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CARRIER-FREE SEPARATION OF THE INDIVIDUAL RARE EARTH RADIONUCLIDES FROM FISSION PRODUCT MIXTURES

by
L. Wish
S.C. Foti

U.S. NAVAL RADIOLOGICAL DEFENSE LABORATORY

SAN FRANCISCO · CALIFORNIA · 94135

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ADMINISTRATIVE INFORMATION

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Eugene P. Cooper
Eugene P. Cooper
Scientific Director

D. C. Campbell, CAPT USN
Commanding Officer and Director

ABSTRACT

A procedure based on solvent extraction with diethylhexylphosphoric acid was tested for the quantitative carrier-free separation of yttrium and the rare earth group elements from other fission products. This procedure is simpler and more rapid than the previously used cation exchange resin separations of rare earths from alkali metals and alkaline earths. Subsequent quantitative carrier-free isolation of individual rare earth fission products was accomplished by their elution from cation exchange resin with alpha-hydroxyisobutyric acid at room temperature.

SUMMARY

The Problem

In the analysis of fission product mixtures, the rare earth elements and yttrium usually are first separated together by gravimetric radiochemistry after the addition of suitable carriers. The rare earths are then separated from each other by adsorption on a hot cation exchange resin and elution with a complexing agent. It would be much more advantageous to eliminate the need for carriers by using techniques which give quantitative yields. Also, ion exchange at room temperature requires simpler apparatus and less care.

Findings

A method was developed for the quantitative separation of the individual rare earths and yttrium from fission product mixtures without the use of carriers. The rare earth activities first are purified as a group by solvent extraction and then recovered individually by ion exchange at room temperature.

INTRODUCTION

The rare earth radionuclides separated from a fission product mixture are important indicators for determining the type of fission nucleus and the number of fissions in a given sample of the mixture. The rare earths and yttrium usually are isolated as a group using standard radiochemical gravimetric procedures after the addition of suitable carriers. This group is then loaded on a hot cation exchange resin column, and the individual rare earths are recovered by elution with a suitable complexing agent. It would be advantageous if quantitative carrier-free separations could be used at room temperature. Such separations would not require time-consuming yield determinations and would give sharper ion exchange elutions. Also, room temperature columns would be much easier to work with and would require simpler apparatus that is much more adaptable to routine analysis.

Wish[10-12] developed a method for the carrier-free quantitative separation of the rare earth group from fission product mixtures using ion exchange almost exclusively. The procedure is relatively slow and the rare earth group is finally eluted from a cation column with alpha-hydroxyisobutyric acid (AHIB). This would require further treatment for removal of the complexing agent before the separation of the individual elements. Peppard, et al.,[7] showed that 1.5 M diethylhexylphosphoric acid (HDEHP) is an excellent extractant for yttrium from very dilute hydrochloric acid solution. Strontium and barium are not extracted under these conditions. McCown and Larsen[5] found that the rare earth group can be recovered quantitatively from a gross fission product mixture by extraction with HDEHP in toluene. They used 0.1 N hydrochloric acid for the aqueous phase and stripped the activities from the organic phase with 8 N acid. The decontamination from the other fission products was 99 % or greater. However, they did not actually find out whether the decontamination is good enough for a subsequent satisfactory separation of the individual rare earths. Since this extraction is much faster and simpler than previous methods, the extent of the decontamination was investigated here to determine whether it could be used prior to the recovery of the component rare earth radionuclides.

This recovery of the individual rare earths has been investigated. In 1956, Choppin, Harvey, and Thompson[3] reported the use of AHIB as a new eluant for the ion exchange separation of the actinide elements. In the same year Choppin and Silva[4] found that the separation factors in the elution of the rare earth tracers are greater for this reagent than for the commonly used lactic acid. Both of these investigations were carried out at 87°C. Smith and Hoffman[9] carried out carrier-free actinide and lanthanide separations from Dowex-50X4 with AHIB at room temperature. They reported results similar to those obtained with hot columns. More recently Seyb and Herrmann[8] separated the rare earths from fission product mixtures in 9 hr with a hot Dowex-50X8 cation column and 1 M AHIB. They added a definite volume of eluant at a prescribed pH for the removal of each element. They used 0.1 mg of carrier for each and rigidly controlled the conditions to obtain reproducibility of elution. Wolfsberg[14] separated carrier amounts of individual rare earths from Dowex 50X4 in 30 hr at room temperature with 0.5 M AHIB. He used the gradient elution technique[1,6] to give a gradual increase in pH to the eluant.

Through utilization of AHIB as an eluant together with gradient elution, a method was developed for the quantitative separation of individual rare earths at room temperature. This method, together with the HDEHP extraction, was applied to the recovery of these rare earths from a fission product mixture.

EXPERIMENTAL

Apparatus

The resin columns were made from precision 3-mm-bore capillary glass tubing with a sintered ground-glass plug in the bottom. Exactly 1.0 g of air-dried resin gave a column height of 17.5 ± 0.2 in. after settling. The resin was resettled after each run and reused.

The gradient elution arrangement was similar to that used by Bunney, et al.[2] Two 400-ml beakers served as eluant reservoirs. The Tygon tubing leading from the low pH reservoir to the resin column was 10 ft long and of 1/8-in. internal diameter, and contained a volume of 23 ml. Since both beakers had the same dimensions, the volume removed from the low pH reservoir was replaced with half as much volume from the higher pH reservoir, thus giving a gradual increase in the pH of the eluant. The fractions were collected in 10-ml glass tubes placed in an automatic sample changer. These were transferred directly to a gamma-ray well scintillation instrument for counting.

Reagents

The AHIB was obtained from the Aldrich Chemical Co. of Milwaukee, Wisconsin. Two batches of reagent were used. These showed little difference in their elution properties. The solutions when initially made up were slightly turbid and were filtered before the pH was adjusted with ammonium hydroxide solution. The HDEHP was obtained from the Virginia Carolina Chemical Corp. The Dowex 2 and Dowex 50W resins were 200-400 mesh from the BioRad Laboratories of Berkeley, Calif.

Radionuclides

Yttrium-88, europium-155, praesodymium-143, and cerium-144 as carrier-free tracers were obtained commercially or separated from fission product mixtures. Gadolinium-159, promethium-145, samarium-145, neodymium-147, and terbium-160 were of high specific activity and were made by neutron irradiation of suitable stable isotopes.

PROCEDURE

Isolation of the Rare Earth Group

It was desirable to have carrier-free activities in as strong an acid solution as possible in order to minimize adsorption on the walls of the container. Therefore they were extracted by HDEHP from 0.05 N instead of 0.01 N hydrochloric acid solution as used by McCown and Larsen.[5] A further increase in acid strength will give lower extraction yields.[7] Also, it was found that when cerium-144 tracer was extracted with 1.5 M HDEHP from 0.05 N acid solution, 2-5 % of the activity remained in the aqueous phase even after prolonged shaking. However, the cerium was quantitatively extracted with 2.0 M HDEHP. This higher reagent concentration was used for all subsequent extractions.

To test the combined solvent extraction and ion exchange procedure the rare earth group was extracted with HDEHP from a 1-day old fission product sample. After mixing for 5 min, the phases were separated and the HDEHP in toluene was back washed with 2 portions of 0.05 N hydrochloric acid. The rare earths were stripped from the organic phase by mixing with 3 portions of 10 N hydrochloric acid. The combined acid solutions were evaporated to a small volume, adjusted to 0.1 N and loaded on a Dowex 50W cation column. The column was washed with 0.1 N hydrochloric acid and the rare earths were then eluted with AHIB. All the effluents were measured in a γ-ray scintillation well-counter. The loading, wash, and pre-yttrium solutions contained significant amounts of γ-ray activity consisting mainly of the molybdenum-technetium-99 pair, with lesser amounts of ruthenium-103, 106, rhodium-105, the tellurium-iodine-132 pair, and zirconium-95, 97. The rare earth fractions were contaminated with smaller amounts of these radionuclides. The total gamma-ray contamination was estimated as 1-3 % of the rare earth gamma-ray activity. Two extraction cycles on another aliquot reduced the contamination to 0.05 %.

Since molybdenum-99 was the main contaminant it was found that prior to the rare earth separation a molybdenum extraction with 0.1 % alpha-benzoinoxime in chloroform[13] resulted in only 0.01 % or less gamma ray contamination of the rare earth fraction. A similar purification was obtained by passing the 10 N hydrochloric acid stripping-solution through a Dowex-2 anion column. This is desirable since at the same time the resin removes the small amount of HDEHP that is soluble in the stripping solution. This HDEHP gave an insoluble residue when the hydrochloric acid solution was evaporated to dryness and taken up in AHIB for loading on a cation column. Peppard has found recently that this undesirable HDEHP residue also can be removed easily by washing the stripping solution with n-octyl alcohol.

A satisfactory decontamination procedure is summarized as follows:

1. Extract the molybdenum with 0.1 % alpha-benzoinoxime in chloroform from 1 N hydrochloric acid solution.

2. Extract the rare earth group with 2.0 M HDEHP in toluene from 0.05 N hydrochloric acid solution.

3. Strip the rare earth group from HDEHP with 10 N hydrochloric acid solution.

4. Pass the 10 N hydrochloric acid solution through an 0.3 x 7.0 cm Dowex 2 anion resin column at a flow rate of 1 drop/12-15 sec. Wash column with 12 ml of 10 N hydrochloric acid, and evaporate load and wash solution to dryness.

5. Repeat steps 2, 3, and 4.

6. Take up the residue with 1 M AHIB.

Ion Exchange Separation of Individual Rare Earths

Prior to use the Dowex 50WX4 column was equilibrated with 1 M AHIB eluant at pH 2.79. During the equilibration the flow rate was adjusted to 1 ml per 10 min by raising or lowering the reservoirs. The volume of collected fractions of eluate (of known density) was determined by weighing.

A mixture of rare earths and yttrium tracers in hydrochloric acid solution was evaporated to dryness and the residue taken up in 1 M AHIB. Loading the column with the same concentration of eluting agent prevents any change in the resin volume, thus maintaining the equilibrium conditions. This solution was transferred quantitatively to the top of the resin column with the aid of a polyethylenc policeman and three 0.5-ml washes (1 M AHIB pH 1.9). The loading solution was forced into the resin column in 5 min by air pressure. The space in the column above the resin was then filled with the pH 2.79 eluant, and the tube from the low pH reservoir was attached to the column. The reservoirs contained 206 ml of the pH 2.79 and 200 ml of the pH 3.40 eluant. The excess of 6 ml in the first reservoir prevents premature flow from the high pH solution due to any difference in pressure. Ten-minute fractions were collected.

RESULTS

Synthetic Mixtures

Figure 1 shows the elution history of a mixture of rare earth radionuclides. The lightest element, cerium, was recovered in 35 hours. All the elements were separated from their neighbors, and no evidence of cross-contamination was obtained with gamma-ray spectrometry. The times between the elution of gadolinium and euoropium and between that of neodymium and praesodymium were the smallest.

In order to determine the reproducibility of elution, similar mixtures were separated on each of three columns with essentially the same flow rate. The procedure was then repeated. The results are given in Table 1, which lists the 10-min fraction range in which each element was eluted completely. At the bottom of the table is the elution range which best covemall six runs. The yttrium started to elute on the tenth 10-min fraction and was completely eluted by the 20th fraction, the terbium was found in the next 20 fractions, etc. The range was exceeded by 2 and 3 fractions in a few cases. However, if the activities within the given fractions were to be collected, any cross-contamination would be less than 1 %.

Additional runs were made on the same 3 columns, but this time 100-min fractions were collected for each element. These were combined in accordance with results in Table 1. No cross-contamination was detected with a gamma-ray spectrometer. Table 2 gives the recovery of known quantities of the various rare earth elements. No gadolinium

TABLE 1

Elution Range for Complete Recovery of Rare Earths From Dowex 50WX4 at Room Temperature
(10-min fractions)

Col.	Flow Rate	Fraction Numbers								
		Y	Tb	Gd	Eu	Sm	Pm	Nd	Pr	Ce
1	1.02 ml/ 10-min fraction	11-17 11-17	21-28 22-23	48-57 -	63-77 63-76	87-101 86-101	115-135 115-133	143-157 141-157	160-178 160-178	186-207 185-207
2	1.03 ml/ 10-min fraction	10-17 11-18	21-28 21-31	49-58 -	63-77 63-77	87-103 87-101	118-137 116-134	144-160 142-157	162-182 160-178	190-210 186-207
3	1.03 ml/ 10-min fraction	10-17 11-17	20-30 22-32	48-59 -	63-78 63-77	87-103 86-100	117-137 114-134	143-159 139-154	161-180 157-175	187-208 182-204
	Elution Range	10-20	21-40	41-60	61-80	81-110	111-140	141-160	161-180	181-210

TABLE 2

Percent Recovery of Rare Earth Radionuclides

Column	Y	Tb	Gd	Eu	Sm	Pm	Nd	Pr	Ce
1	99.2	100.4	-	99.9	99.7	101.5	103.1	97.3	99.4
	99.2	101.9	-	99.0	99.9	97.4	102.5	98.9	99.7
2	100.1	100.1	-	100.3	100.1	99.6	98.6	100.5	98.8
	99.6	99.6	-	99.9	101.0	100.2	101.1	98.9	99.3
3	99.9	99.9	-	100.2	100.2	99.2	102.6	99.1	-
	100.9	100.9	-	100.2	99.5	99.8	99.6	99.2	99.5
Mean ± Standard Dev.	99.8±0.6	100.8±0.8		99.9±0.5	100.1±0.5	99.6±1.3	101.3±1.7	99.0±1.0	99.3±0.3

tracer was available at this time. The average recovery for each radionuclide was equal to or greater than 99 % with a standard deviation of 1-2 %.

The data in Tables 1 and 2 show that the carrier-free elution of the rare earths was sufficiently reproducible so as to allow collection of these elements in a predetermined definite time interval. Each column must be calibrated and the flow rate kept within narrow limits, e.g. 1.0 ± 0.05 ml per 10 min.

Fission Product Mixtures

The rare earths were separated from a fission product mixture obtained from a thermal neutron irradiation of uranium-235. The separations were made on four aliquots, two at 15 and two at 22 days after irradiation. Since at these times the very low yield of terbium-161 activity is negligible, terbium 160 tracer was added to each aliquot as a check on the yield of the procedure. Each aliquot contained approximately 10^{13} fissions. However, for very young fission-product samples the terbium fraction will be contaminated with significant amounts of yttrium due to the great difference in their yields. This will necessitate recycling the terbium fraction through a second ion exchange column.

Table 3 gives the results of the four column runs in gamma-ray counts per minute for each element. Fifty-minute fractions were taken and combined as indicated by Table 1 for activity measurements. For comparison the 15-day fractions were counted at the same time as the 22-day. The terbium-160 was recovered quantitatively and it was assumed that the other rare earths behaved similarly. There was good agreement between the duplicate runs, with a maximum deviation of 3 %, except for promethium in the 15-day sample. The overall results showed standard deviations of 1-2 %, except for promethium for which it was 4 %. The lanthanum-140 daughter activity of barium-140 is carried along with the rare earths and was eluted after the cerium. The duplicate lanthanum results showed excellent agreement. Pulse height analysis on a gamma-ray spectrometer showed no cross-contamination of any of the rare earth elements.

RAPID SEPARATION

To further speed up the separation procedure, a mixture of rare earth radionuclides was adsorbed on a Dowex 50X4 column and eluted as before with 1 M alpha-AHIB acid solution with a gradient pH of 2.75-3.30.

TABLE 3

Comparison of Rare Earth Activities from 15- and 22-Day-Old Aliquots from a Fission Product Mixture

Run		Gamma-Ray Activities (cpm) at Common Counting Time								
		Y	Tb	Eu	Sm	Pm	Nd	Pr	Ce	La
15 day	1	$1.71\text{x}10^{5}$	$1.74\text{x}10^{4}$	$1.35\text{x}10^{4}$	$8.00\text{x}10^{2}$	$7.92\text{x}10^{2}$	$2.62\text{x}10^{6}$	$9.03\text{x}10^{4}$	$6.40\text{x}10^{6}$	$3.37\text{x}10^{6}$[a]
	2	$1.71\text{x}10^{5}$	$1.69\text{x}10^{4}$	$1.36\text{x}10^{4}$	$8.14\text{x}10^{2}$	$8.66\text{x}10^{2}$	$2.60\text{x}10^{6}$	$8.95\text{x}10^{4}$	$6.32\text{x}10^{6}$	$3.37\text{x}10^{6}$[a]
22 day	1	$1.71\text{x}10^{5}$	$1.72\text{x}10^{4}$	$1.32\text{x}10^{4}$	$8.10\text{x}10^{2}$	$8.30\text{x}10^{2}$	$2.59\text{x}10^{6}$	$9.09\text{x}10^{4}$	$6.34\text{x}10^{6}$	$1.75\text{x}10^{5}$
	2	$1.76\text{x}10^{5}$	$1.73\text{x}10^{4}$	$1.33\text{x}10^{4}$	$7.99\text{x}10^{2}$	$8.42\text{x}10^{2}$	$2.60\text{x}10^{6}$	$9.34\text{x}10^{4}$	$6.31\text{x}10^{6}$	$1.75\text{x}10^{5}$
Mean ± Standard Dev.		$1.72\text{x}10^{5}$ ± 0.03	$1.72\text{x}10^{4}$ ± 0.02	$1.34\text{x}10^{4}$ ± 0.02	$8.06\text{x}10^{2}$ ± 0.07	$8.33\text{x}10^{2}$ ± 0.31	$2.60\text{x}10^{6}$ ± 0.01	$9.10\text{x}10^{4}$ ± 0.17	$6.34\text{x}10^{6}$ ± 0.04	
Control			$1.72\text{x}10^{4}$							

a. At time of Ba-La separation.

The flow rate was increased sharply from 1 ml/10 min to 1 ml/min using a displacement pump inserted between the reservoir and the column. The elution curve is shown in Fig. 2. The last of the cerium was recovered in 5-1/2 hr. The resolution of the heavier elements through europium was good although no data was available for gadolinium. The interval between terbium and europium is not as large as before and gadolinium might cause some difficulty. Beginning with promethium the separations are not sharp, and there is significant cross-contamination. Further investigation is being carried on to improve the resolution of the lighter rare earths.

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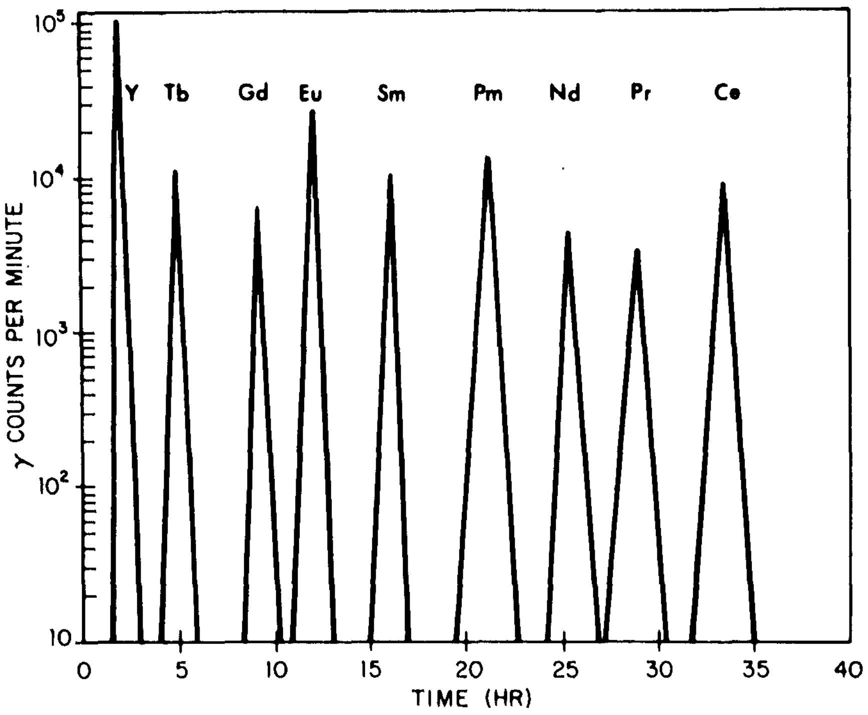

Fig. 1 Elution of Rare Earths and Yttrium from a Dowex-50WX4 Cation at Room Temperature

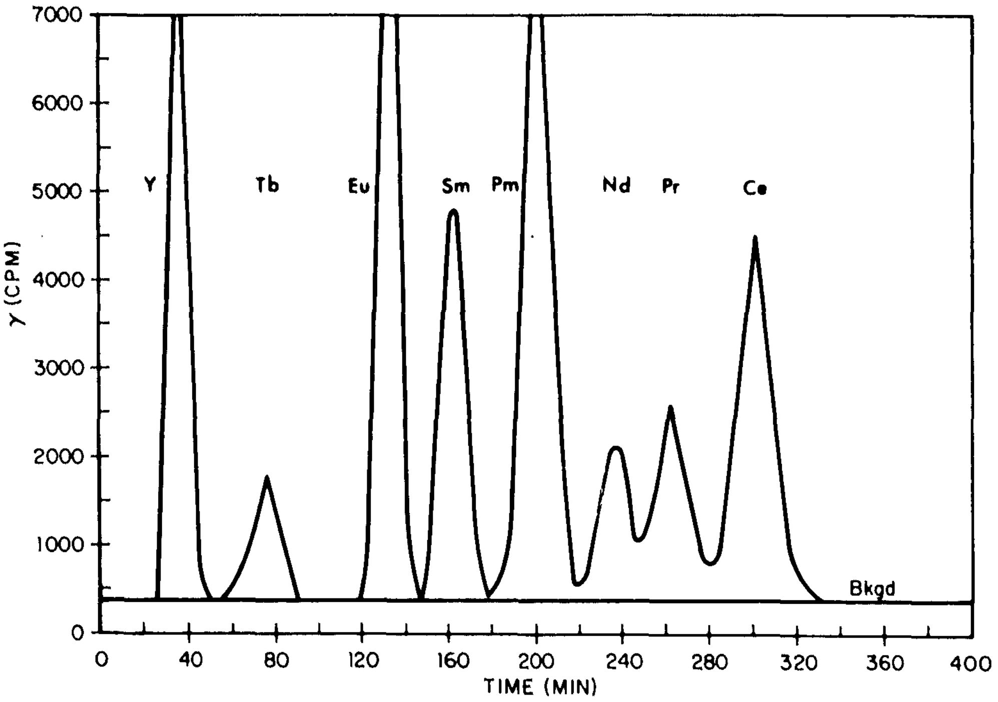

Fig. 2 Rapid Elution of Rare Earths at Room Temperature. Flow rate = 1 ml/min.

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