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The Anion-Exchange Separation of Zirconium and Hafnium

E. H. Huffman and R. C. Lilly

August 3, 1949

Berkeley, California

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ABSTRACT

A successful anion-exchange separation of zirconium and hafnium has been attained by elution of their fluo-salts from Amberlite IRA-400 with hydrochloric acid containing a small amount of hydrofluoric acid.

The Anion-Exchange Separation of Zirconium and Hafnium

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The separation of zirconium and hafnium by cation-exchange has recently been reported.(1) The possibility of a similar separation by the exchange

(1) Kenneth Street, Jr., and G. T. Seaborg, J. Am. Chem. Soc. 70, 4268 (1948).

of complex anions of these elements was obviously presented, but earlier attempts to carry out such a separation in this laboratory had been unsuccessful, using the weakly basic anion-exchange resins first available. The later appearance of strongly basic resins has suggested a re-examination of the problem, and a successful separation is reported below.

The scheme used was to adsorb zirconium and hafnium in the form of fluo-ions from a solution in hydrofluoric acid and to elute them with dilute hydrochloric acid containing very dilute hydrofluoric acid. In the absence of hydrofluoric acid colloid formation takes place in hydrochloric acid of the concentration used, with subsequent sticking of the colloid particles to the resin. Although the resin used was Amberlite IRA-400, it is probable that other strongly basic resins available will serve the purpose.

Twenty milligrams of zirconium and 10 mg. of hafnium, in the form of their oxides, were added to zirconium and hafnium tracers in a small amount of hydrofluoric and nitric acids in platinum and warmed until dissolved, and the solution was fumed with 0.5 ml. of sulfuric acid to expel hydrofluoric and nitric acids. The residue was dissolved in water and the hydroxides were precipitated with ammonium hydroxide. The precipitate was centrifuged, washed with water, dissolved

in 5 ml. of 0.64 M hydrofluoric acid in a Lusteroid tube and diluted to 10 ml. with water. Six hundred milligrams of the resin, in the chloride form in which it was received and which had been ground to 200-325 mesh, was added to the sample in the tube. The tube was shaken for three hours, the resin was centrifuged and the supernatant liquid removed and tested. The tracer count indicated that 96% adsorption had taken place and this remained constant after the resin was washed well with 10 ml. of water. This portion of resin was slurried onto the top of a column of the same resin 30 cm. in length and 0.78 sq. cm. in cross section. The column was contained in Tygon tubing with Lucite fittings and cotton was used to hold back the resin at the lower tip. Elution with a solution of 0.2 M hydrochloric acid and 0.01 M hydrofluoric acid at the rate of 6 ml. per hour gave the results shown in Fig. 1. The solid parts of the curve were obtained by counting Zr^{95} (65 day) and Hf^{181} (46 day) tracers and the dotted parts by spectrographic analysis. The order of elution of the two elements is the reverse of that obtained by cation-exchange[1].

Combined fractions of elutriant from 300 ml. to 653 ml. containing 13.8 mg. of zirconium, or 69% of the starting material, were found to contain no detectable hafnium by spectrographic analysis. A similar portion from 300 ml. to 686 ml. containing 17.0 mg. of zirconium, or 85% of the starting material, was found to contain 0.04% hafnium. Spectrographic analysis of the 752-1020 ml. portion showed 0.02% zirconium in the 6.9 mg. of hafnium (69% of the starting material). Similarly, 0.03% zirconium was found in the 704-1020 ml. portion containing 8.3 mg. of hafnium (83% of the starting material). The amounts of the major constituents in these portions was determined from the curves.

Anion adsorption also serves to separate zirconium and hafnium from impurities which do not form negative fluo-ions, such as calcium, barium

and magnesium which frequently occur in commercial products. Such separations are being investigated.

This work was done under the auspices of the Atomic Energy Commission.

Fig. 1 --- Elution of fluozirconate and fluohafniate with 0.2 M hydrochloric acid and 0.01 M hydrofluoric acid.