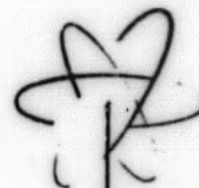


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AEC RESEARCH AND
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DECEMBER 1, 1963

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ACCURATE NUCLEAR FUEL BURNUP ANALYSES

EIGHTH QUARTERLY PROGRESS REPORT SEPTEMBER 1963 - NOVEMBER 1963

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VALLECITOS ATOMIC LABORATORY

GENERAL  ELECTRIC

ATOMIC POWER EQUIPMENT DEPARTMENT
SAN JOSE, CALIFORNIA

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FOREWORD

Accurate measurements of fuel burnup are required to aid in reactor design for high burnup fuels and to aid in evaluating nuclear fuel costs. The usefulness of current radiochemical methods is limited because of: (a) limited availability of long-lived radioactive isotopes among the fission products; (b) the accuracy with which these fission products can be measured because of uncertainties in their physical constants; and (c) the volatile nature of the few available isotopes. Mass spectrometric techniques are being investigated to obtain a more accurate burnup analysis by measuring the quantity of the nonradioactive refractory fission products produced from a variety of fuels. By using the accurate isotope dilution technique on stable fission products, errors that limit the final accuracy of radiochemical measurements are eliminated, such as errors in counting efficiencies, decay schemes, and decay constants, as well as those found in out-of-pile and in-pile decay corrections.

I. SUMMARY

The development of accurate methods for determination of nuclear fuel burnup began on the present program in December 1961. The report of the first year's progress, in cooperation with the Phillips Petroleum Company, appeared in TID-17385, "Burnup Determination of Nuclear Fuel", January 1963. The first seven quarterly reports appeared as GEAP-4053-1, GEAP-4053-2, GEAP-4082, GEAP-4137, GEAP-4201, GEAP-4278, and GEAP-4361. The work performed during the eighth quarter is summarized as follows:

A. CATION AND ANION EXCHANGE STUDIES

The determination of stable fission product neodymium in irradiated UO_2 in the presence of stainless steel or zircaloy, has been demonstrated successfully. The all-column procedure by which this was accomplished is described in GEAP-4278. A survey of unclassified fuels and cladding materials indicates that in addition to stainless steel and zircaloy, a common cladding for low-temperature fuels is aluminum. Cladding materials for high temperature fuels may include such high-melting metals as molybdenum and tungsten.

Aluminum was found to follow neodymium through the above chemical separation procedure and to add unwanted mass to the mass spectrometer filament. Separation of aluminum from neodymium has been achieved by elution from Dowex-50 with 20% ethanol-80% water saturated to 12M with HCl gas. Neodymium remains adsorbed under these conditions and is eluted later with 6M HCl.

To evaluate effects of molybdenum and tungsten on the neodymium separation, samples of each were activated by neutron irradiation. Each metal was then dissolved separately and followed radiochemically through the neodymium separation procedure. In each case, the metal was held up on Dowex-1 from 12M HCl, whereas the neodymium passed through. Decontamination factors (df) for each metal on this column were $\sim 10^2$. An additional df of 10^2 for each metal was obtained by adsorption of neodymium on Dowex-50 and washing the molybdenum and tungsten as well as aluminum and americium through with 20% ethanolic 12M HCl. The resulting over-all decontamination

from each, therefore, is in excess of 10^4 , which appears to be satisfactory.

B. HEAVY ELEMENT BEHAVIOR ON A D2EHP COLUMN

A further investigation of the interference of the heavy elements Pu, Am, and Cm in the neodymium procedure employing a Di-(2-ethylhexyl) phosphoric acid (D2EHP) column was carried out. A 100 cm long column was used to improve resolution, and excellent results were obtained. The width of the elution peaks at half height were 8.3 and 9.3 percent of the peak position for Ce-144 and Nd-147, respectively. Observed relative peak positions for Ac^{+3} , Ce^{+3} , Am^{+3} , Cm^{+3} , and Nd^{+3} were 0.04, 0.48, 0.72, 0.85, and 1.00, respectively. The Nd df from curium was 10^2 . The americium df was about 10^4 and the cerium df was over 10^5 . Although Pu^{+3} had been shown previously [3] to appear very near the Ce^{+3} peak, the chemical instability of this valence state apparently assures that plutonium will not be a significant interference, but will be tightly held as Pu^{+4} under the experimental conditions. One significant observation concerning the behavior of heavy elements on a D2EHP column has been made, i.e., the separation factors of heavy elements is at least as good as the best existing methods based on Dowex-50. It is apparent that heavy element separation factors even greater than those of the Dowex-50 — α -hydroxyisobutyric acid system can be obtained with the D2EHP system in many cases.

C. DISASSEMBLY AND ANALYSIS OF IRRADIATION ASSEMBLIES

Irradiation assembly GEV-1, which was irradiated in MTR in a thermal flux, has been disassembled in the hot cells and the individual capsules opened. One contained a U-235 foil, one a Pu-239 foil, and one a U-233 foil. Each foil was dissolved and the xenon collected for analysis. The resulting solutions were each analyzed for stable fission product neodymium nuclides and for the heavy element fuel isotopic content. Additional analyses for Cs, Ba, Ce, and Sm isotopes, which represent a major portion of the heavy mass fission yield peak, will permit calculation of the ratio of Nd-148 to all isotopes in the heavy mass peak. By normalizing the total of all isotopes to 100%, Nd-148 fission yields for U-233, Pu-239,

in addition to U-235 will be obtained.

Irradiation assembly GEV-2, which was irradiated in VBWR in a hardened thermal flux, has also been opened. This was a 40-inch long assembly containing foils of U-235, Pu-239, and U-233 similar to those in GEV-1, and specimens of stable fission product elements, both unshielded and shielded with CdO. The stainless steel outer tube has been gamma-scanned over its full length using the RCL 256-channel analyzer set to accept only the Co-60 photopeaks. An integrated, relative thermal flux profile was thus obtained. Also, cobalt and iron flux wires from within the individual capsules have been extracted and sectioned in 1/2-inch lengths to determine the absolute thermal flux and fast flux exposures. These data will be used for the determination of capture cross sections of the stable fission product nuclides selected for use as burnup indicators.

Assembly GEV-3 has completed its irradiation in VBWR and is cooling. This assembly contains Th-232 and U-238 oxides for the determination of fast fission yields of stable fission product nuclides.

II. PROGRAM PROGRESS

During the quarter, the ion exchange behavior of aluminum, tungsten, and molybdenum in the analysis of stable fission product neodymium has been studied; the behavior of heavy elements such as plutonium, americium, and curium in the neodymium separation on a D2EHP column has been further investigated; and the disassembly and analysis of the irradiation assemblies GEV-1 and GEV-2 has continued.

A. CATION AND ANION EXCHANGE STUDIES

1. Behavior of Non-Fission Product Species

A procedure for the determination of stable fission product neodymium as a burnup indicator in irradiated UO_2 fuel was developed and reported previously.^[1] It was found, however, that some aluminum appeared in the neodymium fraction when appreciable amounts of aluminum were present in the sample. Since some fuels contain aluminum, a method for elimination of its interference has been devised. In addition, several high temperature fuel constituents, including tungsten and molybdenum have been tested for interference in the neodymium procedure. Finally, the ion-exchange behavior of americium, which follows closely the chemistry of neodymium, has been further investigated in an attempt to optimize conditions for its removal. Results of these experiments are reported below.

a. Aluminum. A preliminary investigation showed that although aluminum was eluted slightly ahead of neodymium from a chromatographic column of di-(2-ethylhexyl) phosphoric acid supported on Chromosorb, it tailed excessively into the neodymium fraction.^[2] Figure 1 shows the elution characteristics of aluminum on such a column. Note the position of the neodymium band as indicated by an arrow. Since there were no suitable radioactive species of aluminum available as tracer, the elution of the aluminum was followed by a chemical method. The eluate was collected in small increments and the aluminum concentration in each increment was determined spectrophotometrically by the aurin tricarboxylic acid method. Solutions were buffered to pH 6.3 before color development. The range of the method as applied was from 1 to 250 μg of aluminum.

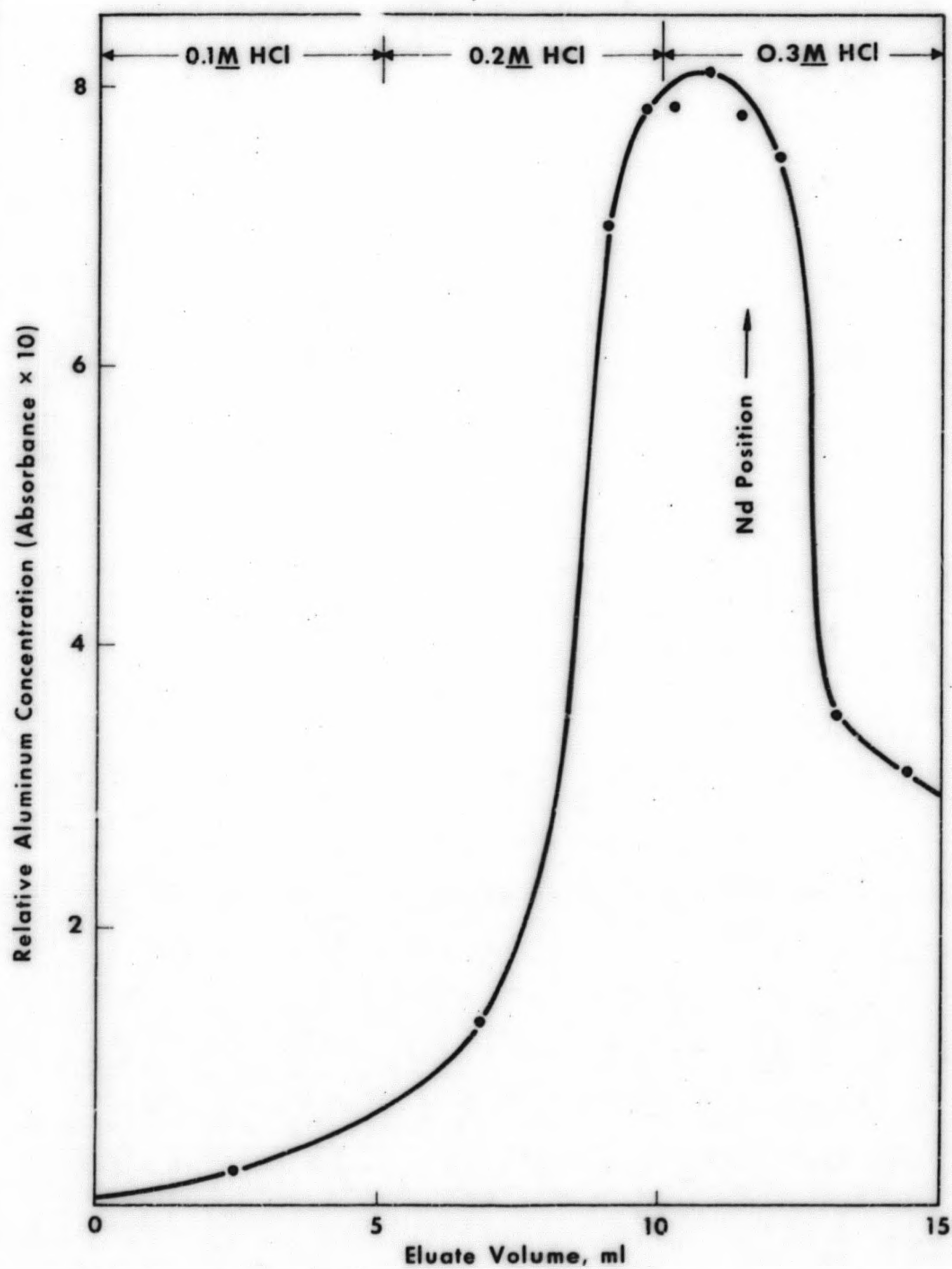


FIGURE 1. ELUTION OF Al FROM A COLUMN CONTAINING 0.1gm OF D2EHP ON CHROMOSORB-W

The most satisfactory method yet found for elimination of the aluminum interference has been elution from Dowex-50 with ethanolic 12M HCl. Under these conditions, neodymium is retained on the resin. Figure 2 shows the effectiveness of this separation. The column (4 mm x 2 cm) of Dowex-50 W x 8 was operated at room temperature. After preconditioning with 1M HCl, a suitable aliquot of aluminum was evaporated to dryness, redissolved in 125 μ l of 1M HCl, and loaded directly onto the column, which was then washed with two free column volumes (250 μ l) of 1M HCl at a flow rate of < 1 cm/min. The aqueous phase was displaced from the column with two free column volumes (250 μ l) of ethanol. Then the aluminum was eluted with 20% ethanol-80% water saturated with HCl gas to 12M. The neodymium was finally eluted with 6M HCl.

The effect of cross-linkage on the resin was also explored. Dowex-50 x 12 showed greater affinity for aluminum, giving greater tailing characteristics into the neodymium fraction. Dowex-50 x 4 released the aluminum quickly, but exhibited excessive swelling. Dowex-50 x 8 appeared to be the best all around choice of cross-linkage for this separation.

It is recommended strongly that this column separation be added between the initial Dowex-1 separation and the D2EHP column in the neodymium separation procedure.

b. Tungsten. The behavior of tungsten in the neodymium separation procedure was followed radiochemically with W-187. A Dowex-1 x 8 (200-400 mesh) anion exchange column (4 mm x 10 cm) was prepared for room temperature operation and preconditioned with 12M HCl. A suitable aliquot of W-187 was evaporated to dryness and redissolved in 0.5 ml of 12M HCl. The sample was loaded onto the column and washed with 3 free column volumes (1.5 ml) of 12M HCl at a flow rate of < 1 cm/min. The eluate was collected and counted in a NaI well-type scintillation counter. Over 99% of the W-187 activity was adsorbed on the column and remained there. Rare earths, including neodymium, were not adsorbed. The tungsten is, therefore, removed effectively from neodymium in this first step of the procedure.

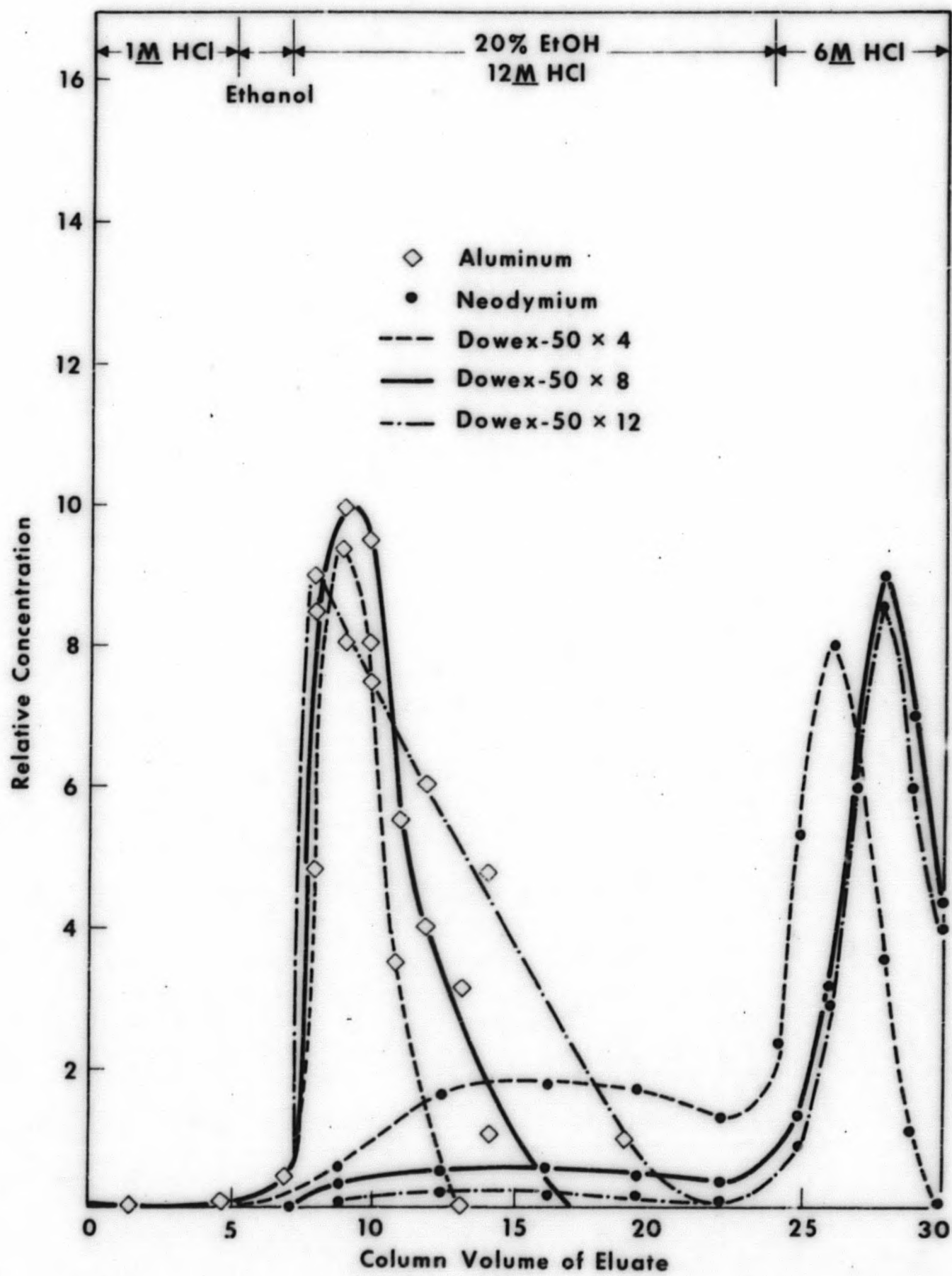


FIGURE 2. ELUTION OF Al AND Nd-147 FROM DOWEX-50 COLUMN

The behavior of tungsten on the Dowex-50 column was also observed. A Dowex-50 x 8 (200-400 mesh) cation exchange column (4 mm x 2 cm) was prepared for room temperature operation and preconditioned with 1M HCl. A suitable aliquot of W-187 was evaporated to dryness and redissolved in 125 μ l of 1M HCl. The sample was loaded onto the column and washed with 2 column volumes (250 μ l) of 1M HCl, then washed with 2 column volumes of ethanol to displace the aqueous phase. Then, 10 column volumes of 20% ethanolic 12M HCl were passed through the column, followed by 6 column volumes of 6M HCl to elute the neodymium fraction. Again, by use of the NaI well-type scintillation counter, over 99% of the W-187 activity was found to have been eluted in the ethanolic 12M HCl wash and less than 1% of the W-187 activity was found in the 6M HCl fractions which contain the neodymium.

These two experiments indicate that tungsten will not interfere with the neodymium separation, provided the 10-mg capacity of the resin is not exceeded. A df of 10^4 is achieved prior to the D2EHP column which appears to be entirely satisfactory.

c. Molybdenum. The behavior of molybdenum in the neodymium separation procedure was followed radiochemically with Mo-99. The Dowex-1 x 8 (200-400 mesh) anion exchange column (4 mm x 10 cm) was prepared for room temperature operation and preconditioned with 12M HCl. An aliquot of Mo-99 was evaporated to dryness and redissolved in 0.5 ml of 12M HCl. This sample was loaded onto the column and washed with 3 free column volumes (1.5 ml) of 12M HCl at a flow rate of < 1 cm/min and eluate (which contains the neodymium fraction) collected in a one dram glass vial.

The vial of eluate was counted in the well-type NaI scintillation counter. The Dowex-1 x 8 anion column itself was counted in a similar manner. Over 98% of the Mo-99 was found to be adsorbed on the column and < 2% to have passed through into the neodymium fraction. Thus, the bulk of the molybdenum is removed in the first step in the neodymium procedure.

The Dowex-50 column which was found useful in removing aluminum interference, as described above, was found also to remove 99% of the Mo-99 activity in the ethanolic 12M HCl wash portion and to pass less than 1% into the neodymium fraction. The overall df for molybdenum was found to be 10^4 through the first two columns, as long as the 10 mg molybdenum capacity of the resin is not exceeded.

d. Americium. Moderate levels of alpha radioactivity due to Am-241 have been found in neodymium fractions prepared for mass spectrometry. Since the americium content in fuel increases with exposure, at high burnups it can be expected to present a radiation hazard. The americium follows neodymium through the first Dowex-1 anion exchange column, but is separated by Dowex-50 cation exchange column. Some Am-241 activity was traced through the Dowex-50 x 8 column in a manner similar to that employed with the aluminum, tungsten, and molybdenum above. The Am-241 was eluted to the ethanolic-HCl wash and was decontaminated from the neodymium fraction by a factor of 10^4 .

The effect of ethanol concentration in the ethanolic 12M HCl was also evaluated. Differences between 10%, 20%, and 40% ethanol were slight. The effect of ethanol is primarily to decrease the polarity of the solvent and thereby reduce the degree of ionization and subsequent adsorption of the interfering metals. A 20% ethanolic content appears to be adequate.

On the basis of the above experiments upon Dowex-50 cation exchange resins, it is recommended that the 8% cross-linked resin washed with 20% ethanolic 12M HCl be added to the neodymium procedure between the initial 12M HCl elution from Dowex-1 and the subsequent D2EHP column. Such a modification assures an adequate decontamination factor of 10^4 for aluminum, tungsten, molybdenum, and americium.

B. HEAVY ELEMENT BEHAVIOR ON A DI-(2-ETHYLHEXYL)PHOSPHORIC ACID COLUMN

The study of heavy element interference has been continued on several elements which exist in the +3 valence state. The objective was to observe the elution behavior of these elements, in particular Am and Cm relative to Nd, in order to determine if under relatively good conditions an

adequate separation could be achieved. From these data, some comparison of this system could be made relative to the Dowex-50 system employing various complexing agents.

1. Column Preparation

A D2EHP column (4 mm x 100 cm) was prepared with 4.2 grams of 120-140 mesh Chromosorb-W which contained 0.7 gram of D2EHP. The column was jacketed for operation at 87°C, maintained with the vapor from boiling trichloroethylene. The column was preconditioned prior to the loading of the activities by passing through 6M HCl + 0.01M NH₂OH to destroy any oxidizing agents present in the chromatographic column which could react with the plutonium. The column was then washed with 0.19M HCl in preparation for loading.

The tracer activities consisted of ~ 10⁶ dpm of Nd-147, Ce-144, Ac-227, Am-241, and Cm-242. The Ac-227 was purified from its daughter activities just prior to the column study by passing it through a short (4 mm x 10 cm) D2EHP column. Plutonium was prepared in the +3 state by dissolving about 2 mg of plutonium metal in 0.5M HCl containing a dissolved crystal of NH₂OH and yielded the characteristic blue solution. The six activities were combined, adjusted to 0.1M HCl, and loaded onto the column.

2. Performance

After loading, the blue Pu⁺³ complex was visible on the column and occupied a band 3 cm long. The elution was then begun with 0.19M HCl at a flow rate of 0.80 cm/min. This corresponded to 0.084 ml/min. The blue Pu⁺³ band was observed to traverse the first 10 cm of column bed below which the opaque column insulation prevented further visual observation.

The first 50 ml were collected in 0.5-ml increments; the 51st through 80th milliliter in 1-ml increments, and 82nd through 120th milliliter in 2-ml increments. The vials were gamma-scanned in a well counter to determine their Ce-144, Nd-147, and Am-241 content. Aliquot volumes from nearly all of the vials were mounted and alpha pulse-height

analyzed to obtain the distribution of Ac-227, Pu-239, Am-241, and Cm-242. The results of this study are summarized in the chromatogram shown in Figure 3. The relative position of the peaks is the same as observed previously³ and the resolution of the system is slightly better than that obtained previously with the 70 cm column.⁴

The Pu-239 alpha activity, which was 100 times greater than that of the other activities, was not found in any of the fractions. There was no visual evidence of the location of the plutonium on the column after disassembly which indicates that the oxidation did not occur instantaneously but took place over a period of time distributing plutonium over an appreciable length of column. Apparently, oxidation to the Pu^{+4} state is greatly enhanced by the strong stabilization of the Pu^{+4} with D2EHP. These data show that the preliminary conclusion reached previously³ regarding the oxidation of macro amounts should be modified in that the extent of oxidation is a function of the length of time the plutonium is in contact with the D2EHP. These data further indicate the effectiveness with which plutonium interference is removed from the neodymium.

A comparison of separation factors relative to neodymium for the light rare earth D2EHP column⁴ and for the Dowex-50 system with different complexing agents is shown in Table 1. A similar comparison for the heavy elements is shown in Table 2, together with data on 2-ethylhexylphenyl phosphoric acid (2-EH(HP)A). In each table, the inverse of the separation factor is shown for the Dowex-50 case. These data, which are proportional to the distribution coefficient, are plotted in Figure 4 as a function of rare earth or heavy element atomic number. The calculated points in the figure were taken from Figure 2 of the Sixth Quarterly Report.⁴ It is seen that the points below neodymium (atomic number 60) deviate from a line drawn through the points above neodymium. This deviation has been attributed by Peppard to the effect

TABLE 1. SEPARATION FACTORS OF LIGHT RARE EARTHS IN DOWEX-50
AND D2EHP SYSTEMS RELATIVE TO NEODYMIUM

Element	Dowex-50			D2EHP	
	Glycolate ^[5]	Lactate ^[5]	α -HIBA ^[5]	Exper.	Calc.
La	0.18	0.23	0.19	0.18	--
Ce	0.48	0.42	0.39	0.48	--
Nd	1.00	1.00	1.00	1.00	--
Pm	1.25	1.33	1.61	2.10	--
Eu	1.97	2.24	4.68	12.0	--
Gd	2.10	2.74	6.60	--	31.0
Tb	2.70	4.88	13.5	--	73.0

TABLE 2. SEPARATION FACTORS OF HEAVY ELEMENTS IN DOWEX-50
AND D2EHP SYSTEMS RELATIVE TO CURIUM

Element	Dowex-50			D2EHP		2-EH(ϕ P)A
	Glycolate ⁵	Lactate ⁵	α -HIBA ⁵	Exper.	Peppard ⁶	Baybarz ⁷
Ac	--	--	--	0.05	--	--
Am	0.88	0.82	0.69	0.78	0.77	0.71
Cm	1.00	1.00	1.00	1.00	1.00	1.00
Bk	1.43	1.54	2.12	--	10.	30.
Cf	1.67	2.43	4.54	--	47.	99.
Es	--	3.00	7.05	--	--	130.
Fm	--	4.34	13.1	--	--	325.

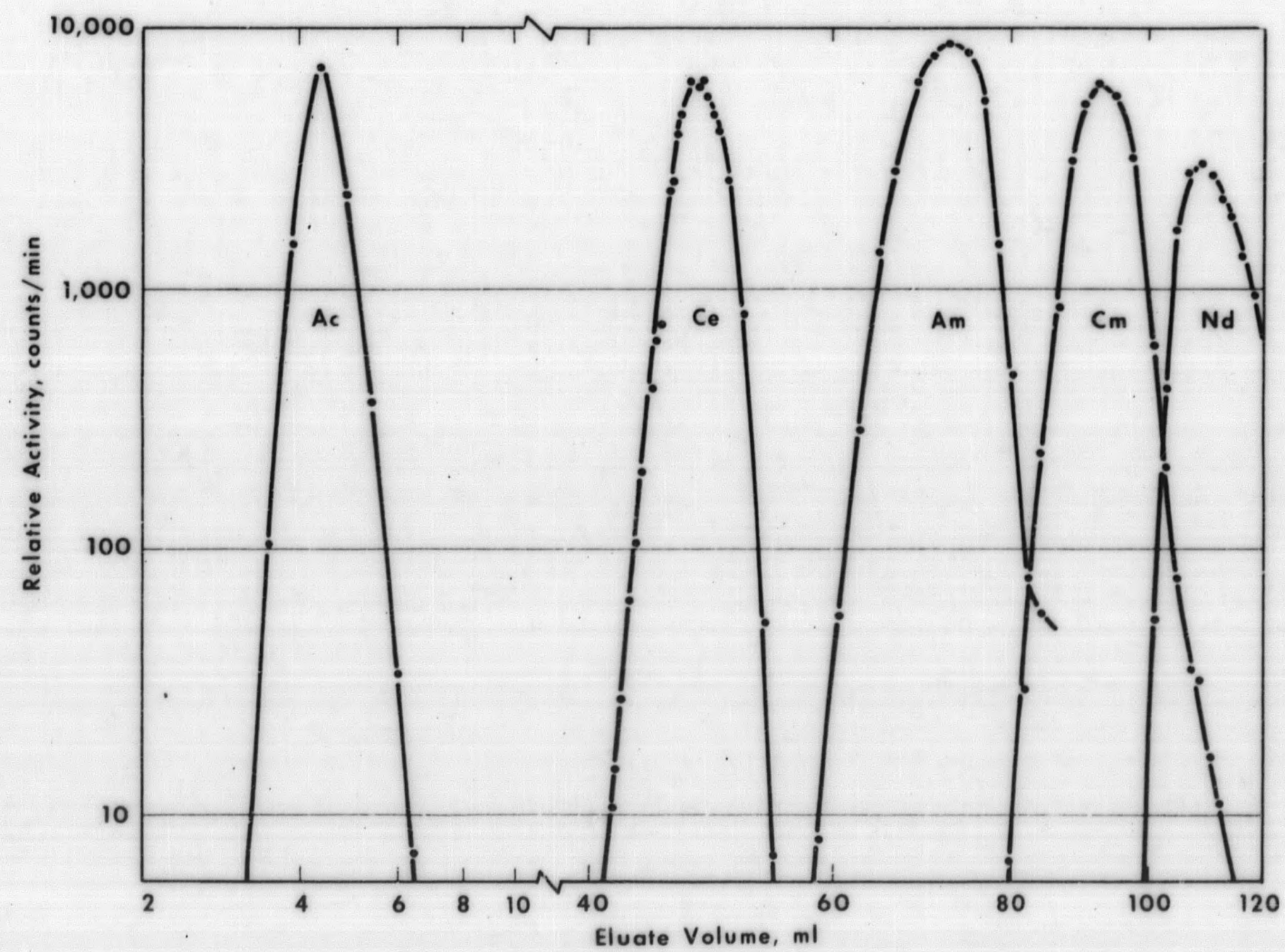


FIGURE 3. CHROMATOGRAM OF A MIXTURE OF Ac, Ce, Am, Cm, AND Nd
AT 87°C ON D2EHP COLUMN CONTAINING 0.7gm D2EHP

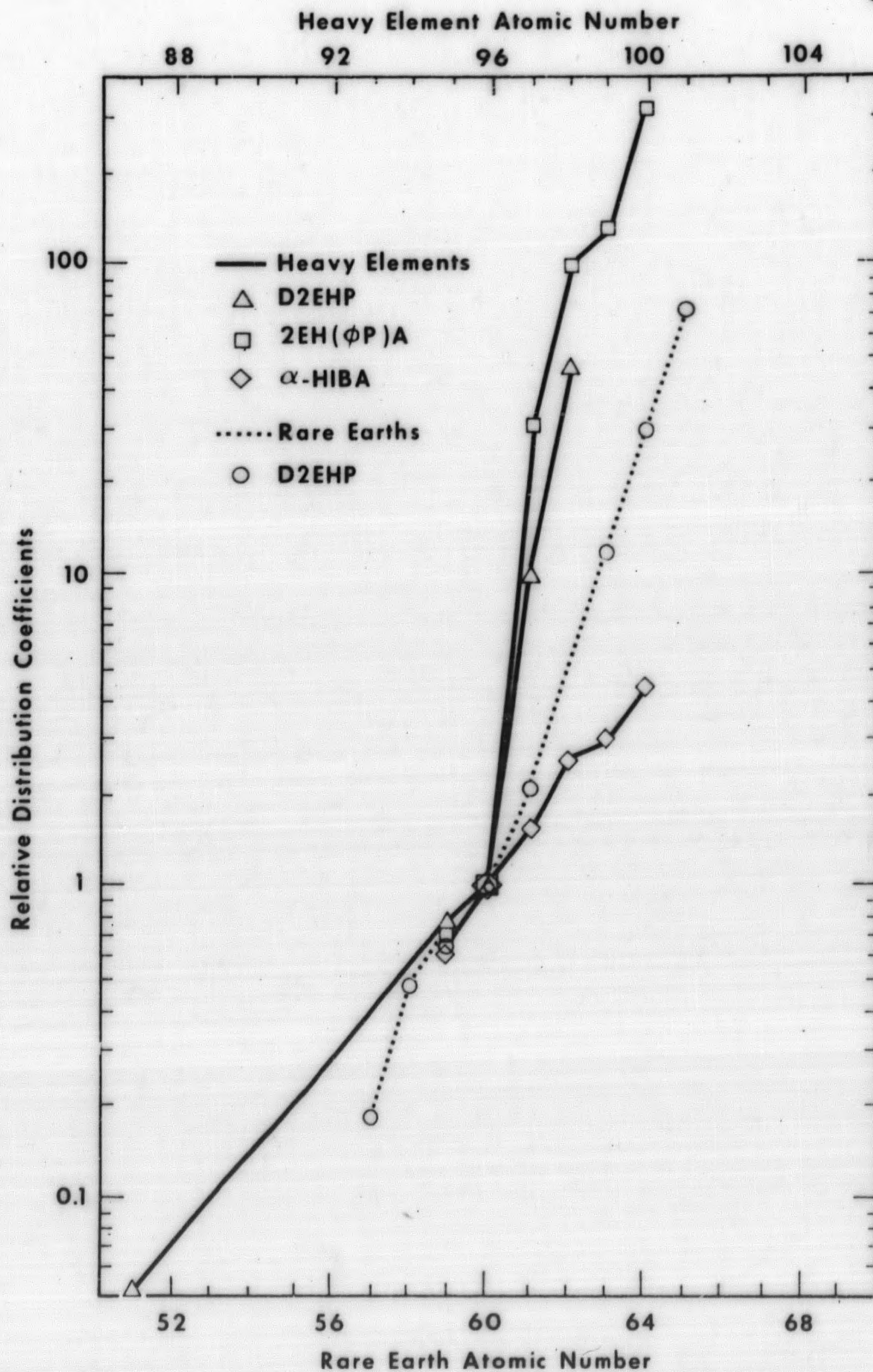


FIGURE 4. RELATIVE DISTRIBUTION COEFFICIENTS FOR RARE EARTHS AND HEAVY ELEMENTS VERSUS ATOMIC NUMBER

of the mono species which has been determined to be $\sim 0.01\%$ in the present D2EHP study. [1]

The heavy element points are also plotted in Figure 4. It is seen that a large change in the separation factor occurs between curium and berkelium for both the D2EHP and 2-EH(ϕ P)A. The α -HIBA system on the other hand shows only a uniform variation. The relatively large distribution coefficients which occur for cerium and berkelium may be related to their tendency to form +4 valance, not unlike the plutonium only to a much smaller extent.

The data indicate that separation factors for rare earths in the D2EHP system are greater than those of the Dowex-50 system. For the heavy elements, the separation factors appear to be greater with D2EHP or 2-EH(ϕ P)A in most instances than with Dowex-50 system.

On the basis of the foregoing, it is concluded that the D2EHP system will separate with large decontamination factors all heavy elements except curium from neodymium. Even the curium can be separated by a factor of 100 from the neodymium (Figure 3) which is adequate for many fuel irradiation conditions.

C. DISASSEMBLY AND ANALYSIS OF IRRADIATION ASSEMBLIES

Three assemblies have been irradiated. The first, identified as GEV-1, was irradiated in the MTR in a thermal flux in a position where the cobalt cadmium ratio is 30. It held three capsules, one of which contained a 1-gm foil of U-235, one a 1-gm foil of Pu-239, and the third a 1-gm foil of U-233. The relative thermal fission yields for stable fission product nuclides useful for burnup indicators are to be determined on these foils.

The second assembly, GEV-2, was irradiated in VBWR in a hardened neutron spectrum and contained, in addition to the same three fissionable foils, some stable fission product elements in both unshielded and cadmium-shielded capsules. The effect of the hardened neutron spectrum on the fission yields of the stable fission product nuclides and the thermal neutron absorption cross section and resonance integrals for stable fission product nuclides selected as burnup indicators are to be determined from this irradiation.

The third assembly, GEV-3, was irradiated in VBWR and contains primarily Th-232 and U-238 to measure the fission yield of the stable nuclides in the fast fission of these fertile materials. The progress which has been made on the opening and analysis of these assemblies is described below:

1. MTR Irradiated GEV-1

This assembly has been opened in the Radioactive Materials Laboratory hot cells and each of the three fueled capsules has been cut open. The U-235 foil, the Pu-239 foil, and the U-233 foil have each been individually dissolved. The xenon and krypton fission gases from each have been trapped on charcoal for subsequent mass analysis. The resulting solutions were each analyzed for stable fission product neodymium nuclides and for the fuel isotopic content. Additional analyses for Cs, Ba, Ce, and Sm, which comprise a major portion of the heavy mass fission yield peak, will permit the calculation of the relative thermal fission yields of neodymium isotopes for U-233, Pu-239, in addition to those for U-235. No difficulties are foreseen in the analysis of these additional isotopes.

2. VBWR Irradiated GEV-2

This assembly which was irradiated in a hardened thermal flux has also been opened. The assembly was 40 inches long and contained foils of U-235, Pu-239, and U-233 similar to those in GEV-1 and specimens of stable fission product elements including neodymium in both unshielded as well as in CdO-shielded capsules.

The stainless steel outer tube from this assembly has been gamma scanned over its entire length through a 125-mil slit in the 3-ft wall of the hot cell by pulling it upward across the slit by means of a motor driven winch. A sodium iodide scintillation spectrometer displayed the γ spectrum on a RCL 256-channel analyzer. The analyzer was set to accept only the Co-60 photopeaks and the relative integrated thermal flux profile over the length of the assembly was obtained by operating the pulse height analyzer in the multiscaler mode. Cobalt and iron flux wires were

removed from inside the individual capsules and sectioned into 1/2-inch lengths to determine the absolute values of thermal and fast flux exposures which are necessary to determine absolute capture cross sections of stable fission product nuclides to be used as indicators of burnup.

3. VBWR Irradiated GEV-3

This assembly has completed its irradiation and is cooling at the present time. It contains primarily Th-232 and U-238 oxides under cadmium oxide. This material was irradiated to furnish material for the determination of fission yields of stable fission product nuclides, including neodymium, in fast fission of Th-232 and U-238. In the use of fast reactors, and particularly breeder reactors, the fast fission of these isotopes takes on increasing importance and measurement of the fission yields of the stable fission product nuclides in these materials will be undertaken when the analysis of foils from the first two assemblies are further along.

The principal effort during the next quarter will be concentrated on the determination of fission yields and capture cross sections of the nuclides of stable fission product neodymium.

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END