THE METALLIC PRECIPITATION OF ZIRCONIUM

Sir:

The preparation of zirconium of a high degree of purity is not easy. The element combines or alloys itself with the reducing agents employed to prepare it. Probably the best method now used was devised by A. E. van Arkel and H. de Boer, who have obtained the metal by passing the vapor of the tetraiodide over a heated tungsten filament. Many metals may be prepared by precipitation from solution by a more electropositive substance than themselves. H. N. Warren[1] appears to have been the only one to employ this method for the preparation of zirconium. He states that if a rod of magnesium be wrapped with a few coils of asbestos paper and immersed in a solution of a metallic salt, the metal is precipitated on the asbestos in crystalline form. "Even zirconium was gradually reduced." This statement is erroneous, for zirconium compounds hydrolyze in water with the formation of free acid which would react with the magnesium.

It was thought, however, that zirconium might be precipitated from a solution other than aqueous. It was found that zirconium sulfate was quite soluble in methyl alcohol, the salt dissolving with a hissing sound and crystallizing out on evaporation. The alcoholic solution of zirconium sulfate was treated with metallic zinc. Hydrogen was given off, and the surface of the zinc was covered with a dark deposit. The reactions were accounted for by the equations

$$\mathrm{Zn} + \mathrm{H_2SO_4} \longrightarrow \mathrm{ZnSO_4} + \mathrm{H_2} \qquad (1)$$

$$\mathrm{2Zn} + \mathrm{Zr(SO_4)_2} \longrightarrow \mathrm{2ZnSO_4} + \mathrm{Zr} \qquad (2)$$

Since all zirconium sulfate contains a small amount of free sulfuric acid, introduced by methods of manufacture and by hydrolysis, the presence of hydrogen may be accounted for by Equation 1. As the reaction continued, the amount of hydrogen evolved decreased until no more gas was given off. The acidity decreased proportionally until at this point it took as much as five minutes for blue litmus to turn red. When a part of the deposit was scraped off and treated with hydrochloric acid, the zinc removed along with the deposit dissolved, leaving undissolved zirconium on the bottom of the container.

When magnesium was substituted for zinc, a white precipitate of undetermined composition was formed and no zirconium was precipitated.

The inability to continue this study for some time has caused this preliminary report to be prepared.

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[1] Warren, *Chem. News*, 61, 183 (1890).