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THE ION-EXCHANGE SEPARATION OF ZIRCONIUM AND HAFNIUM

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The Ion-Exchange Separation of Zirconium and Hafnium

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In the course of a rather cursory examination of the elution of tetra-positive ions from the cation exchange resin Dowex 50 with hydrochloric acid solutions, we have discovered a very effective method of separating zirconium from hafnium. In view of the great labor involved in preparing even reasonably pure hafnium compounds by existing methods, we feel that this procedure will prove very valuable to those interested in obtaining hafnium compounds free of zirconium.

Although the conditions which give satisfactory separation were first worked out using microgram amounts of material and the radioactive tracer technique, the run described below, involving milligrams of material, illustrates the applicability of the method to the production of significant amounts of pure hafnium and zirconium.

35 mg. of zirconium oxide 15 mg. of hafnium oxide were dissolved in sulfuric and hydrofluoric acids, hafnium and zirconium tracer added, and the mixture fumed to dryness. The residue was taken up in concentrated hydrochloric acid and the hydroxides precipitated with ammonium hydroxide and washed. The hydroxides were again dissolved in hydrochloric acid and the oxychlorides crystallized by evaporation. 1 cc. of 250 to 500 mesh Dowex 50 spheres, in the ammonium form, were suspended in 30 cc. of 2 molal perchloric acid and the oxychlorides added a few mg. at a time over a period of 15 minutes, the mixture being continually agitated by bubbling

air through it. Under these conditions, i.e.~0.01M zirconium and hafnium in 2M perchloric acid, the zirconium and hafnium are not appreciably polymerized[1] and about 80 percent of each goes on the resin. The slurry of resin was placed on the top of an ion-exchange column 1 sq. cm. in area and 30 cm. long which had been packed with the same resin and washed with 6 molal hydrochloric acid to convert it to the acid form. On elution with 6 molal hydrochloric acid the curve shown in Fig. 1 was obtained. The outline of the curve was obtained by counting the tracers (Zr^{95} and Hf^{181}) and the dotted portions by optical spectrographic analysis.

Thus it can be seen that ~66 percent of the starting hafnium oxide, i.e. 10 mg., is obtained containing ~0.1 percent zirconium oxide by weight. The column used in these experiments was relatively small and thus gram amounts of material should be easily handled on columns of only moderate size.

This work was done under the auspices of the Atomic Energy Commission at the Radiation Laboratory, University of California, Berkeley, California.

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1. Connick, R. E., and W. H. McVey, Private Communication.

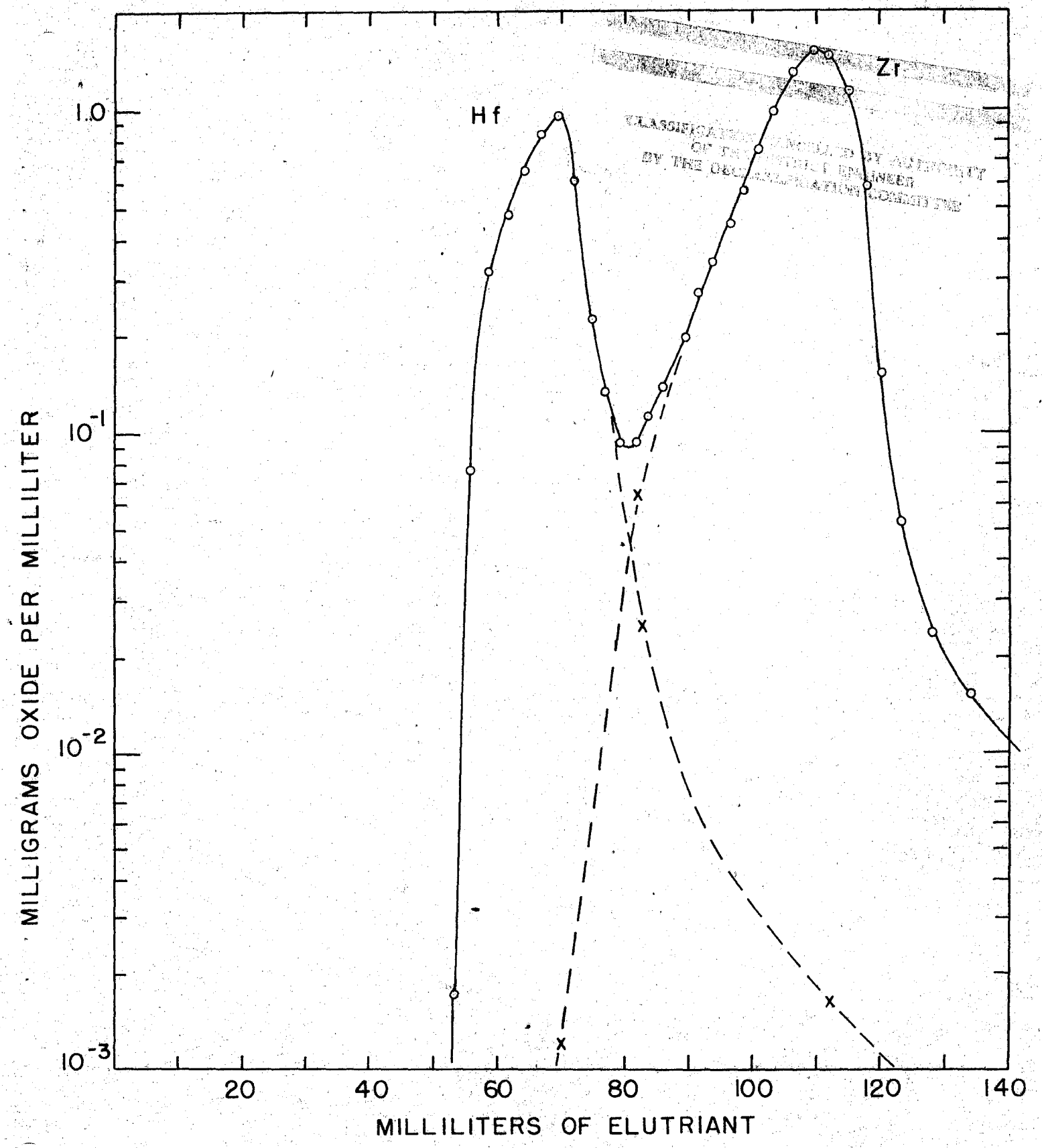

FIG. 1

ELUTION OF Zr AND Hf WITH 6.0 MHCl