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Separation Science and Technology

Publication details, including instructions for authors and subscription information:

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Online publication date: 01 April 2000

To cite this Article Mohapatra, P. K. , Sriram, S. , Manchanda, V. K. and Badheka, L. P.(2000) 'Uptake of Metal Ions by Extraction Chromatography Using Dimethyl Dibutyl Tetradecyl-1,3-malonamide (DMDBTDMA) as the Stationary Phase', Separation Science and Technology, 35: 1, 39 – 55

To link to this Article: DOI: 10.1081/SS-100100142

URL: <http://dx.doi.org/10.1081/SS-100100142>

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Uptake of Metal Ions by Extraction Chromatography Using Dimethyl Dibutyl Tetradecyl-1,3-malonamide (DMDBTDMA) as the Stationary Phase

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ABSTRACT

The uptake of several actinides and fission products such as U(VI), Pu(IV), Am(III), Eu(III), Cs(I), and Sr(II) from nitric acid solutions by a novel extraction chromatographic resin containing dimethyl dibutyl tetradecyl-1,3-malonamide (DMDBTDMA) has been investigated. The order of uptake follows the order $\text{Pu(IV)} > \text{U(VI)} \gg \text{Am(III)} \sim \text{Eu(III)} > \text{Sr(II)} \sim \text{Cs(I)}$. The possibility of this resin material sorbing trace concentrations of actinide ions from nitric acid solutions containing relatively larger amounts of Nd(III) and U(VI), as well as from simulated pressurized heavy water reactor (PHWR) waste solution, has been evaluated. The uptake of Am(III) in the presence of NaNO_3 , as well as in the presence of a macro concentration of Fe(III), has been investigated. The capacity of the resin for Am(III) and the elution behavior of the actinide ions from the resin were also studied.

INTRODUCTION

Recovery of trivalent actinide ions from waste solutions containing moderate concentrations of nitric acid has been a challenging task. Organophosphorous extractants such as substituted carbamoyl methyl phosphonate (CMP) and carbamoyl methyl phosphine oxide (CMPO) have been found useful for the extraction of trivalent actinide ions from moderate nitric acid concentra-

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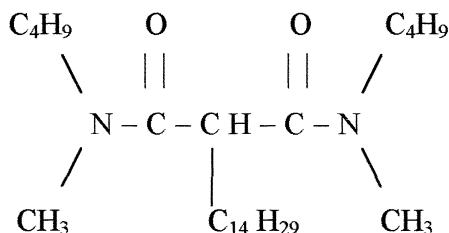


FIG. 1 Structure of dimethyl dibutyl tetradecyl-1,3-malonamide (DMDBTDMA).

tions (3–6 M HNO₃) (1, 2) while reagents such as trialkyl phosphine oxide and their mixtures with substituted isoxazolones have been quite promising at lower acidities (1–2 M HNO₃) (3, 4).

Pentaalkyl malonamides are becoming increasingly more relevant as the extractants for such applications due to their special characteristics like 1) complete incinerability which helps in reducing the secondary waste volume significantly, 2) relatively innocuous degradation products which can easily be washed out, and 3) reasonably high decontamination factors obtained for the trivalent actinide ions vis-à-vis fission products at moderate acidity (3–5 M HNO₃). Out of the several diamides studied by Nigond et al. (5–7) and Mahajan et al. (8), dimethyl dibutyl tetradecyl-1,3-malonamide (DMDBTDMA), shown in Fig. 1, was found to hold excellent promise for the minor actinide partitioning from high level nuclear waste solutions.

Extraction chromatographic separation methods combine the advantages of both solvent extraction and ion-exchange techniques (9). Horwitz et al. (10) prepared a CMPO-coated chromatographic resin material for the separation and assay of trivalent actinide ions from environmental as well as nuclear waste samples. The present work deals with the preparation of a novel chromatographic resin material with DMDBTDMA as a stationary phase and its use for the uptake of Am³⁺, UO₂²⁺, Pu⁴⁺, and fission products under varying nitric acid and loading conditions. Musikas et al. (11) reported that the presence of Fe(III) severely affects the extraction behavior of Am(III). The effect of a macro concentration of Fe(III) (in the presence of oxalic acid as a complexing agent) on the distribution of Am(III) has also been investigated.

EXPERIMENTAL

Reagents

The ligand DMDBTDMA was synthesized in the laboratory following a reported method (8). Chromosorb W (60–80 mesh) and Chromosorb 102 (100–120 mesh) obtained from Johns Manville and Amberlite XAD-4 and Amberlite XAD-7 (both 20–60 mesh) procured from Sigma Chemical Co.,



USA, were thoroughly washed with acetone followed by air drying before use. Spec pure Nd_2O_3 was obtained from Johnson Matthey, England, and uranyl nitrate was prepared by dissolving UO_3 (obtained from Uranium Metal Plant, B.A.R.C.) in nitric acid. All the other reagents were of A.R. grade and were used as received.

Preparation of the Extraction Chromatographic Resin

The extraction chromatographic resins were prepared by impregnating Chromosorb W, Chromosorb 102, Amberlite XAD-4, and Amberlite XAD-7 with an equivalent quantity of the diamide (w/w) solution in acetone. The slurry was equilibrated for 24 hours, followed by solvent removal by flushing with nitrogen gas. The resultant material was vacuum dried to constant weight. Alternatively, the resin material was prepared by making a slurry of diamide in a methanol–water solution (1:1) (12). The results obtained by using the impregnated resins obtained by both these methods were comparable. However, for the present studies the material obtained by the former procedure was used.

Radioactive Tracers

The tracers ^{241}Am , ^{233}U , and ^{239}Pu were prepared and purified as described earlier (13). ^{137}Cs and $^{152,154}\text{Eu}$ were procured from BRIT, Mumbai, while $^{85,89}\text{Sr}$ was obtained by irradiating SrCO_3 in a nuclear reactor with a flux of 1×10^{13} neutrons/cm²/s. The valency state of Pu was adjusted to +4 by a reported method (14).

Radiometric Measurements

Assay of ^{241}Am , $^{152,154}\text{Eu}$, ^{137}Cs , and $^{85,89}\text{Sr}$ was done by gamma counting in a NaI(Tl) scintillation counter. Alpha counting for ^{233}U and ^{239}Pu was done by a liquid scintillation counter employing a toluene-based cocktail of 0.7% (w/v) PPO, 0.03% (w/v) POPOP, and 2% (v/v) HDEHP in toluene as the scintillator.

Distribution Ratio Measurement

The sorption of the radionuclides from nitric acid medium was measured by contacting a known amount of the resin material (usually 15 mg in the case of tracer studies and 25 mg in experiments containing U/Nd/Fe carrier) with a known volume of the desired acid containing the radiotracer. Agitation of the two phases was carried out in stoppered glass tubes in a thermostated water bath, usually for 1 hour, though equilibrium was attained in under 20 minutes. Subsequently, the tubes were centrifuged and 100 μL of the aqueous phase

was withdrawn for assaying the activity radiometrically. The distribution ratio values, $D_{w,M}$, were estimated using

$$D_{w,M} = [(C_0 - C_s)/W]/(C_s/V) \quad (1)$$

where C_0 and C_s are the concentrations of the metal ions (in counts per unit time per unit volume) before and after equilibration, respectively; W is the weight of the resin employed (in grams); and V is the volume of the aqueous phase (in mL). For determining D_{w,HNO_3} in place of $D_{w,M}$, equilibration experiments were carried out similarly in the absence of tracer. The concentration of HNO_3 in the aqueous phase was determined volumetrically before and after equilibration.

Column Preparation and Characterization

The column was prepared by packing about 500 mg of the resin material in a glass column of about 4 mm diameter. In the present studies the flow rate was adjusted to about 4–6 drops (0.3 mL/min) by changing the packing density of the glass wool plug in the column and the liquid column height above the glass wool plug. The bed volume of the column was estimated from the length of the bed and the i.d. of the column. The bed density was calculated from the bed volume and the weight of the packed chromatographic material. The volume of the stationary phase (v_s) and the volume of the mobile phase (v_m) were estimated by the method reported by Horwitz et al. (15).

Elution Behavior

The elution profiles were obtained after loading a known amount of the tracer followed by elution with 0.01 M nitric acid or the other strippants such as 0.05 M oxalic acid and 0.1 M sodium carbonate. All the runs were carried out at ambient temperature ($25 \pm 0.1^\circ\text{C}$). The elution profiles of U(VI), Pu(IV), and Am(III) were obtained in this manner.

RESULTS AND DISCUSSION

Comparison of Various Resin Materials

As mentioned above, the resin material was prepared by two different methods, viz., from acetone medium as well as from a methanol–water (1:1) medium (12). The sorption data of Am(III) from the HNO_3 medium in the 0.01–6 M range suggests that there is reasonable agreement between the results obtained using materials prepared by these two methods (Fig. 2). The data for Am^{3+} uptake from 3 M HNO_3 with different resin materials, viz., Chromosorb 102, Chromosorb W, Amberlite XAD-4, and Amberlite XAD-7 coated with DMDBTDMA, is shown in Table 1. With the exception of Chromosorb W, which is Celite diatomaceous silica, the rest of the inert supports employed are



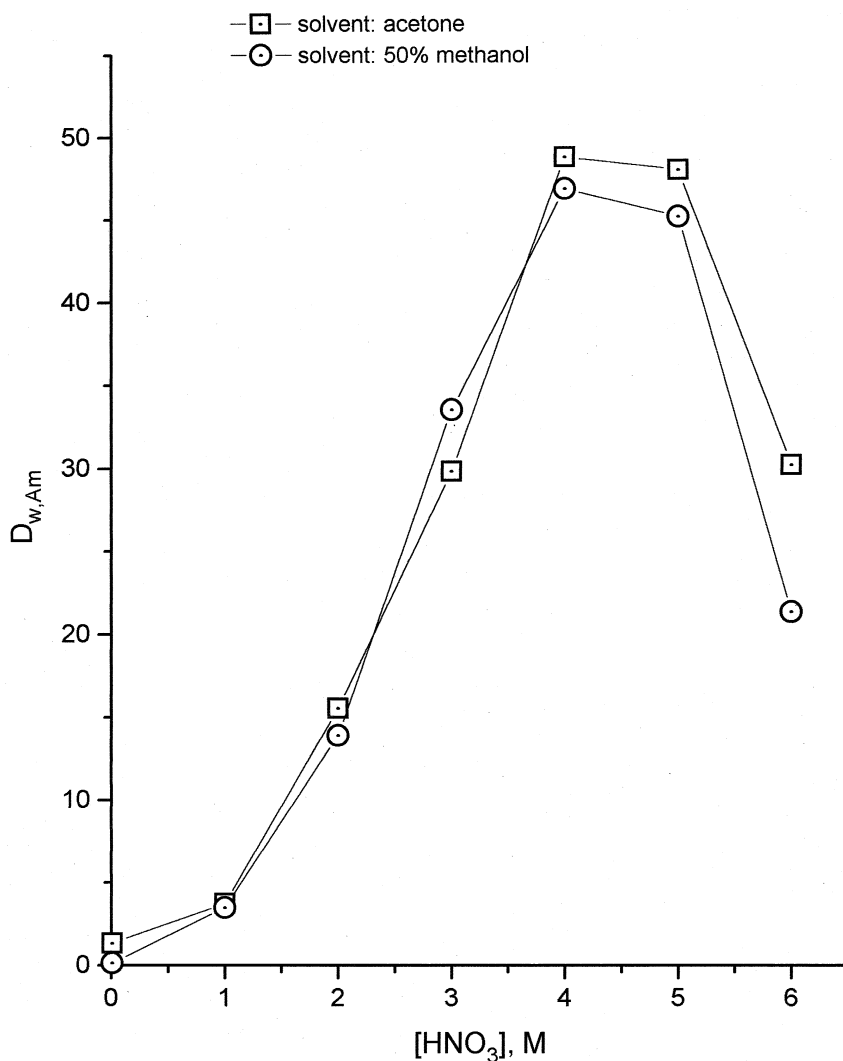


FIG. 2 Variation of $D_{w,Am}$ with nitric acid concentration. Data obtained with the resin material prepared from acetone as well as 50% methanol in water medium.

TABLE 1
 Distribution Ratio Values for ^{241}Am between HNO_3 and Chromatographic Resin Prepared from Different Support Materials. Weight of Resin Material: ~ 15 mg; Volume of Aqueous Phase: 0.5 mL; $[\text{HNO}_3]$: 3.0 M

Solid support material	Mesh size	$D_{w,Am}$
Amberlite XAD-4	20-60	1.88
Amberlite XAD-7	20-60	16.3
Chromosorb W	60-80	31.2
Chromosorb 102	100-120	27.2



TABLE 2A
Characteristics of the Novel Extraction
Chromatographic Resin Material Using the Diamide
DMDBTDMA

Stationary phase	DMDBTDMA
Support material	Chromosorb W
Mesh size	60–80
Extractant loading	50 wt%
Average density of the resin	1.096 g/mL

TABLE 2B
Characteristics of the Packed Column Using the Resin Material in
Table 2A

Bed volume	1.07 mL
Density of stationary phase (g/mL)	0.921
Stationary phase volume (v_s)	0.254 mL/mL of bed
Bed density (g/mL)	0.467
Mobile phase volume (v_m)	0.574 mL/mL of bed
Capacity of column	11 mg of Am(III)/g of resin

nonionic polymeric resins. The highest $D_{w,Am}$ value was obtained for Chromosorb W (60–80 mesh). Therefore, this material was chosen for subsequent study. Variation of $D_{w,M}$ with extraction chromatographic resins using CMPO as the stationary phase and different inert supports was also reported previously (16). It was found that $D_{w,Am}$ varied by a factor of ~ 80 by changing the inert support from XAD-4 ($D_{w,Am} = 30.8$) to Chromosorb 102 ($D_{w,Am} = 2.5 \times 10^3$). Various factors like particle size (mesh), pore diameter, pore volume, and the presence of impurities could influence the extraction chromatographic behavior. Tables 2A and 2B give the specifications for the resin material as well as the packed column used in the present work.

Time of Equilibration

The agitation time for metal ion uptake by the resin material was studied by carrying out equilibration for different time intervals. Though equilibrium was attained in about 20 minutes (Fig. 3), for all subsequent experiments an equilibration time of 1 hour was used.

Uptake of Metal Ions from Varying HNO_3 Concentration

The uptake of metal ions such as UO_2^{2+} , Pu^{4+} , Am^{3+} , Cs^+ , and Sr^{2+} from varying concentrations of nitric acid was investigated by equilibrating the



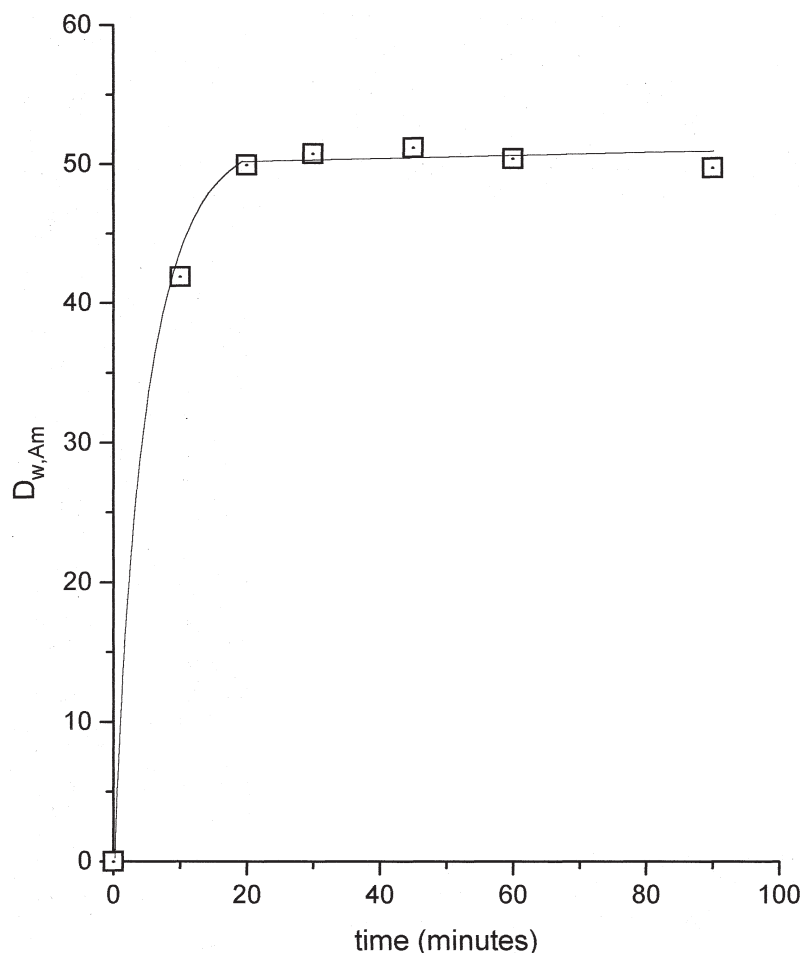


FIG. 3 Effect of agitation time on Am(III) uptake. $[HNO_3] = 4.0\text{ M}$.

aqueous solutions of desired compositions spiked with the required radioactive tracer with a known quantity of the resin material (15–20 mg). Figure 4 shows the plot of $D_{w,M}$ with varying HNO_3 concentration which suggests that the order of extraction is $Pu^{4+} > UO_2^{2+} > Am^{3+} \sim Eu^{3+} > Cs^+ \sim Sr^{2+}$. A similar trend was observed with the solvent extraction studies reported earlier (8). However, the absolute $D_{w,M}$ values in the extraction chromatography and solvent extraction studies can not be compared. Distribution ratio values for different actinide ions in the present system follow the trend reported earlier for CMPO (16) and Diphonix (17) resins (Table 3). Absolute values (at 4 M HNO_3) for each metal ion follow the order CMPO > Diphonix > DMDBDT-DMA. Relatively lower values in the present work do not present any problem since column parameters (like bed volume and flow rate) can be suitably selected for the desired separation. There is distinct advantage of employing DMDBDT-DMA over organophosphorous extractants in waste management



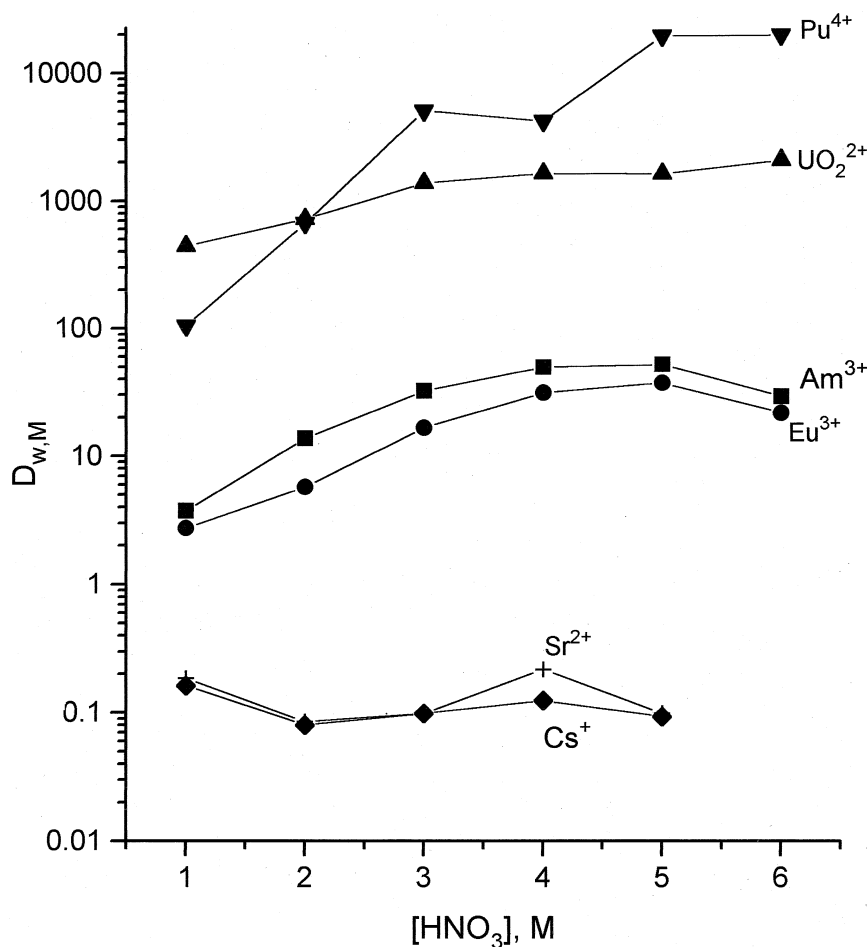


FIG. 4 Variation of $D_{w,M}$ values with HNO_3 concentration for several actinides and fission products.

TABLE 3
Comparison of Distribution Ratio Values ($D_{w,M}$) for Different Actinide Ions
Using DMDBTDMA, CMPO, and Diphonix-Based Resins

Metal ion	$D_{w,M}$		
	DMDBTDMA resin	CMPO ^a resin	Diphonix ^b resin
Pu(IV)	4000	3×10^5	9×10^4
U(VI)	1600	1×10^5	6×10^4
Am(III)	50	3.5×10^3	150

^a Ref. 16.

^b Ref. 17.



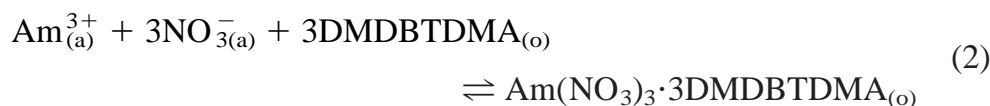
TABLE 4
Nitric Acid Uptake by the Resin Material

Weight of resin (mg)	Initial [HNO ₃] (M)	D_{w,HNO_3}
100.52	1.00	0.28
100.87	2.00	0.30
100.01	2.95	0.32
100.54	4.24	0.42
99.84	5.31	0.47

due to the complete incinerability of the former. Nitric acid uptake was determined by equilibrating aqueous solutions of various nitric acid concentrations (1–5 M HNO₃) with ~100 mg of the resin material (Table 4). D_{w,HNO_3} was found to be steady between 1 and 3 M HNO₃ and increased thereafter, suggesting a change in the nature of the species extracted at higher nitric acid concentrations.

Sorption of Am under Loading Conditions

Extraction of Am³⁺ by DMDBTDMA follows the chemical equilibrium



The sorption behavior of Am(III) was investigated by batch studies under varying Nd(III) and U(VI) loading conditions (Fig. 5) up to ~20 g/L at 4.0 M HNO₃. The $D_{w,Am}$ value of 48 decreases sharply to ~1.8 with increasing U loading to 20 g/L and to 4.0 with increasing Nd(III) loading to 18.25 g/L. Qualitatively, similar results have been reported by us in earlier solvent extraction studies (8). The decrease in $D_{w,U}$ values with increasing U loading has, on the other hand, been more spectacular (Fig. 6). About 3.4 g/L of uranium corresponded to complete saturation of the resin. The increase in the $D_{w,U}$ values at lower concentration of U(VI) is attributed to the salting-out effect while the sharp decrease at higher concentration is due to the competition of natural U (U-238) with U-233 tracer.

Sorption of Am(III) from Simulated Waste of Pressurized Heavy Water Reactor (PHWR)

Batch studies were carried out with a simulated PHWR waste solution, the composition of which is given in Table 5. The results suggest a sharp decrease in the $D_{w,M}$ values due to the large amount of cations present in the waste so-



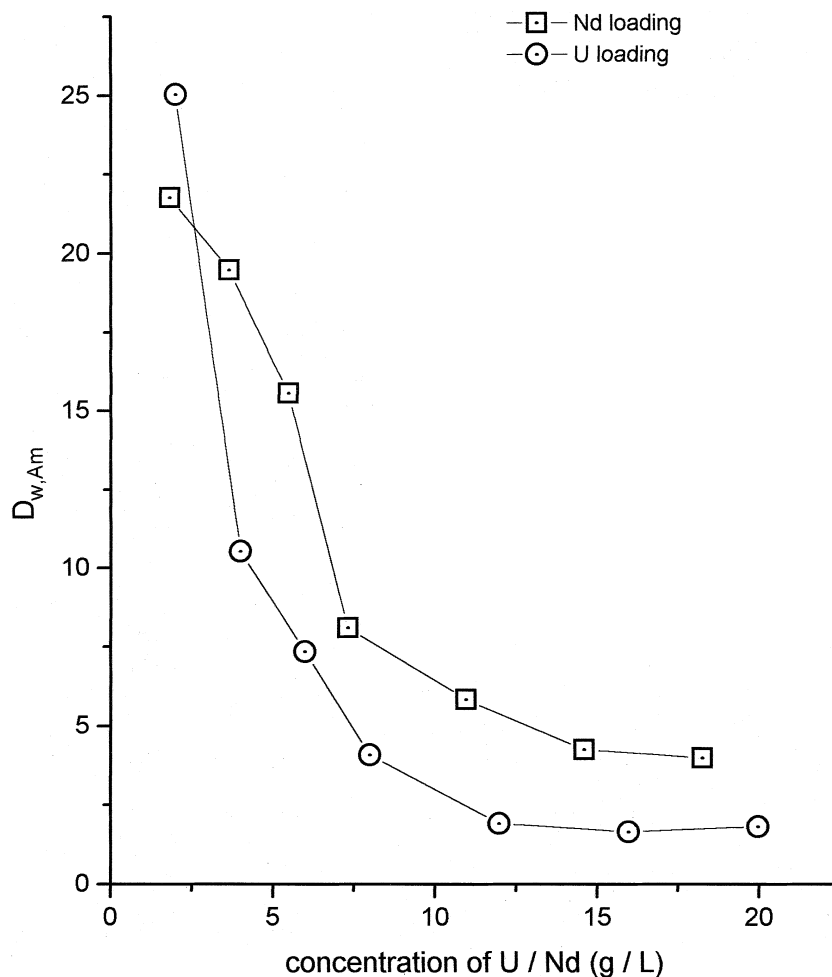


FIG. 5 Am uptake from 4.0 M HNO₃ under varying U/Nd loading.

lution (Table 6). Major cations responsible for the decrease in Am, U, and Pu sorption by the resin are lanthanides and Fe. These elements are known to extract with DMDBTDMA and thereby decrease the free ligand available for the extraction of actinides. These values increased significantly if the waste solution was preequilibrated twice with 20% tributyl phosphate (TBP) in dodecane. Thus chromatographic resin material can be used for the uptake of actinide ions including Am(III) from PHWR-HLW if pretreated with 20% TBP.

Sorption of Am(III) from Sodium Nitrate Medium

The sorption of Am(III) from sodium nitrate solution is particularly useful for the recovery of minor actinides from sodium-bearing waste solutions. Figure 7 shows the batch distribution data at various nitric acid concentrations in the presence as well as in the absence of 2.0 M NaNO₃. It is interest-



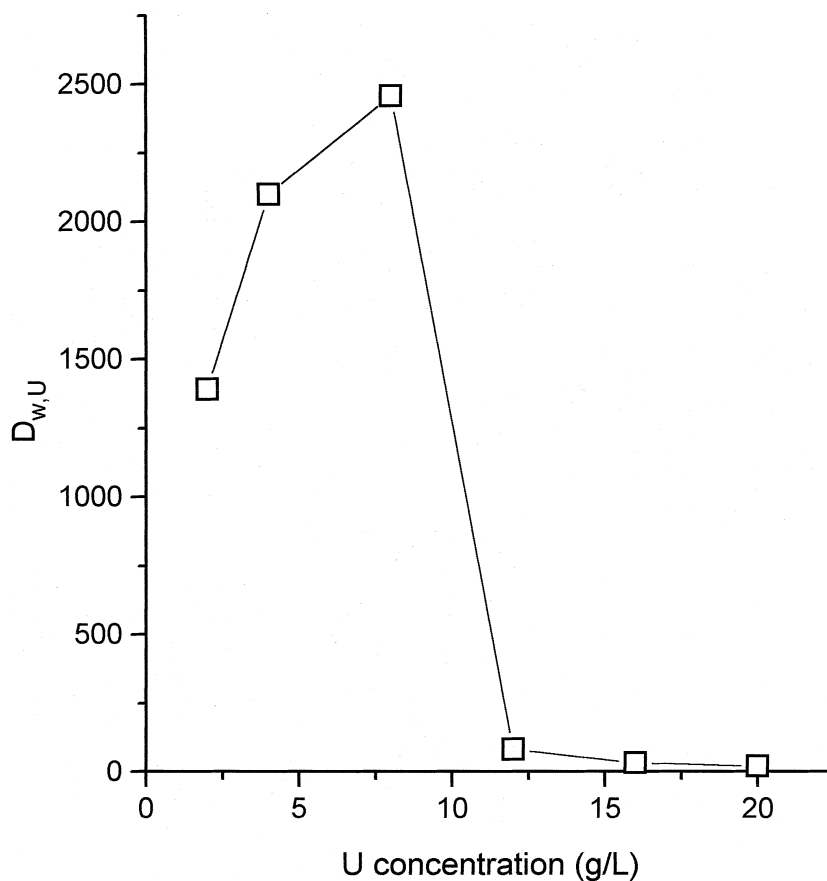


FIG. 6 U uptake from 4.0 M HNO_3 under varying U loading.

TABLE 5
Composition of Simulated PHWR High Level Waste (HLW) in 4.0 M HNO_3

Salt	Amount (mg/L)	Salt	Amount (mg/L)
SeO_2	13.37	$\text{Rb}_2\text{U}(\text{SO}_4)_3$	149.4
$\text{Sr}(\text{NO}_3)_2$	186.3	$\text{YCl}_3 \cdot 6\text{H}_2\text{O}$	100.7
ZrO_2	771.0	$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$	735.1
$\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$	503.0	Rh metal	132.3
PdCl_2	267.5	AgNO_3	19.68
$\text{Cd}(\text{CH}_3\text{COO})_2$	16.33	SnCl_2	15.56
Sb metal	4.7	Te metal	102.7
CsNO_3	546.4	$\text{Ba}(\text{NO}_3)_2$	309.4
$\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$	308.7	$\text{Ce}_2(\text{SO}_4)_3$	1,066.6
$\text{NdCl}_3 \cdot 6\text{H}_2\text{O}$	866.9	Eu_2O_3	11.96
$\text{SmCl}_3 \cdot 6\text{H}_2\text{O}$	211.3	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	507.2
NaNO_3	2,648	$\text{K}_2\text{Cr}_2\text{O}_7$	100
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	100.1	KMnO_4^a	181.7
$\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	20,000		

^a Taken in place of Tc.



TABLE 6
Distribution Ratio Values of Am^{3+} , Pu^{4+} , and UO_2^{2+} (present as tracers) in the Simulated PHWR-HLW Solution (Table 5)

Aqueous solution	D_{Am}	D_{Pu}	D_{U}
Using simulated PHWR-HLW as such	0.925	23.50	13.56
Above solution after 1st preequilibration ^a	4.873	2354	1124
Above solution after 2nd preequilibration ^a	6.822	3343	1423

^a Preequilibration was done using 20% TBP in dodecane.

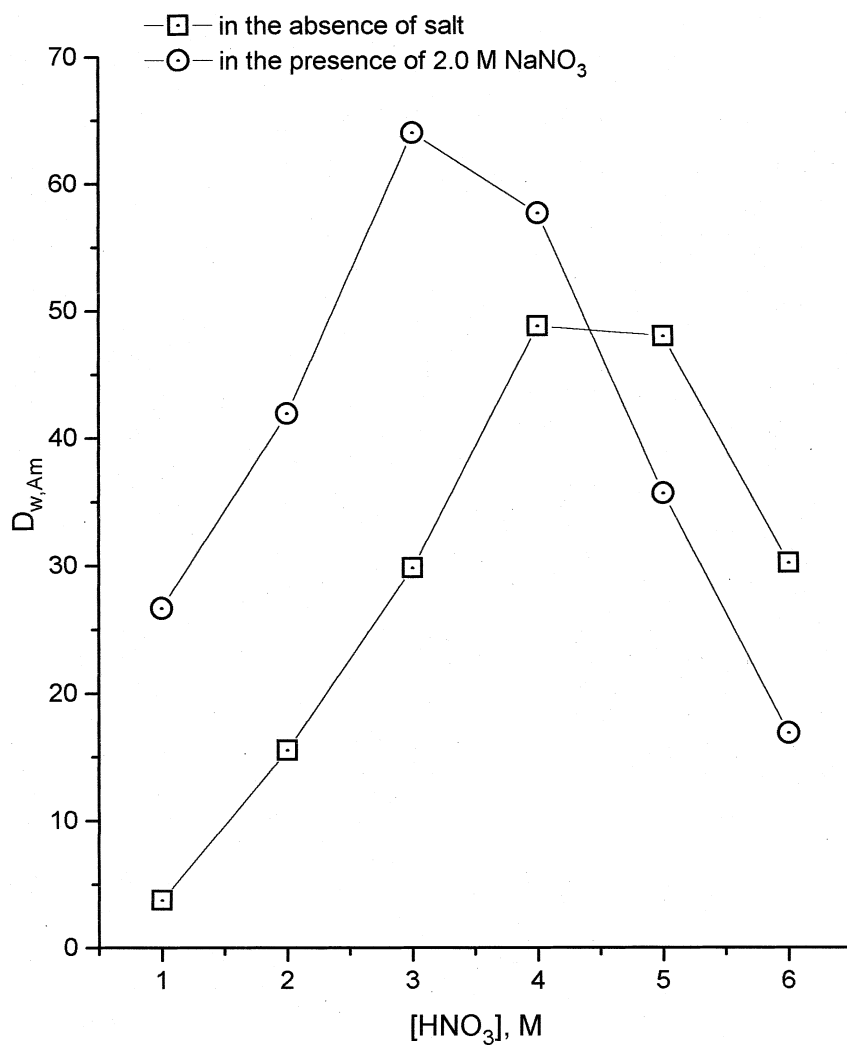


FIG. 7 Am uptake from varying HNO_3 concentration in the presence and absence of 2.0 M NaNO_3 .



ing to note that the $D_{w,Am}$ values increased in the presence of $NaNO_3$ at lower HNO_3 concentrations (1–4 M), which was ascribed to the salting-out effect. On the other hand, the $D_{w,Am}$ values decreased in the presence of 2.0 M $NaNO_3$ at 5.0 and 6.0 M HNO_3 , which was due to increased nitrate ion complexation of Am(III).

Sorption of Am(III) in the Presence of a Macro Concentration of Fe(III)

The presence of Fe(III) affects the americium ion extraction significantly (11). The effect of a macro concentration (0.5–6 g/L) of Fe(III) on Am(III) sorption was investigated at 4.0 M HNO_3 (Fig. 8). The $D_{w,Am}$ decreased significantly from ~48 to 1.85 in the presence of 6 g/L of Fe(III). Figure 8 shows

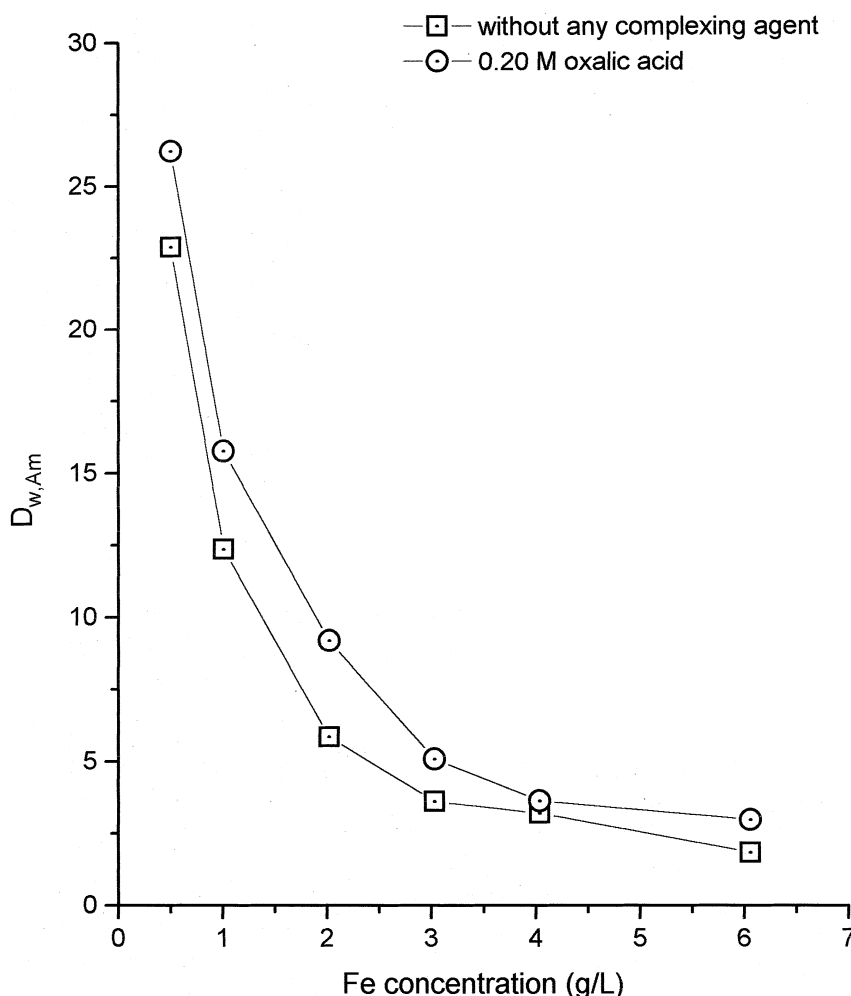


FIG. 8 Am uptake from 4.0 M HNO_3 in the presence of macro concentration of Fe(III) in the presence and absence of 0.2 M oxalic acid.



a marginal increase in the $D_{w,Am}$ values when 0.2 M oxalic acid is used to complex Fe(III) preferentially.

Column Capacity and Stripping Behavior

The capacity of the column (details given in Table 2B) with respect to Am(III) was evaluated using Nd(III) solution (1 g/L) spiked with ^{241}Am . In a triplicate experiment it was observed that 11.0 ± 0.3 mg of Nd(III) could be loaded on the column at 4 M HNO_3 without any discharge of the ^{241}Am activity.

Stripping experiments were performed after loading the column with the desired pure tracers. The stripping behavior of Am(III) was investigated by employing 0.01 M HNO_3 and 0.05 M oxalic acid as the strippants (Fig. 9). Oxalic acid was found to be a more efficient strippant for Am(III) as quantitative stripping is accomplished in ~ 3.0 bed volumes of the strippant. With 0.01 M HNO_3 ,

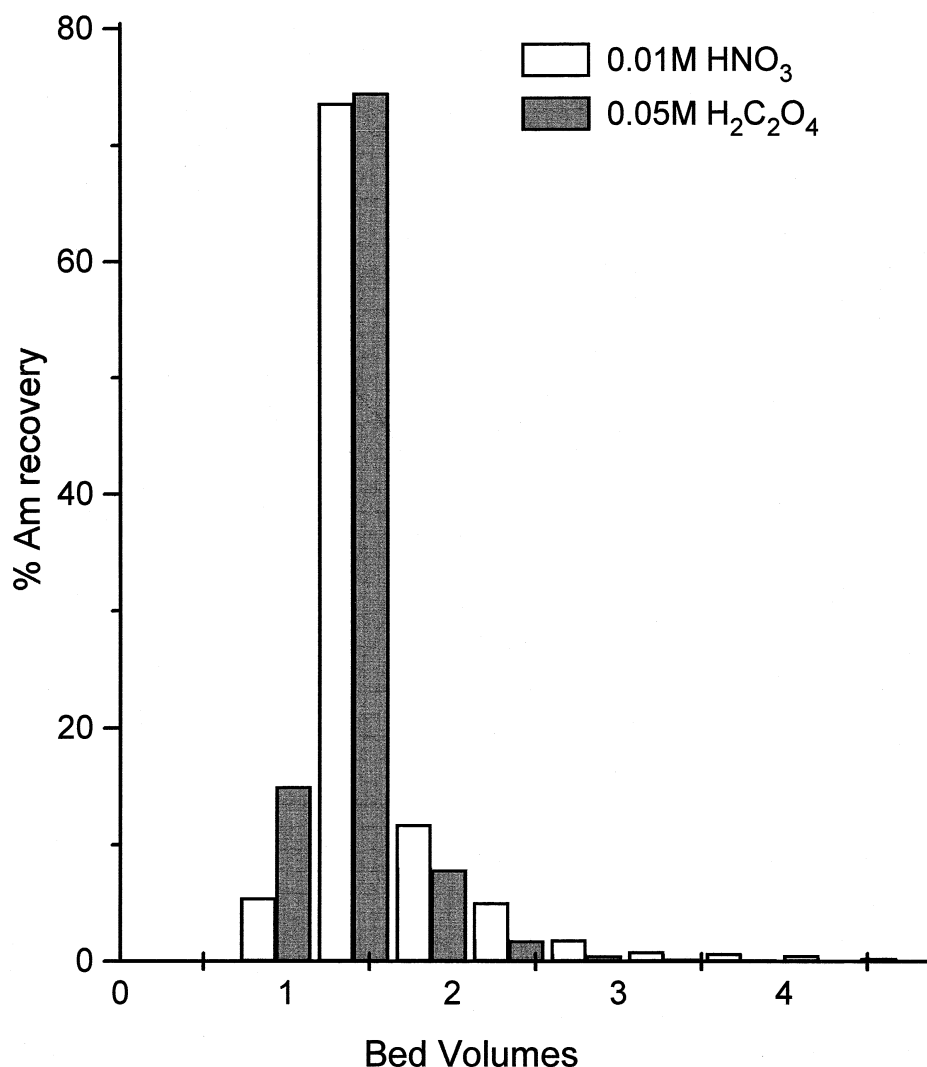


FIG. 9 Elution behavior of Am from the column using 0.01 M HNO_3 and 0.05 M oxalic acid.



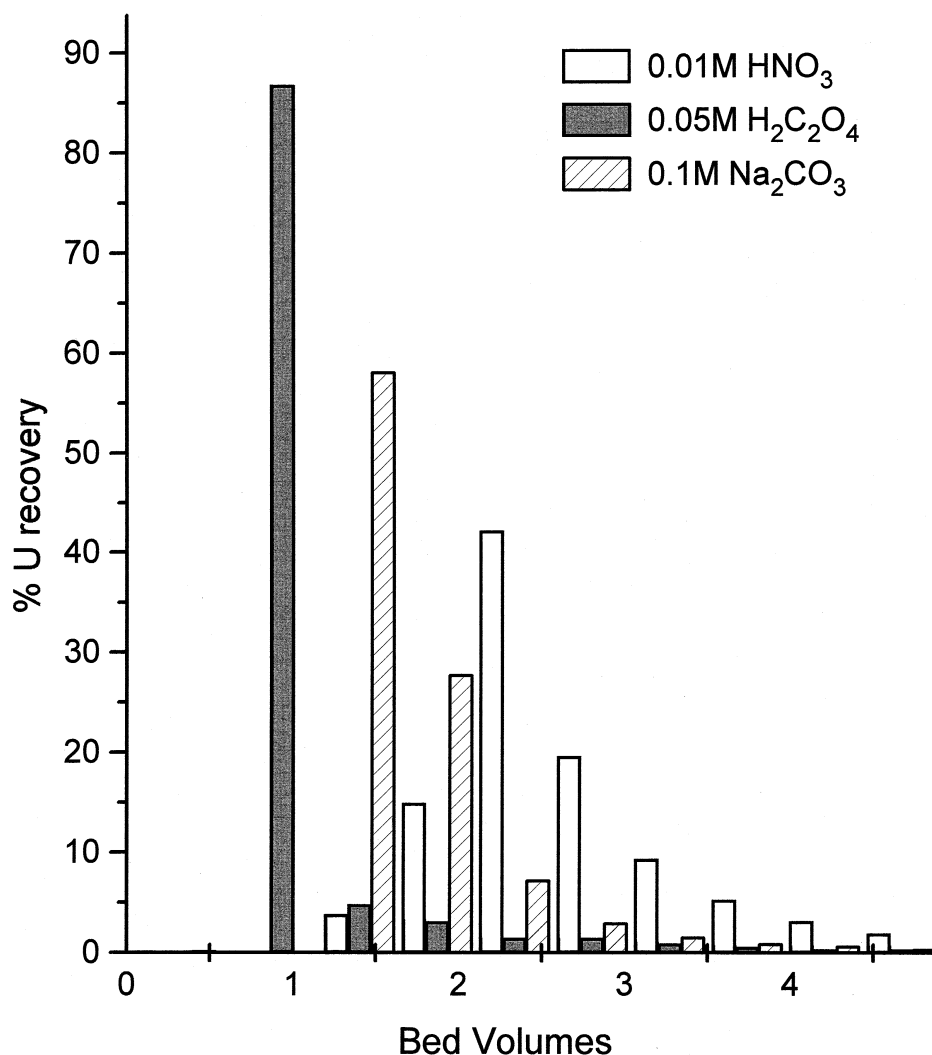


FIG. 10 Elution behavior of U from the column using 0.01 M HNO₃, 0.05 M oxalic acid, and 0.1 M Na₂CO₃.

on the other hand, though near quantitative stripping is possible with ~4.5 bed volumes of the strippant, a tailing effect was clearly noticed. The stripping behavior of U(VI) was studied employing 0.01 M HNO₃, 0.05 M oxalic acid, as well as 0.1 M sodium carbonate solution (Fig. 10). In this case Na₂CO₃ as well as oxalic acid were found to be more effective than 0.01 M HNO₃, resulting in near quantitative stripping of U(VI) in ~4.0 bed volumes of the eluants. The stripping of Pu(IV) was carried out by using both 0.05 M oxalic acid and 0.01 M HNO₃. The results with the former are shown in Fig. 11, and they suggest near quantitative stripping of the metal ion in 4.5 bed volumes of the eluant. The stripping data with 0.01 M HNO₃ are not presented here because serious material loss was observed which was ascribed to hydrolysis of the metal ion. Oxalic acid (0.05 M) has thus been found to be a promising strippant for actinides. A regenerated column was found to perform satisfactorily.

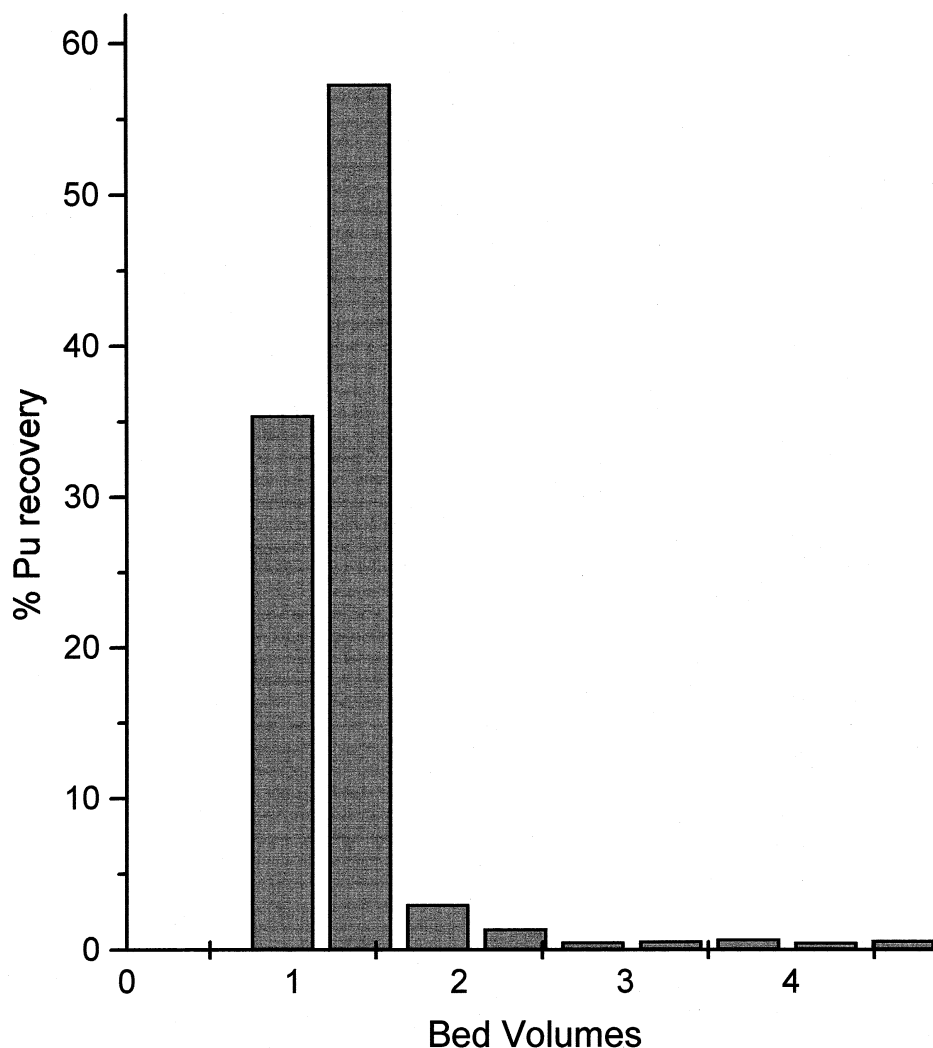


FIG. 11 Elution behavior of Pu from the column using 0.05 M oxalic acid.

CONCLUSIONS

A chromatographic resin material prepared using DMDBTDMA can be efficiently used for the uptake of trace quantity of actinide ions such as Am^{3+} , UO_2^{2+} , and Pu^{4+} from moderate acid solutions (3–5 M HNO_3). The presence of macro concentrations of Nd(III), U(VI), Fe(III), and other fission product ions present in PHWR waste solution affects the $D_{w,M}$ values significantly. Preequilibration with 20% TBP solution shows significant enhancement in the $D_{w,M}$ values. The presence of 2.0 M NaNO_3 enhances the $D_{w,Am}$ values in a lower acidity range (1–4 M HNO_3) while an opposite effect is observed at higher acidity (5–6 M HNO_3). This has been ascribed to the nitrate complexation effect. Oxalic acid (0.05 M) was found to be an effective strippant for all three actinides.



ACKNOWLEDGMENT

The authors thank Dr. S. B. Manohar, Head, Radiochemistry Division, for constant encouragement and keen interest in this work.

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Received by editor September 23, 1998

Revision received March 1999



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