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Use of Alumina as an Ion Exchanger in the Separation of Carrier-Free RaE from RaD

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NOTE

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Abstract

A very simple procedure for the radiochemical separation of carrier-free RaE (^{210}Bi) from RaD (^{210}Pb) has been developed. RaE as sulfate was adsorbed in powdered alumina column, which was later quantitatively washed out with hot conc HNO_3. The radiochemical purity of RaE had been proved by studying the β-decay half-life of the isotope.

INTRODUCTION

Separation of RaE in a carrier-free state from its mother RaD has been a problem for a long time. A study of earlier literature (*1*, *2*) reveals that RaE was usually separated from RaD by deposition on metallic foils in a reducing medium and then purified for bringing it to the carrier-free state. The process was complicated, and therefore techniques like chromatography (*3–5*) and ion exchange (*6*) had been carried out by many workers. The application of an ion-exchange resin (*6*) with subsequent elution with HCl was also not found to be satisfactory. Electrochromatographic separation studies (*7*, *8*) with inactive and nonisotopic carriers have been also made, but these were more complicated and involved a number of steps. Since bismuth undergoes hydrolysis in the presence of ammonium acetate, a separation method was developed (*9*) where bismuth was adsorbed in glass powder and later eluted with nitric acid. The authors (*9*), however, claimed 97% recovery of carrier-free RaE in this process.

In the present investigation an attempt has been made to utilize alumina column for separation of 100% RaE from RaD. Chromatographic alumina has been claimed by different authors (*10–15*) as a popular all-purpose adsorbent, uniquely suited for many separation problems, as well as for decontamination of the desired isotope in the case of the separation of fission products or others. Separation of ^{35}S, ^{32}P, and ^{36}Cl by using an alumina column showed (*16*) interesting findings. It was further thought that the unique adsorption behavior of alumina may be utilized for RaD-RaE separation by taking recourse to the fact that their sulfates differ in solubility in acids, crystalline forms, ionic radii, etc.

EXPERIMENTAL

Carrier-free RaD in equilibrium with RaE was procured from Philips Duphar. Chromatographic aluminum oxide of G.R.E. Merck quality was used in the investigation. The acids used were of AnalaR variety.

RaD in equilibrium with RaE in dilute nitric acid solution was first concentrated to a very small volume, 2 ml of concentrated H_2SO_4 was then added, and the solution was evaporated to fumes. Evaporation with H_2SO_4 was repeated twice, cooled, and 50 ml of water was added. The concentration of acid was about 0.5 *N*. The solution (pH $\sim$ 1) was aged for 1 hr over a steam bath and then fed into a glass column having an internal diameter of 1 cm and equipped with glass wool plugs both at the top and at the bottom and packed with 17 g of aluminum oxide. The column had a height of 28 cm. The column was surrounded by an outer glass jacket through which steam was circulated. This arrangement kept the alumina column hot during adsorption and the elution process. The eluate was collected in a conical flask connected to a water pump which regulated the flow of solution to a rate of 12 to 14 drops/min. The column was then washed with 10 ml of 0.5 *N* H_2SO_4 followed by 50 ml of distilled water. The collected solution was tested and no RaE could be detected whereas all the lead has been eluted out. RaE was thus found to be adsorbed in the alumina column.

The alumina column was then repeatedly washed with hot concentrated HNO_3 (in 30 ml portions). It was observed that the first washing brought down about 85% of the RaE. The residual amount could be collected by thoroughly washing with hot nitric acid (four or five times). Final recovery was 98 to 100%. The washings were mixed together, concentrated to a

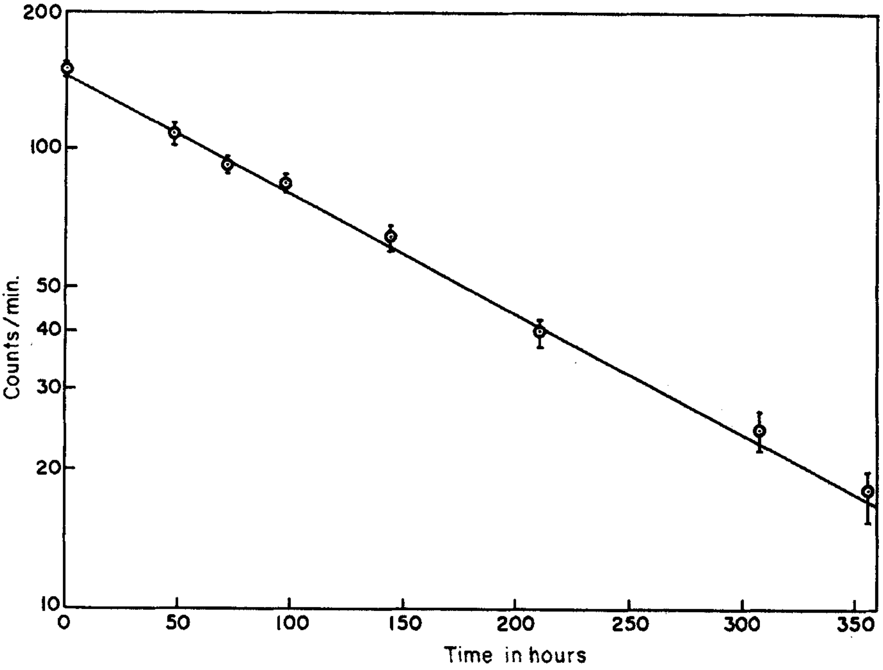

FIG. 1. Decay curve of RaE separated from RaD.

convenient volume, and a 10 ml portion was counted with a G.M. liquid counter. The results are shown in Fig. 1. The experiment was repeated and the findings were confirmed.

DISCUSSION

It is evident from the results that the RaE was retained undisturbed on the alumina column when a RaD-RaE equilibrium solution as the sulfate was fed (at pH ~ 1) into it and all of the RaD was eluted out. Washing with only 30 ml of hot concentrated HNO_3 striped off more than 80% RaE, and further washing gradually carried down the isotope quantitatively. The β-decay curve of RaE (Fig. 1) shows beyond doubt that the product is of high radiochemical purity. The overall procedure takes about 2 hr.

It is interesting to point out that the separation of tracer bismuth from tracer lead through the alumina column does not follow the macrobehavior since the insoluble lead sulfate comes down the column and the more soluble bismuth sulfate is retained in it, probably due to some complex adsorption character of alumina yet to be investigated. Steam circulation keeps the column hot, and this increases (*17*) the sample separability of alumina by converting RaE to the very soluble nitrate form. It was also observed that the application of hot dilute HNO_3 (1:1) elutes out RaE only partially from the alumina surface; the complete removal of the isotope requires more time and a large quantity of dilute nitric acid. In the washing with hot concentrated HNO_3, a small amount of alumina accompanies the RaE. The collected washings were evaporated to a small volume and the solid matter that separated out was filtered, made nitrate-free, and subsequently washed with water. RaE was measured in the collected washings.

The foregoing discussion indicates that chromatographic alumina can be utilized successfully for similar yet unexplored separation problems.

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