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Readily Available Anhydrous Ether Solutions of Hydrogen Chloride

Frequently in chemistry laboratories and demonstrations anhydrous ether solutions of hydrogen chloride are desired. Perhaps the most common use is in the formation of amine hydrochlorides. The usual method of preparation involves passing hydrogen chloride gas from a cylinder into anhydrous ether. To minimize corrosion, the metal control valve is ordinarily removed from the cylinder and cleaned immediately after being used. In small colleges the occasional need for a hydrogen chloride cylinder is generally not considered sufficient to justify its purchase. Even at large universities the obtaining and maintaining of the cylinders and control valves is considered a serious nuisance. Furthermore, the preparation of bulk amounts of anhydrous ether solutions of hydrogen chloride for use as a reagent in undergraduate laboratories is generally not attempted due to the hygroscopic nature of the solution. Consequently procedures calling for the use of these solutions are often omitted in undergraduate laboratories and even in graduate level organic qualitative analysis courses.

The procedure described here circumvents most of the problems usually associated with the preparation of anhydrous ether solutions of hydrogen chloride since it uses readily available materials and does not require anhydrous ether or a gas cylinder. Furthermore, the solutions can be made rapidly and each member of a class can easily make his own whenever it is needed.

The preparation can be made by simply adding anhydrous magnesium sulfate powder to a mixture of ordinary ether and concentrated hydrochloric acid. Removal of the solid by decantation or rapid filtration then yields a satisfactory solution of hydrogen chloride in ether. Addition of the magnesium sulfate in small portions accompanied by gentle swirling is suggested. The first portions tend to cake and the additions should be continued until the added material no longer cakes. The quantities most commonly used in this laboratory have been 5 ml of concentrated hydrochloric acid, 50 ml of ether, and 10–13 g of anhydrous magnesium sulfate.

An interesting didactic technique in an undergraduate organic laboratory is to have some students attempt to use benzene as the solvent after first successfully using ether. The resulting failure of benzene to contain a substantial amount of hydrogen chloride is in sharp contrast to the results obtained using ether. This comparison emphasizes the effect of the presence of an oxygen atom which can form hydrogen bonds with hydrogen chloride.

The procedure can be adapted for the preparation of ether solutions of other hydrogen halides. Experience in this laboratory indicates that concentrated hydrobromic acid may be successfully substituted for hydrochloric acid. One attempt to use hydroiodic acid was also successful when the resulting solution was used immediately, although more ether was required and the solution rapidly darkened upon standing.