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Use of Alumina as an Ion Exchanger in the Separation of Carrier-Free ^{140}La from ^{140}Ba

D. K. Bhattacharyya[a]; S. Basu[a]
[a] NUCLEAR CHEMISTRY DIVISION, SAHA INSTITUTE OF NUCLEAR PHYSICS, CALCUTTA, INDIA

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NOTE

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D. K. BHATTACHARYYA and S. BASU

NUCLEAR CHEMISTRY DIVISION
SAHA INSTITUTE OF NUCLEAR PHYSICS
CALCUTTA-9, INDIA

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Abstract

Radiochemical separation of carrier-free ^{140}La from ^{140}Ba has been performed by adsorbing the barium separately as sulfate and chromate in a powdered alumina column while lanthanum was eluted out. In both cases the adsorbed barium activity was later washed out with hot dilute nitric acid. Radiochemical purity of the separated lanthanum product was ascertained through β-decay studies.

INTRODUCTION

Separation of ^{140}La (40 hr), the daughter activity derived from ^{140}Ba (12.8 days) has long been of importance because of the γ-radiation of ^{140}La used as a calibrating radiation in certain studies of the short-lived gross γ-activity. ^{140}Ba activity obtained from n-induced ^{238}U fission is also commonly used as a reference activity in the studies of fission yields and radiation composition. ^{140}La is usually separated from the fission product mixture as lanthanum fluoride, which was again dissolved and carried with cerium carrier, cerium being later separated as $Ce(IO_3)_4$. Sometimes, in the presence of hold-back barium carrier, lanthanum activity could be carried with lanthanum carrier and then purified as hydroxide.

Among other methods for the separation of ^{140}La from ^{140}Ba, the solvent extraction process by HDEHP in toluene (*1*), adsorption chromatography on MnO_2 (*2*), and thin-layer chromatography on silica gel (*3*, *4*)

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may be mentioned. Use of such synthetic organic resins as cation (*5*) (Dowex-50) and anion (*6*, *7*) (Dowex-1) exchangers has been described where the adsorbed daughter in the Dowex-50 column is eluted with citric acid at pH 3.8 and then the parent is recovered by the same reagent at pH 6.07. In the case of a Dowex-1 column, barium comes out and lanthanum is adsorbed as the hydroxide which is later eluted with nitric acid. Separation of 99% pure lanthanum on an anion exchanger charged with carbonate has also been claimed (*7*). It is well known that the use of organic resins in the separation of radio-isotopes is not always recommended due to their lesser power of withstanding higher radiation doses and higher temperatures than those of inorganic ion exchangers.

Among the inorganic ion exchangers, the use of stannic ferrocyanide for the separation of Ba–La mixtures has been described (*8*), but details of the extent of separation are not mentioned. A considerable number of separations have been carried out (*9–13*) by using alumina as the ion exchanger. Alumina was also observed (*14*) to act as an improved ^{99m}Tc generator when a ^{99}Mo–^{99m}Tc mixture was suitably fed over it. Separation of carrier-free RaE from RaD over the alumina column has also been reported (*15*).

In the present investigation a very simple separation procedure which uses the alumina column for preparing carrier-free ^{140}La from its parent ^{140}Ba has been developed. Alumina was chosen due to its cheaper price, exceptional adsorption character (*16*, *17*), thermal resistance, etc. Differences in valency, solubility, and adsorbabilities of some of the well-known barium and lanthanum compounds at different pH values have been considered in order to achieve a clear separation.

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EXPERIMENTAL

^{140}Ba activity in equilibrium with ^{140}La was supplied in chloride from BARC, Trombay, India. Chromatographic alumina was of G.R.E. Marck quality. All other reagents used in the investigation were of AnalaR grade.

A glass column was prepared with an internal diameter of 1 cm and was equipped with a glass-wool plug, both at the top and at the bottom, with $\sim$18 g Al_2O_3 packed in it. The oxide column had a height of 30 cm. The column was surrounded by an outer jacket through which steam was circulated when necessary. This arrangement made the column hot during the adsorption and elution process.

Two methods [sulfate (A) and chromate (B)] of precipitations have been used for the separation of ^{140}La from ^{140}Ba. In the sulfate method, 10 ml

of activity solution was treated with 20 ml of methanol and then diluted to 100 ml. To the cold solution, 2 ml of dimethylsulfate was added and the mixture was immediately placed on steam bath. It was allowed to digest for about 1.5 hr. During the aging process the volume of solution was maintained constant by the occasional addition of methanol. The solution was next fed into the column, and the effluent was collected in a conical receiver fitted at the bottom and connected to a water pump which regulated the flow of solution at a rate of 14 to 15 drops/min. The column was then washed with a little more 20% methanol to wash out any residual daughter activity. No barium tracer was observed in the collected solution whereas almost all the lanthanum had been eluted out. The barium activity was thus found to be adsorbed on the oxide column.

In the chromate method, the activity solution was mixed with 0.5 g of ammonium acetate and 1.0 g of urea, the solution adjusted to pH 1.7 to 1.8, and the volume kept at 150 ml. About 5 ml of 10% $K_2Cr_2O_7$ solution was then added and the solution was heated to 90°C for 2.5 hr. The pH was then raised to 5.6 and the hot solution was fed into the column kept hot by circulating steam. It was then washed with 40 ml of 1% $K_2Cr_2O_7$ (pH 5.6) and the washings collected in the usual way. About 80 to 85% lanthanum activity was found to be eluted. The residual lanthanum was gradually washed out by washing 2 or 3 times with the same solvent. Final recovery was 100%. The collections were mixed together, concentrated to a convenient volume, and a 10-ml portion was counted with a G.M. liquid counter. The results are shown in Fig. 1.

The barium activity found to be adsorbed in the alumina column in both the cases described above was later washed out by hot 1:1 HNO_3. The experiment was repeated and the results confirmed. The overall separation procedure took about 3 hr in both cases.

DISCUSSION

The decay curves of ^{140}La (Fig. 1) show that the product is of high radiochemical purity. It has been already mentioned that most of the lanthanum activity was eluted out after pouring the chemically processed Ba–La mixture solution into the column. The residual lanthanum activity was also taken out by washing 2 or 3 times with the same solvent. Lanthanum did not react with either the sulfate or the chromate.

In the sulfate method, dimethylsulfate was chosen for its reported ability in the microdetermination of barium to form a relatively coarse, readily filterable, and washable precipitate with a minimum amount of

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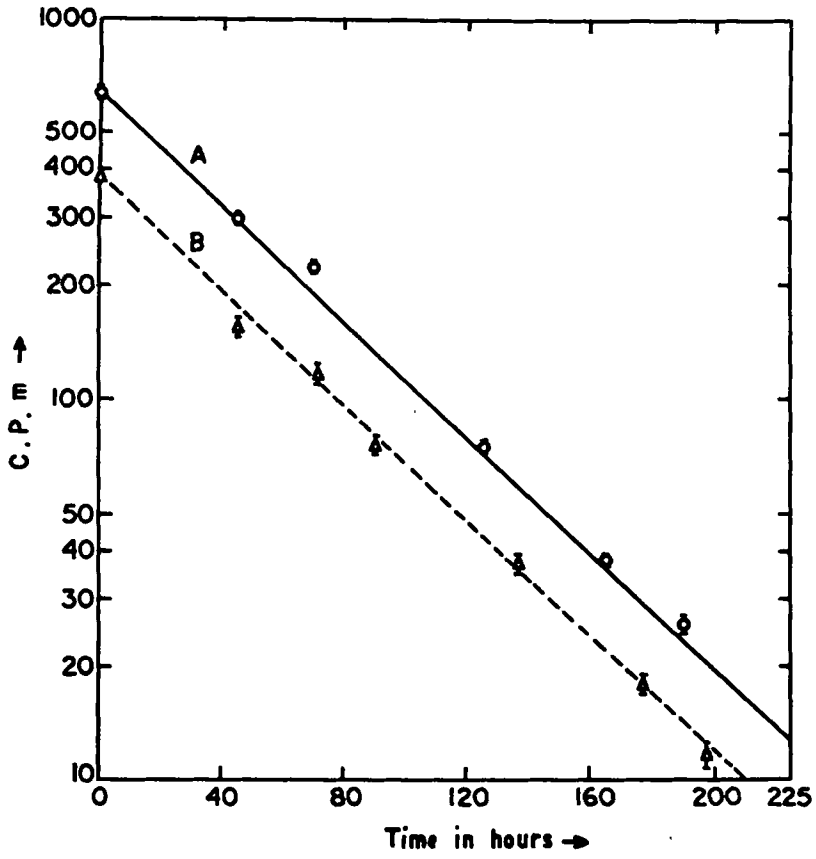

FIG. 1. Decay of ^{140}La separated from ^{140}Ba. Barium was separated as $BaSO_4$ (A) and as $BaCrO_4$ (B).

creeping quality together with high accuracy and precision over the lower concentration ranges. The method has been also reported (*18*) to be free from the difficulties of interference effects of foreign cations and anions, thus saving the analyst's time. Precipitation by 20% methanol has proved efficienct in the case of small quantities of barium for the attainment of quantitative results and the production of a readily filterable precipitate.

Barium chromate was claimed to be slightly soluble in a solution of pH ~2. The solution pH was therefore slowly raised by urea, a practice sometimes adopted in microdeterminations to form large, readily filterable,

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and easily washable crystals (*19*). The final pH was brought to 5.7 to effect quantitative separation. The use of a lower concentration of precipitant was, however, found to effect the separation only incompletely. The use of 10% $K_2Cr_2O_7$ solution proved to be best.

Certain authors (*20*) hold the view that the most hydrous oxides probably adsorb polyvalent anions more than the monovalent ones, and this, besides the fractional precipitation, is responsible for this type of separation. This may be due to the fact that when solid hydrous oxides are moistened by water, surface hydroxyl groups exchange readily with polyvalent anions, such as sulfate or chromate, which are used in the present investigation. It should be noted that a fairly long time was used for aging the processed solution before it was poured into the column in both cases. The effect was only partially successful if the aging process was approximately 1 hr. The kinetics of these reactions were not studied, and they might offer some explanation of the reaction behavior. The removal of barium activity from the alumina surface did not creat any problem in either the sulfate or chromate method.

From the facts stated above, it appears that the preferential adsorption character of alumina could be utilized to investigate many other similar separation chemical problems.

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