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THE SEPARATION OF Zr AND Nb OXALATO COMPLEXES BY ANIONIC EXCHANGE

R. E. Wacker and W. H. Baldwin

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THE SEPARATION OF Zr AND Nb OXALATO COMPLEXES BY ANION EXCHANGE

by R. E. Wacker and W. H. Baldwin

The readily available form of radioactive zirconium[1] has been assigned the mass number 95[2]. It decays by beta and gamma emission with a 65 day half-life to a radioactive niobium daughter that in turn decays with a 35 day half-life by the emission of beta and gamma rays to stable molybdenum.

The separation of zirconium from the daughter niobium (in the carrier-free form) has been accomplished, in the past, by the carrying of niobium on MnO_2 precipitates[3] and by the extraction of zirconium from aqueous solutions into organic solutions with the help of a chelating agent[4].

The data of Venable and Baskerville[5] showing the existence of oxalic acid complexes of zirconium led to the inquiry into the distribution of Zr and Nb between anion exchange resins and oxalic acid solutions. Zr and Hf have already been studied in fluoride systems with Amberlite IRA-400[6] and Dowex-1[7]. It was found that Zr and Nb were adsorbed on Dowex-1 resin from oxalic acid solution (0.1 - 0.4M) and could be preferentially eluted (Zr being removed first) by elution with 1 MHCl and 0.01M oxalic acid solution.

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(1) Isotope Division - U.S.A.E.C. Oak Ridge, Tenn., Catalogue & Price List No. 3 - July, 1949.

(2) Siegel, J. M., J. Amer. Chem. Soc., 68, 2411 (1946).

(3) Connick, R. E. and McVey, W. H., AECD 2272, p. 8.

(4) Broido, A. and Tompkins, P.C., MonN-432, p. 65, Dec. 15, 1947; and CNL-37, p. 97, April 16, 1948.

(5) Venable, F. P. and Baskerville, C., J. Amer. Chem. Soc. 19, 12 (1897).

(6) Huffman, E. H. and Lilly, R. C., AECD 2714.

(7) Kraus, K. and Moore, G. E., J. Amer. Chem. Soc. 71, 3263 (1949).

Experimental

Tracers - Mixtures of Zr and Nb were isolated in 0.4M oxalic acid after separation from mixtures of all the fission products.

Resin and Column - Dowex-1 resin that had been pre-treated with 0.4M oxalic acid was wet-screened and the fraction passing the 80 mesh but retained by the 200 mesh screen was slurried into a glass tube (6 mm. i.d.) to a height of 85 cm.

Column Operation - A solution (as much as 10 ml) containing the mixture of Zr^{95} and Nb^{95} in 0.4M oxalic acid was placed in the top of the column and the liquid level allowed to fall nearly to the resin. At this time the feed flask containing a solution of 1M HCl and 0.01M oxalic acid was connected and all air bubbles were removed. The solution was permitted to flow at a rate of 0.4 cm/min. and the effluent stream was monitored by a modification of the device described by Ketelle and Boyd[8]. The fractions that were separated were identified by radiochemical techniques[9], adsorption curves and decay curves.

Discussion

The experimental results are presented graphically in Figure 1. The four bands of activity that were obtained by the elution have been qualitatively

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(8) Ketelle, B. H. and Boyd, G. E., J. Amer. Chem. Soc. 69, 2804 (1947).

(9) The authors acknowledge their indebtedness to Mr. T. H. Handley of the Analytical Department of the Chemistry Division, O.R.N.L. for the radiochemical identification of these fractions.

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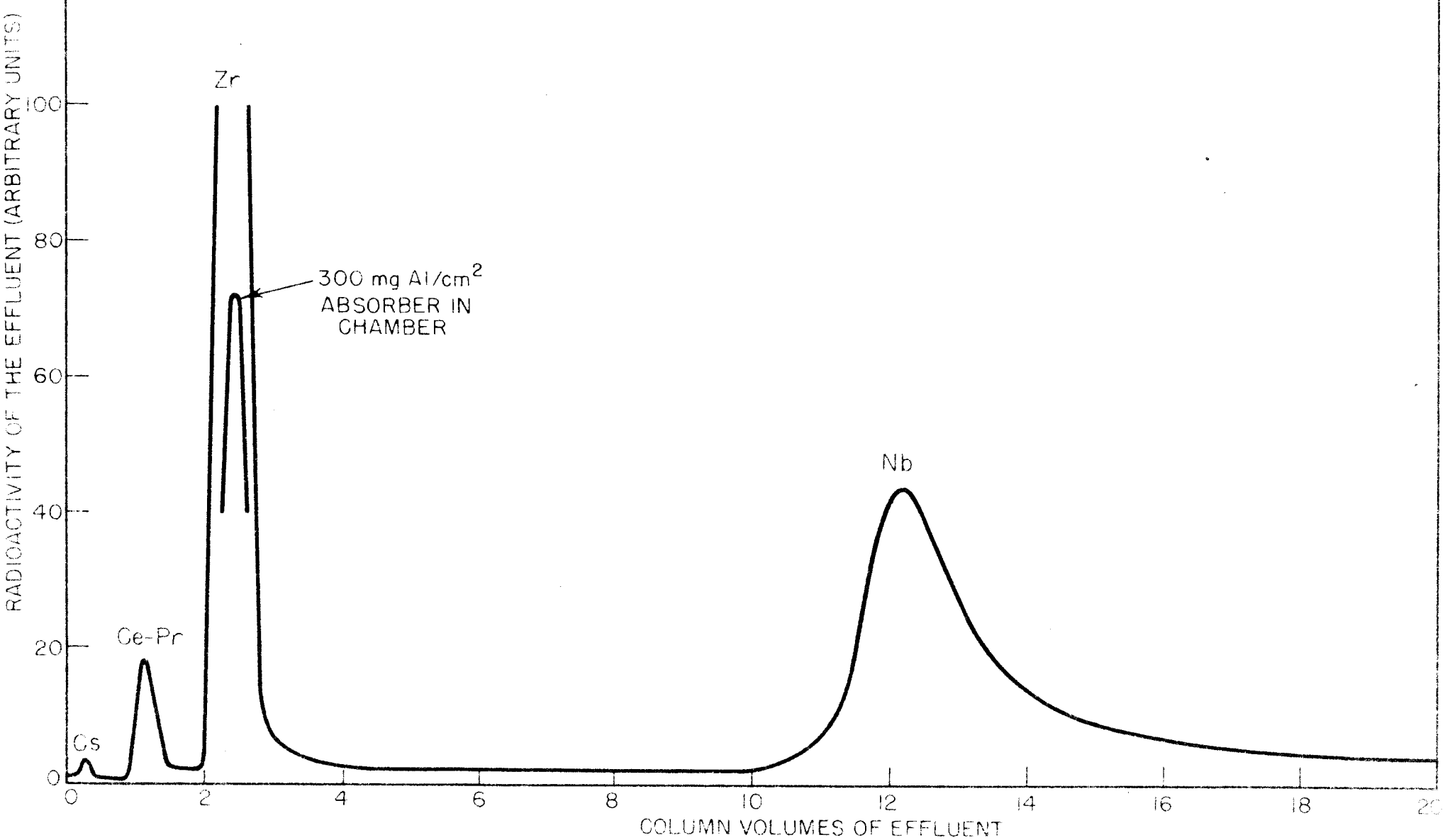
FIG. 1
SEPARATION OF Zr, Nb AND CONTAMINANTS
ELUTION OF OXALATO COMPLEXES FROM A COLUMN OF DOWEX 1 RESIN



identified as Cs, Ce-Pr, Zr and Nb.

It is to be noted that the recorder went off scale during the time when the Zr activity was being washed from the column. An absorber (300 mg. Al/sq.cm.) was placed between the solution and the chamber so that the recorder was returned to the scale. The absorber was removed when the reading returned to the scale.

Zr and Nb activities are separated routinely by this method and are used in tracer experiments.