

A Convenient Way To Generate Hydrogen Chloride in the Freshman Lab

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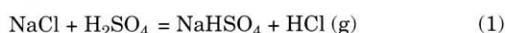
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Hydrogen chloride is an important chemical of interest not only by itself but also for preparative purposes.¹ Although available in cylinders of variable capacity and purity, generation of HCl gas is done frequently in chemistry labs.

Hydrogen chloride can be prepared by a number of methods including (1):

- dehydrochlorination processes
- reactions of chlorine with hydrogen and hydrogenated compounds
- hydrolysis and hydrogenolysis of some chlorides
- protonation of the chloride ion by strong acids, and
- liberation from aqueous solutions

However, most procedures found in preparative handbooks make use of concentrated H₂SO₄ (2, 3), for example:



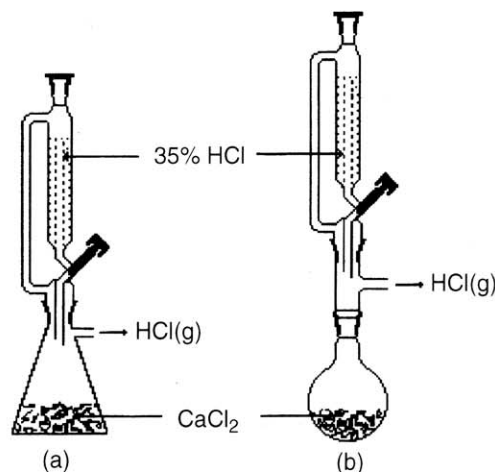
These procedures are the most universally adopted because they work satisfactorily with quite inexpensive chemicals.

Equation 1 can be explained in terms of a proton transfer from the strong (in the first ionization) nonvolatile sulfuric acid to the weakly basic chloride ion. It works at ambient conditions because HCl is a gas. The combination of acidity, redox properties, and low volatility, and cost of H₂SO₄ makes it the acid of choice. The process is conducted conveniently in standard glassware assemblies such as those in the figure. Concentrated H₂SO₄ is poured through the funnel, and salt is placed in the flask. Even though the generator is properly installed, losses of H₂SO₄ through the stopcock may be produced eventually.

In eq 2 H₂SO₄ behaves as a water-scavenging agent leading to a system unable to dissolve efficiently the HCl, so it escapes. The well-known dehydrating ability of H₂SO₄, together with the characteristics mentioned above, invite its use for this purpose.

However, because dehydration of hydrochloric acid can be achieved by reagents other than H₂SO₄ and because the handling of 98% H₂SO₄ in the freshman lab should be reduced as much as possible, especially when a significant amount of acid is involved, I want to recommend CaCl₂ for the following reasons:

- CaCl₂ is considerably safer to handle than concentrated H₂SO₄.
- It also is available at low cost, sometimes as a by-product of the Solvay process.
- The conventional assemblies for gas generation shown in the figure also are appropriate for the preparation of crude HCl(g).
- It is able to fix enough water (over the 30% of its weight considering it hydrates only to CaCl₂·2H₂O, stable up to 150 °C) to deliver a significant amount of HCl gas. In a typical run, 100 g of 35% HCl were added over 100 g of granular CaCl₂ at



Assembled apparatus for the generation of HCl(g).

room temperature (thermal effect is not significant), and 15 g of HCl(g) were obtained. This represents over 40% yield, which may be compared with the 80% yield reported (2) for the process represented in eq 2; however, it uses a weight ratio H₂SO₄/HCl (32%) ≈ 3.

- CaCl₂ also is easier to dispose of than H₂SO₄, and less base is required to neutralize the final waste.

An additional advantage of this procedure with respect to those based on H₂SO₄ concerns educational aspects. By heating at ~200 °C, the resulting waxy mixture (or crystalline mass to which it converts after a few hours), the anhydrous CaCl₂ can be regenerated for re-use once a solution of HCl is obtained. In this case, some calcium oxychloride formed can be ignored. Even though this may be without value from a practical viewpoint, it may be of educational significance because it attracts attention to topics of current interest such as optimizing of processes, recycling of chemicals, and reducing of wastes. To this end, the assembly shown in Figure 1b is more appropriate than that in Figure 1a because the resulting mixture is ready to be distilled.

Literature Cited

1. Pascal, P., Ed. *Nouveau Traité de Chimie Minérale*; Masson: Paris, 1960; Vol. XVI, p 194.
2. Maxson, R. N. *Inorg. Synth.* **1939**, *1*, 147.
3. Brauer, G. *Handbuch der Präparativen Anorganischen Chemie*; Ferdinand Enke, Ed.; Stuttgart, 1954.

¹See, e.g., The preparation of ThCl₄·8H₂O, TiCl₃·6H₂O, CrCl₃·6H₂O, (NH₄)₂[TiCl₆], CrCl₂, CuCl₂, ZnCl₂, CdCl₂, AlCl₃, AlCl₃·6H₂O, SiHCl₃, K₂[PbCl₆], (NH₄)₂[SeCl₆], ClSO₃H in ref 3.