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Chemistry

AEC Research and Development Report

THE PREPARATION OF AN AMERICIUM GAMMA SOURCE

by

R.C. Milham
Analytical Chemistry Division

August 1956

E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory

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ABSTRACT

Americium-241 was separated from lanthanum, plutonium, and common metallic elements by ion exchange methods. The purified americium was embedded in plastic and mounted in a holder for use as a gamma ray source.

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THE PREPARATION OF AN AMERICIUM GAMMA SOURCE

INTRODUCTION

Americium-241 (62 kev gamma) was required as a gamma ray source. Fifty milligrams of americium was available as a solution in 930 milliliters of 3M HCl. The solution also contained gram quantities of plutonium, iron, bismuth, and manganese. The objective of this work was to separate americium from these impurities and to precipitate it in a form suitable for mounting in a source holder. The americium compound was to be contained in such a manner that widespread contamination would not result in the event that the thin aluminum window of the holder was ruptured.

It was desirable to reduce the amounts of the other elements in the final precipitate in order to minimize the self-absorption of the gamma source. Ion exchange procedures have been developed for the separation of americium from various elements(1)(2) and these procedures were adapted for the separation reported here.

SUMMARY

Americium-241 was separated from gram quantities of other elements and the purified product was mounted in a holder for use as a gamma ray source.

The americium was separated by a hydroxide precipitation followed by ion exchange on anion and cation exchange resins. Finally, americium and added lanthanum carrier were precipitated as the fluorides and were heated to destroy the hydrates.

To avoid the hazard of handling a radioactive powder, the fluoride precipitate was embedded in plastic and sealed in an aluminum holder by means of a low-melting alloy.

DISCUSSION

The starting material for this work was 930 milliliters of 3M HCl containing 50 milligrams of Am-241. Spectroscopic analysis showed that the following elements were also present in approximately the concentrations listed:

aluminum	0.05 g/l	lanthanum	0.5 g/l
bismuth	5.	magnesium	0.1
calcium	0.01	manganese	3.
chromium	0.3	nickel	0.03
iron	3.	plutonium	5.

The separation scheme used is shown diagrammatically in Figure 1. An excess of concentrated ammonium hydroxide was added to the HCl solution to precipitate the insoluble hydroxides of bismuth, iron, lanthanum, plutonium, and americium. Upon centrifugation, manganese remained in the supernate.

The precipitate was dissolved in 500 milliliters of concentrated HCl. This solution was then made 0.01M in HNO_3 to oxidize any Pu(III) to Pu(IV). The solution was passed through a "Dowex 2" ion exchange column (6 cm diameter, 30 cm high), prepared in the manner described by Hicks et al.,(1) except that the perchloric acid treatment was omitted. Bismuth, americium, and lanthanum passed through the column while the iron and plutonium were absorbed. The iron and plutonium were then eluted from the resin with 600 milliliters of 1M HCl and discarded.

To remove bismuth, the effluent from the first column was diluted to about 4M HCl and was passed through a second "Dowex 2" column. At a concentration of 0.1 to 6M HCl, bismuth is absorbed by "Dowex 2" resin. The americium and lanthanum again were not absorbed.

An excess of HF was added to the effluent to precipitate LaF_3 and AmF_3. The volume of this precipitate was too large to fit the source holder, so it was necessary to remove the excess lanthanum.

"Dowex 50" ion exchange resin was used to separate the americium and lanthanum as described in AECD-3517(2). To use this method, the americium and lanthanum must be in concentrated HCl solution. This solution was obtained by first dissolving the fluoride precipitate in 40 milliliters of saturated boric acid solution to which 40 milliliters of 8M HNO_3 was added. Saturated sodium hydroxide was added to precipitate americium and lanthanum hydroxides. The precipitate was centrifuged and dissolved in ten milliliters of concentrated HCl. This solution was allowed to absorb on the top of a "Dowex 50" column for two hours before the americium was removed by chromatographic elution with concentrated HCl.

The eluate flowed at a rate of about two milliliters per minute and was collected in twenty-six 70-milliliter fractions. An aliquot of each fraction was taken for alpha counting. Figure 2 is a plot of the relative activity of each fraction as a function of the fraction number.

Fractions 8 through 24 were combined and about ten milligrams of lanthanum carrier was added. An excess of HF was added to precipitate the lanthanum and americium fluoride hydrates. The precipitate was washed with dilute HF and HNO_3 and ignited at 300°C for four hours to destroy the hydrate. It was estimated that no more than 10 to 20 milligrams of impurities was present in the final precipitate.

The source holder was made in two parts (Figure 3), the primary container and the secondary container. The aluminum primary container was designed to hold the liquid plastic in which the powder was suspended while the plastic hardened. The interior was machined with keyed sides so that the hardened plastic could not fall out.

The aluminum secondary container was designed so the primary container could be sealed against the 0.015-inch window with a low-melting alloy (Wood's metal). The secondary container was also keyed to prevent the hardened alloy from falling out. The alloy expanded upon solidifying to form a tight seal.

The americium powder was poured into the primary container and was mixed with a drop of "Araldite" catalyst and ten drops of "Araldite." After the plastic had hardened for 24 hours, the primary container was heated to drive off excess moisture. The primary container was placed in the secondary container with the "Araldite" surface against the 0.015-inch-thick window. The secondary container was then filled with the molten alloy. After the holder was washed with "Alconox" solution, there was no detectable contamination.

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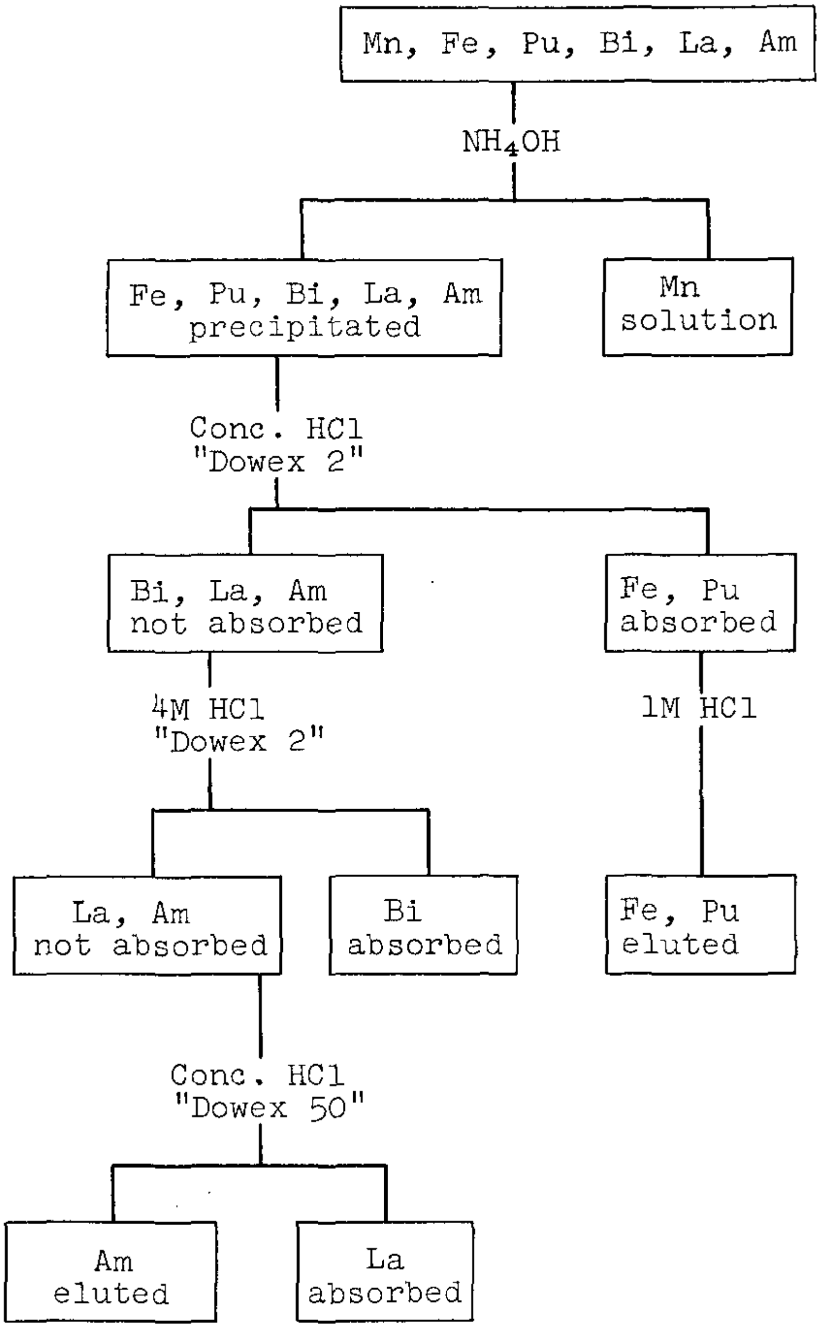
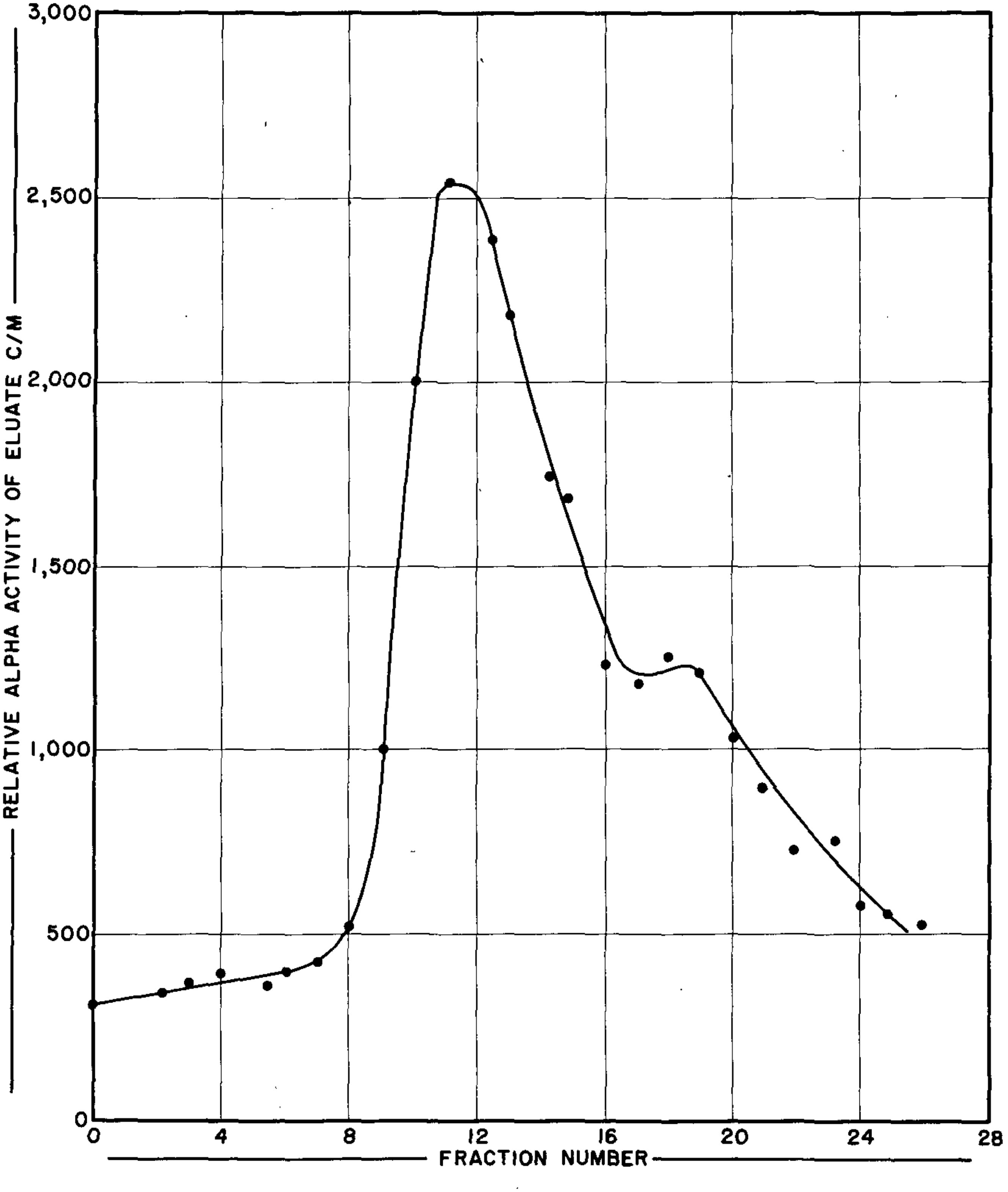
2. Diamond, R. M., Street, K., Jr., and Seaborg, G. T. An Ion Exchange Study of Possible Hybridized 5f Bonding in the Actinides. Radiation Laboratory, University of California, AECD-3517, August 28, 1951.

FIGURE 1



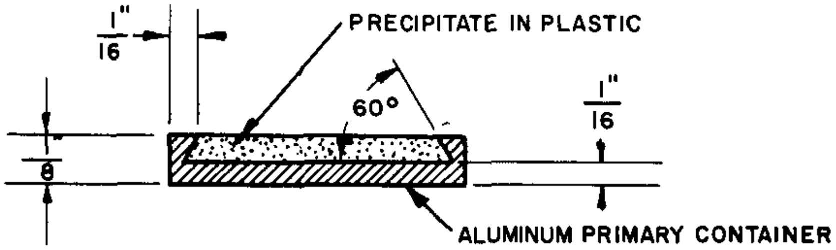
OUTLINE OF PURIFICATION PROCEDURE

FIGURE 2

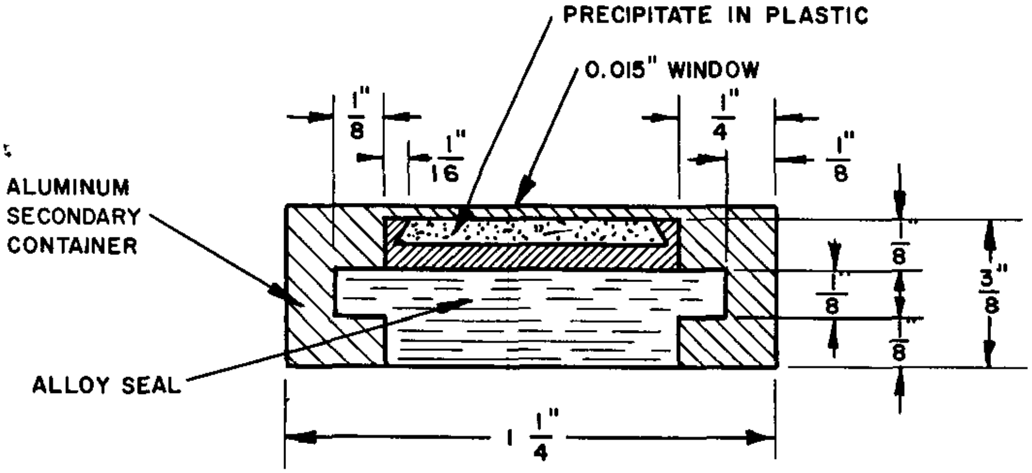


DISTRIBUTION OF AMERICIUM IN ELUATE FRACTIONS

FIGURE 3



PRIMARY CONTAINER WITH PRECIPITATE EMBEDDED IN PLASTIC



SECONDARY CONTAINER WITH PRIMARY CONTAINER INSIDE

SOURCE HOLDER