because of the solubility of the chlorides in triethanolamine or its ethyl alcoholic solution. For instance, using sodium hydroxide, on distilling off the ethyl alcohol, a small amount of the sodium chloride crystals was observed on the bottom of the flask and on attempting to distil the triethanolamine *in vacuo* much decomposition occurred. This observation led us to believe with Knorr[1] that the presence of any appreciable amount of chlorides causes decomposition on distillation.

Since sodium chloride is less soluble in the higher than in the lower alcohols, it was thought possible that isopropyl alcohol might be substituted with advantage for the ethyl alcohol in the saponification of the hydrochloride, provided the hydrochloride itself was sufficiently soluble in it. Measurements showed its solubility to be 1.1 g. per liter in isopropyl alcohol as contrasted with 3.0 g. per liter in ethyl alcohol, both at 65°.

One hundred grams of triethanolamine hydrochloride was then added to 400 cc. of isopropyl alcohol, to which an equivalent quantity of sodium hydroxide, quickly pulverized in a mortar, had been added. This mixture was refluxed for three hours on a water-bath and after standing overnight the precipitated sodium chloride was removed on a suction filter. The isopropyl alcohol was then distilled off at atmospheric pressure, after which the triethanolamine itself distilled over at 194–195° at 10 mm. pressure. A 90% yield of odorless, clear, pale straw-colored oil, specific gravity, 1.1239 at 20/20°, was obtained, which on titration with sulfuric acid showed a purity of 99%. Its nitrogen content determined on nine different samples by the Kjeldahl–Gunning–Arnold method was on the average 9.35% as against 9.39% calculated, with a maximum variation of 0.09%.

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(1) Knorr, *Ber.*, **30**, 909, 1492 (1897).