Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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R. Veeraraghavan[a]; P. K. Mohapatra[a]; V. K. Manchanda[a]
[a] RADIOCHEMISTRY DIVISION, BHABHA ATOMIC RESEARCH CENTRE, TROMBAY, MUMBAI, INDIA

To cite this Article Veeraraghavan, R. , Mohapatra, P. K. and Manchanda, V. K.(1999) 'Extraction of Americium from Nitric Acid Medium Using 3-Phenyl-4-benzoyl-5-isoxazolone and Tri-*n*-octylphosphine Oxide', Separation Science and Technology, 34: 1, 123 — 137

To link to this Article: DOI: 10.1081/SS-100100641

URL: http://dx.doi.org/10.1081/SS-100100641

SEPARATION SCIENCE AND TECHNOLOGY, **34**(1), pp. 123–137, 1999

Extraction of Americium from Nitric Acid Medium Using 3-Phenyl-4-benzoyl-5-isoxazolone and Tri-*n*-octylphosphine Oxide

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R. VEERARAGHAVAN, P. K. MOHAPATRA
and V. K. MANCHANDA*
RADIOCHEMISTRY DIVISION
BHABHA ATOMIC RESEARCH CENTRE
TROMBAY, MUMBAI-400 085, INDIA

ABSTRACT

The extraction behavior of americium from acid solutions by a mixture of 3-phenyl-4-benzoyl-5-isoxazolone and tri-*n*-octyl phosphine oxide in carbon tetrachloride medium has been investigated in 1–6 M nitric acid. It is shown that Am(III) can be removed along with U(VI) and Pu(IV) quantitatively from 1 M nitric acid in a single contact and that Am(III) can be selectively stripped by using higher (>6 M) nitric acid concentrations. The distribution profile for Am(III) has been evaluated in the presence of varying amounts of U(VI) and Nd(III) in varying nitric acid media. The distribution behavior of several fission products has also been reported.

INTRODUCTION

The extraction of actinides by β-diketones and the synergistic enhancement of their extraction by neutral oxodonors has been investigated extensively (1–4). Based on the available data on the extraction of U(VI) by a series of β-diketones, Batzar et al. (5) showed an inverse relationship between the extraction constant (log K_{ex}) and acid dissociation constant (pK_a) values. The observation implied that metals which are not readily extracted from acid medium by β-diketones of high pK_a values may be extracted by those with lower pK_a values. In this connection, extensive work has been carried out in

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* To whom correspondence should be addressed. E-mail: acsrcd@magnum.barct1.ernet.in

our laboratory on the extraction behavior of actinides with 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone (PMBP), a β-diketone with a pK_a value of 4.1 (6, 7). In recent years the focus has shifted to 4-acyl derivatives of 5-isoxazolone in general, and the benzoyl derivative in particular, for the extraction of several metal ions (8–14). In view of its significantly lower pK_a value (1.1), 3-phenyl-4-benzoyl-5-isoxazolone (PBI) is capable of extracting metal ions from stronger acidic and complexing media (15). Le et al. (11) investigated the extraction of trivalent lanthanides by PBI and reported quantitative extraction of lanthanides at pH 3 in chloroform medium. The reported literature on the extraction behavior of Am(III) using PBI are restricted to pH 2 (16, 17), and there are no data available for moderate acidic media ($[H^+] > 0.1$ M). In this paper we report the solvent extraction of Am(III) from nitric acid medium ($[HNO_3] \geq 1$ M) by PBI in the presence of tri-*n*-octyl phosphine oxide (TOPO) in CCl_4 medium. The results obtained in this study may be relevant for the recovery of Am from analytical/waste/environmental samples at 1–2 M HNO_3.

EXPERIMENTAL

Materials

The PBI used in the present study was synthesized in our laboratory by the method described by Korte and Storiko (18). After recrystallization the compound was a creamish yellow-colored material melting at 145.9°C. Elemental analysis of the compound showed the C, H, and N contents as 73.3% (72.5%), 4.1% (4.2%), and 5.6% (5.3%), respectively. Pro analysi grade TOPO obtained from E. Merck, Darmstadt, was used as such. All other chemicals and reagents used were of AR grade. Actinide tracers purified by standard procedures were used in this work. ^{137}Cs and ^{85,89}Sr tracers obtained from the Isotope Division, B.A.R.C., Mumbai, were used as procured.

Distribution Studies

Equal volumes (usually 1 mL) of an aqueous phase containing the radioactive tracer and the organic phase containing the extractant mixture were taken in a glass tube and equilibrated for 1 hour in a thermostated water bath at 25°C. After equilibration and centrifugation, suitable aliquots were pipetted out for assaying. ^{239}Pu and ^{233}U were estimated by α-liquid-scintillation-counting and ^{241}Am, ^{85,89}Sr, and ^{137}Cs by γ-counting using a well-type NaI(Tl) scintillation detector. The distribution ratio (D) is defined as the ratio of counts per unit time per unit volume in the organic phase to that in the aqueous phase. All experiments were carried out in duplicate, and the precision was within ±5%.

Solubility Studies

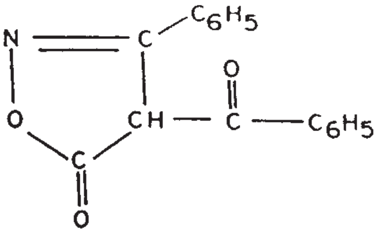
Small quantities (~10 mg) of PBI were weighed accurately and taken in six different 10 mL volumetric flasks. About 5 mL of the desired solvent (viz., xylene, CCl_4, Solvesso 100, cyclohexane, *tert*-butyl benzene, and dodecane) was added to each of these flasks. These flasks were kept in an ultrasonic bath for 30 minutes, after which the reagent was found to be completely dissolved in all cases except for the dodecane medium. Therefore, the solubility studies were restricted to the other five diluents. The volume was made up to 10 mL with the respective diluent, and absorption spectra (in the UV region) of each were recorded using a Beckman DU7 spectrophotometer after appropriate dilution. The molar absorption coefficient (ϵ) of PBI in each solvent was calculated from the absorbance obtained with the respective solvent.

In another experiment, about 50–100 mg of PBI was taken in each of the five tubes and about 2 mL of the five solvents (viz., xylene, carbon tetrachloride, cyclohexane, Solvesso 100, and *tert*-butyl benzene) was added. These mixtures were agitated for 24 hours to obtain saturated solutions of PBI in their respective diluents. The saturated solutions were appropriately diluted and their UV absorption spectra were recorded. From the absorbance values and the molar absorption coefficients in the respective medium, the solubility of PBI was evaluated.

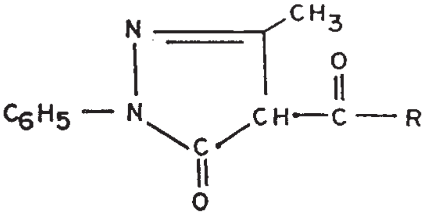
RESULTS AND DISCUSSION

Comparison with Other β-Diketones

The extraction of Am(III) with a mixture of PBI and tri-*n*-octyl phosphine oxide (TOPO) in xylene from 1 M HNO_3 was studied. For comparison purpose, experiments were also carried out with three other β-diketones [viz., TTA, PMBP, and PMAP (Fig. 1)] both in the presence and absence of TOPO. Based on the low pK_a value (1.1) which is a consequence of the number of canonical forms in which the isoxazolonate ion can exist (Fig. 2), PBI is expected to be a better extractant than the other β-diketones with their higher pK_a values. This was clearly demonstrated in our earlier work (16) where the two-phase extraction constant for Am(III) with PBI ($\log K_{ex} = 2.3$) was found to be few orders of magnitude larger than those with PMBP ($\log K_{ex} = -2.7$) and TTA ($\log K_{ex} = -7.8$). However, $\log K_{ex}$ values for the Am(III)–PBI system are not sufficiently high to enable the extraction of metal ion at 1 M HNO_3 ($D < 10^{-3}$). Table 1 shows distribution data suggesting that the percent extraction of Am(III) by the 0.05 M PBI + 0.01 M TOPO mixture in xylene medium was ~90 while in all other cases it was only ≤0.1.

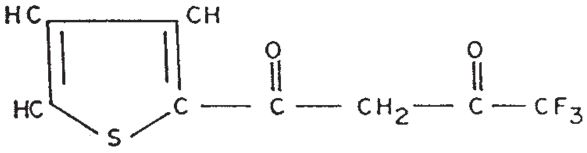


I 3-phenyl-4-benzoyl-5-isoxazolone (PBI)



II R=C_6H_5; 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone (PMBP)

III R=CH_3; 1-phenyl-3-methyl-4-acetyl-5-pyrazolone (PMAP)



IV 2-thenoyltrifluoroacetone (TTA)

FIG. 1 Structure and chemical names of β-diketones. The structures of PMBP and PMAP are similar except that the benzoyl group at the 4-position in the former has been replaced by an acetyl group in the latter.

Solubility of PBI

Though β-diketones are known to be good extractants, their poor solubility in various organic diluents creates limitations. Most of the work on the extraction by PBI reported in the literature is in the 10^{-2} to 10^{-3} M concentration range, and no data on solubility are available. Hence, it was considered appropriate to determine the solubility of PBI in some common solvents. For solubility studies on PBI alone, aromatic/cyclic diluents were initially chosen. CCl_4 was included as it is particularly suitable for processing fabrication scrap due to its high flash point (19). When a small quantity of PBI (~10 mg) was

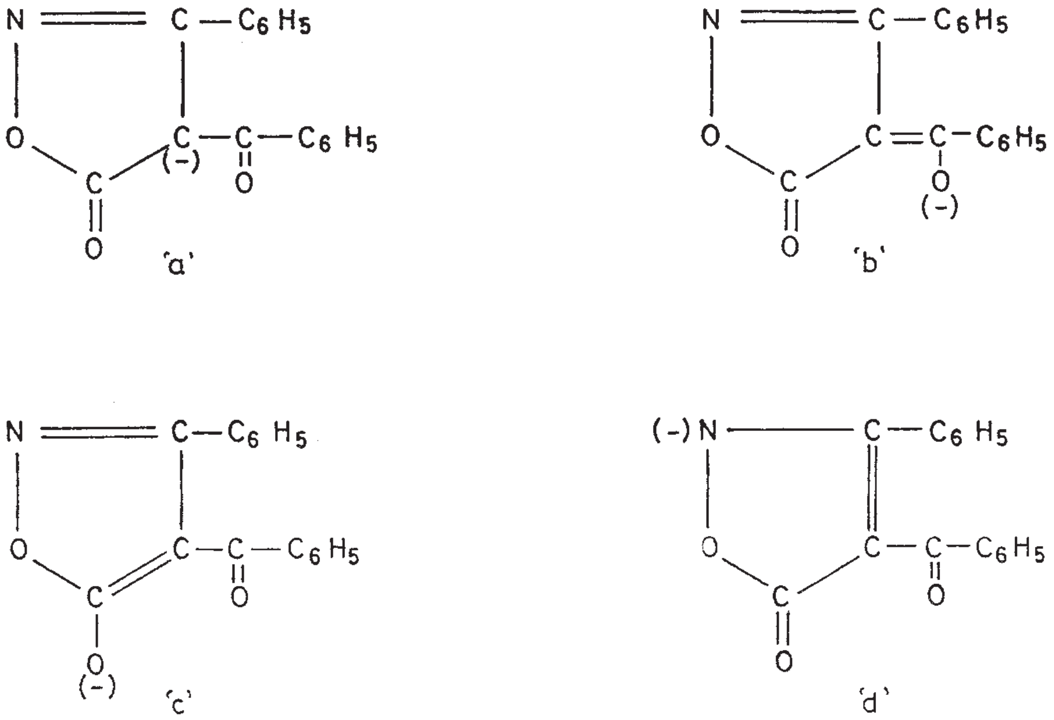


FIG. 2 Resonance structures of isoxazolonate ion.

added to 5 mL of each of the six solvents chosen, it was observed that while colored solutions were obtained in five solvents, no color was seen in the case of dodecane, suggesting poor solubility. Hence the solubility in only the other five solvents was determined. The values are presented in Table 2. The solubility of PBI was found to vary in the order xylene > *tert*-butyl benzene

TABLE 1
Extraction of Am with Different β-Diketones ($[HNO_3] = 1$ M; [β-diketone] = 0.05 M; [TOPO] = 0.01 M; diluent: xylene)

β-Diketone	pK_a	D_{Am} (β-diketone alone)	D_{Am} (β-diketone + TOPO)
PBI	1.13	$<10^{-3}$	7.86
PMAP[a]	3.2	$<10^{-3}$	$\sim10^{-3}$
PMBP	4.1	$<10^{-3}$	$\sim10^{-3}$
TTA	6.23	$<10^{-3}$	$<10^{-3}$
[b]	—	—	$<10^{-3}$

[a] 1-Phenyl-3-methyl-4-acetyl-5-pyrazolone.
[b] Only 0.01 M TOPO.

TABLE 2
Solubility of PBI in Various Diluents

Diluent	ϵ ($M^{-1} \cdot cm^{-1}$)	λ_{max} (nm)	Solubility (M)
Xylene	9,541	321–324	0.127
tert-Butyl benzene	5,334	314	0.077
Carbon tetrachloride	10,900	322	0.045
Solvesso-100	7,037	306	0.008
Cyclohexane	9,562	320	0.006

$>$ carbon tetrachloride $>$ Solvesso 100 $>$ cyclohexane. Solubility behavior is influenced by several features of diluents: dipole–dipole interaction, hydrogen bonding, and aliphatic/aromatic character. In the present work the aromatic character of xylene and *tert*-butyl benzene appears to favor the solubility of PBI. It was interesting to note, however, that the dissolution of PBI in dodecane was facilitated by the presence of TOPO. Ishii et al. (9) studied the absorption spectra of 4-(4-fluorobenzoyl)-3-phenyl-5-isoxazolone in 12% v/v aqueous 1,4-dioxane solutions of various pH values and they obtained absorption maxima in the 300–310 nm range. In the present work, PBI in various organic solvents was shown to have absorption maxima in the 306–324 nm wavelength range. The molar absorption coefficient value was found to be largest in the carbon tetrachloride medium (10,900 ± 385) and smallest in *tert*-butyl benzene (5334 ± 264).

Comparison of Various Diluents

In order to identify the best diluent to be used in synergistic extraction, an attempt was made to evaluate the efficiency of the PBI–TOPO mixture in various diluents. An aromatic diluent (xylene), an aliphatic diluent (dodecane), chlorinated solvents (CCl_4, $CHCl_3$, and CH_2Cl_2), and a polar diluent (nitrobenzene) were chosen. Extraction of Am(III) from dilute nitric acid (pH 2) by the PBI–TOPO mixture in the above solvents was evaluated. At 0.01 M HNO_3, using 0.2 M TOPO in decane as the extractant, D_{Am} has been reported to be ~1 (20). Since the solvation number of the species extracted is typically 3 for TOPO in various solvents (21), it is reasonable to assume that 0.001 M TOPO alone would give negligible extraction of Am(III). The data (Table 3) showed that antagonism occurred for the nitrobenzene medium while no change was observed for chloroform. In dodecane and dichloromethane media, the synergism was just marginal. It is generally observed that polar diluents interact with a neutral solvating extractant like TOPO, thereby reducing the effective concentration of free ligand available for synergism.

TABLE 3
Comparison of Various Diluents for Extraction of Am ($[HNO_3] = 0.01$ M; [PBI] = 0.005 M; [TOPO] = 0.001 M)

Diluent	D_{Am} (PBI alone)	D_{Am} (PBI + TOPO)
Xylene	7.35	325
Dodecane	14.3	16.6
Carbon tetrachloride	30.7	899
Chloroform	0.86	0.84
Dichloromethane	0.86	5.39
Nitrobenzene	512	156

Since the highest D_{Am} value was obtained in CCl_4, a nonpolar diluent, all further work was carried out in this medium.

Extraction of Am(III), U(VI), and Pu(IV)

The effect of TOPO concentration on Am(III) extraction was investigated at 3 M HNO_3. Figure 3 shows the extraction behavior of Am(III) at 3 M HNO_3 (0.05 M PBI) with varying TOPO concentrations in the 0.01 to 0.5

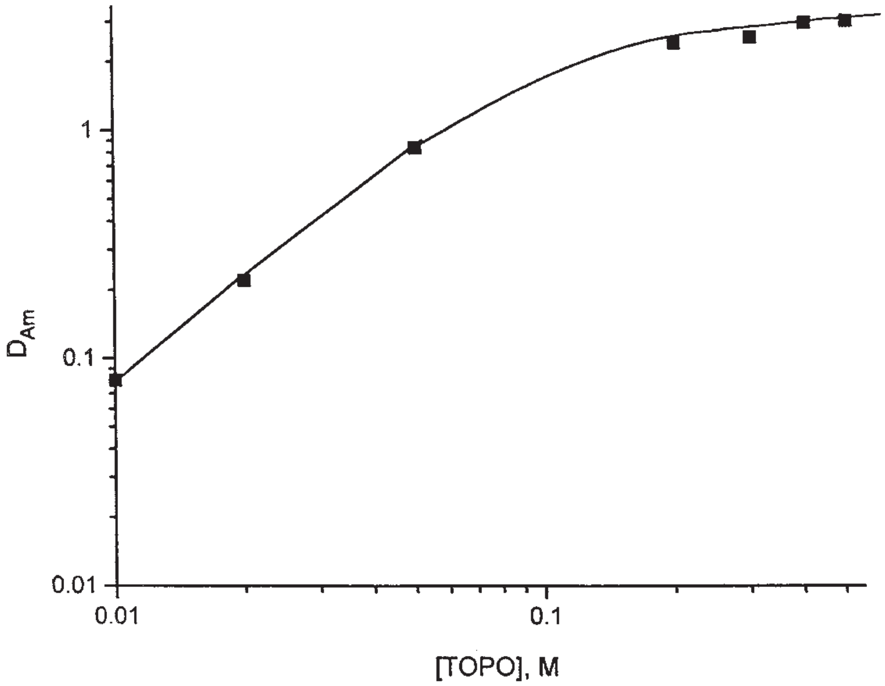


FIG. 3 Variation of D_{Am} with TOPO concentration in CCl_4. $[HNO_3] = 3$ M; [PBI] = 0.05 M.

TABLE 4
Distribution Ratio Values of U(VI), Pu(IV), and Am(III) ([PBI] = 0.05 M; [TOPO] = 0.2 M; diluent: carbon tetrachloride)

$[HNO_3]$, M	D_U	D_{Pu}	D_{Am}
1	81.4	6886	731
2	78.2	853	26.9
3	73.4	125	1.5
4	62.4	59.9	0.11
5	—	65.1	0.02
6	59.6	73.3	0.009

M range. It appears that beyond 0.2 M TOPO, PBI–TOPO interaction (22) results in a lower concentration of free PBI, thereby adversely affecting D_{Am} values. The increase in D_{Am} was observed to be sharper at lower TOPO concentrations as compared to that at higher TOPO concentrations. Healy et al. observed a similar trend in the extraction of Am(III) from 0.01 M HCl by a TTA–dibutyl butyl phosphonate mixture in hexane (23). In this range of TOPO concentration, ever though D_{Am} increased with TOPO content initially, at [TOPO] $\geq$ 0.2 M the increase is marginal (Fig. 3). In addition, at $\geq$0.3 M TOPO, on equilibration with 3 M HNO_3, solid material separated out from the mixture. All further work was therefore restricted to 0.2 M TOPO.

The extraction of Am(III), U(VI), and Pu(IV) by a mixture of 0.05 M PBI + 0.2 M TOPO in carbon tetrachloride medium was studied from 1 to 6 M HNO_3 (Table 4). U(VI) and Pu(IV) were found to be quantitatively extracted in the entire range of acidity while Am(III) extraction was quantitative only at $[HNO_3] \leq 2$ M. Figure 4 shows the comparative distribution behavior of Am as a function of HNO_3 concentration (1–6 M) for three synergistic combinations: viz., 0.2 M octyl phenyl *N*,*N*-diisobutyl carbamoyl methyl phosphine oxide (CMPO) + 1.2 M TBP in dodecane (TRUEX solvent); 0.3 M di-2-ethyl hexyl phosphoric acid (HD2EHP) + 0.2 M TBP in dodecane; and 0.05 M PBI + 0.2 M TOPO in carbon tetrachloride (the solvent used in the present work) as well as with 1 M *N*,*N*′-dimethyl-*N*,*N*′-dibutyl tetradecyl-1,3-malonamide (DMDBTDMA) in dodecane. Though the TRUEX solvent has been suggested as a potential extractant for actinide partitioning from high level waste (HLW) (24), the reagent mixture used in the present work (0.05 M PBI + 0.2 M TOPO in CCl_4) is a stronger extractant at lower acidities (<2 M HNO_3). D_{Am} was found to fall sharply beyond 2 M HNO_3, and the values suggested that $[HNO_3] \geq 6$ M could be used to reextract

Am(III) from the organic phase back into the aqueous phase. Data on the extraction behavior of Am(III) with 0.05 M PBI in CCl_4 and 0.2 M TOPO in CCl_4 are also included in Fig. 4. The extraction of Am(III) by only TOPO in carbon tetrachloride was significant only up to 2 M HNO_3. Even at these acidities, D_{Am} values were far lower than the corresponding values for the PBI + TOPO mixture. The extraction of Am by TOPO is expected to increase with HNO_3 concentration due to the salting-out effect. However, the strong interaction between HNO_3 and TOPO ($H^+ + NO_3^- + TOPO \; D \; HNO_3 \cdot TOPO$; $K_h = 8.9$) leads to the depletion of free TOPO concentration as the HNO_3 concentration increased (25). This suggested that the synergistic component of extraction is low at higher concentrations of HNO_3. Since

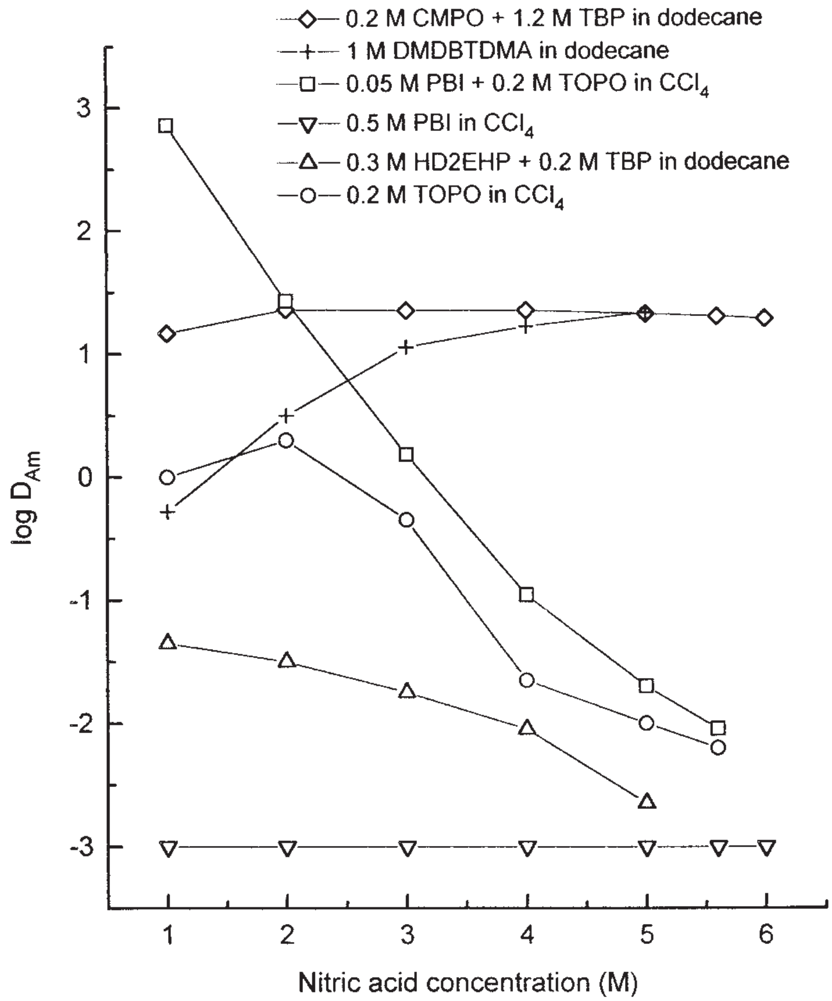


FIG. 4 Comparative data on Am(III) extraction from nitric acid medium employing different types of extractants.

Am(III) extraction was essentially through synergism, unlike the case of Pu(IV) and U(VI) (where the tetra- and hexavalent actinides may be extracted as simple chelates to a considerable extent), the fall in D_{Am} was significant.

Extraction Equilibria

The extraction of trivalent americium ion, Am^{3+}, by PBI can be expressed as

$$\mathrm{Am}^{3+}_{(\mathrm{aq})} + 3\mathrm{PBI}_{(\mathrm{org})} \overset{K_{\mathrm{ex}}}{\mathrm{C\ E}} \mathrm{Am(PBI^{*})}_{3(\mathrm{org})} + 3\mathrm{H}^{+}_{(\mathrm{aq})}$$

where PBI* denotes isoxazolonate ion (deprotonated PBI). The value of log K_{ex} had been evaluated as 0.04 ± 0.02 by Reddy et al. (17). However, if TOPO is also present in the above system, the extraction mechanism would include the following equilibrium:

$$\mathrm{Am}^{3+}_{(\mathrm{aq})} + 3\mathrm{PBI}_{(\mathrm{org})} + n\mathrm{TOPO}_{(\mathrm{org})} \overset{K_{\mathrm{syn}}}{\mathrm{C\ E}} \mathrm{Am(PBI^{*})}_{3}\cdot n\mathrm{TOPO}_{(\mathrm{org})} + 3\mathrm{H}^{+}_{(\mathrm{aq})}$$

where n is a small number like 1 and/or 2. Using TBP and bis-2-ethylhexyl sulfoxide (B2EHSO) in place of TOPO, Reddy et al. (17) showed the value of n to be 2 for both the oxodonors. The log K_{syn} values have been reported to be >8, indicating the substantial synergistic enhancement of Am extraction. Since the present work aims at studying the extraction behavior of PBI–TOPO mixture toward Am(III) from $\mathrm{HNO_3}$ medium, no attempt was made to evaluate the values of the equilibrium constants.

Stripping Behavior

Even though the above extraction data on Am(III) showed that it could be stripped with 6 M $\mathrm{HNO_3}$, a systematic study to ascertain the stripping behavior of Am(III), Pu(IV) and U(VI) by various commonly used strippants was carried out. The results are shown in Table 5. 10 M $\mathrm{HNO_3}$ could be used to strip Am(III) selectively. For U(VI) and Pu(IV), $\mathrm{Na_2CO_3}$ appears to be a satisfactory strippant.

Am Extraction from Synthetic HLW Solution

Composition of HLW solutions varies with the nature of the fuel used, burn up, and cooling period. Loading of the organic phase is principally influenced by the presence of U and rare earths in the waste solution. When present along with Am, U was expected to be extracted preferentially and rare earths competitively with it. Hence the extraction profile of Am in the presence of U (in the range of 1 to 20 g/L) and a typical representative of rare-earth elements present, Nd (in the range of 0.5 to 10 g/L), were obtained at various aqueous nitric acid concentrations (Figs. 5 and 6). The extraction

TABLE 5
Stripping Behavior of Am, Pu, and U ([PBI] = 0.05 M; [TOPO] = 0.2 M; diluent: carbon tetrachloride)

Strippant	D_{Am}	D_{Pu}	D_U
0.05 M $NH_2OH \cdot HCl$ in 0.35 M HNO_3	~6000	3721	1052
0.5 M Na_2CO_3	543	0.222	0.008
0.3 M Ascorbic acid	~4300	216.3	207.3
0.1 M Hydroquinone in 6 M HCl	0.25	5.08	815
0.1 M Oxalic acid + 0.1 M ammonium oxalate	1000	7	1065
10 M HNO_3	0.003	40	79.5

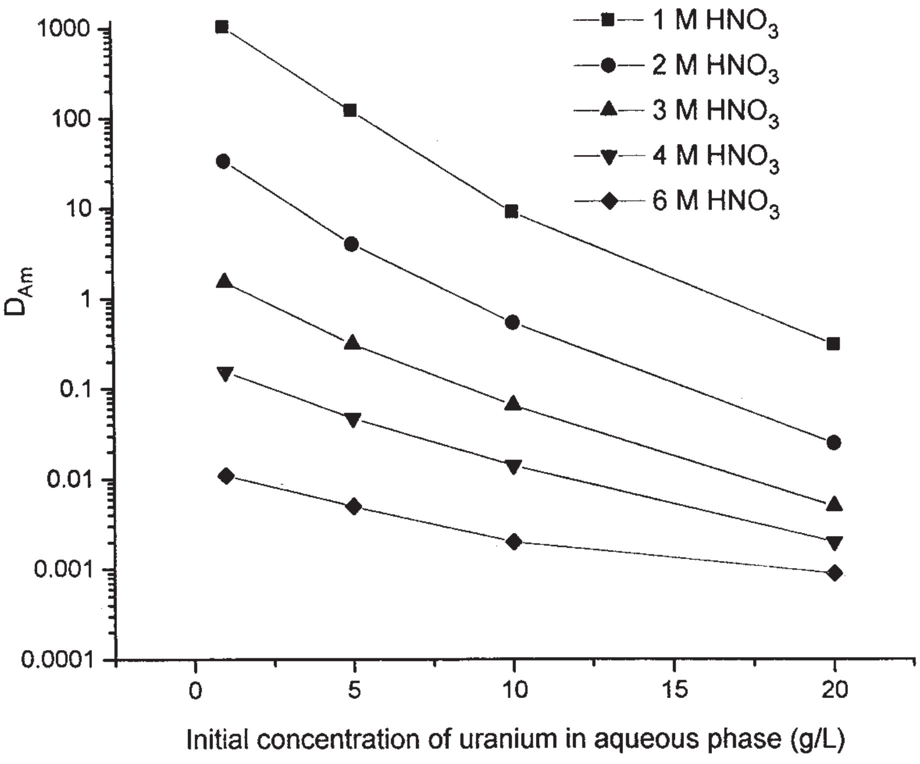


FIG. 5 Effect of uranium loading on Am(III) extraction by 0.05 M PBI + 0.2 M TOPO in CCl_4.

of Am(III) decreased with an increase in acidity as well as with an increase in U(VI) or Nd(III) concentration. At 1 M HNO_3, quantitative extraction of Am was achieved when U(VI) was present up to 5 g/L. At 10 and 20 g/L of U, the Am extraction dropped to 90 and 22%, respectively. On the other hand, at 2 M HNO_3, even with 1 g/L of U, the extraction of Am decreased to 97% while the presence of U at 5 and 10 g/L further brought down the extraction to 85 and 35%, respectively. Thus, for quantitative extraction of Am in the presence of U, not only did the acidity need to be lowered to 1 M but also the U content should be brought down to the $\leq$5 g/L level. The latter could be achieved by preequilibrating the waste solution mixture with ~30% TBP in CCl_4. Similarly in the case of Nd loading, near quantitative extraction was achieved only with 1 M HNO_3 up to about 2 g/L of Nd; at 2 M HNO_3, Nd at the 2 g/L level decreased the extraction to ~90%.

In view of the possibility of extraction of Am in acid medium along with actinides and its selective stripping, it was worth considering the scope of utilizing this extractant for removal of Am from HLW solution after adjusting the acidity to 1 M. In view of the observation that the present reagent is

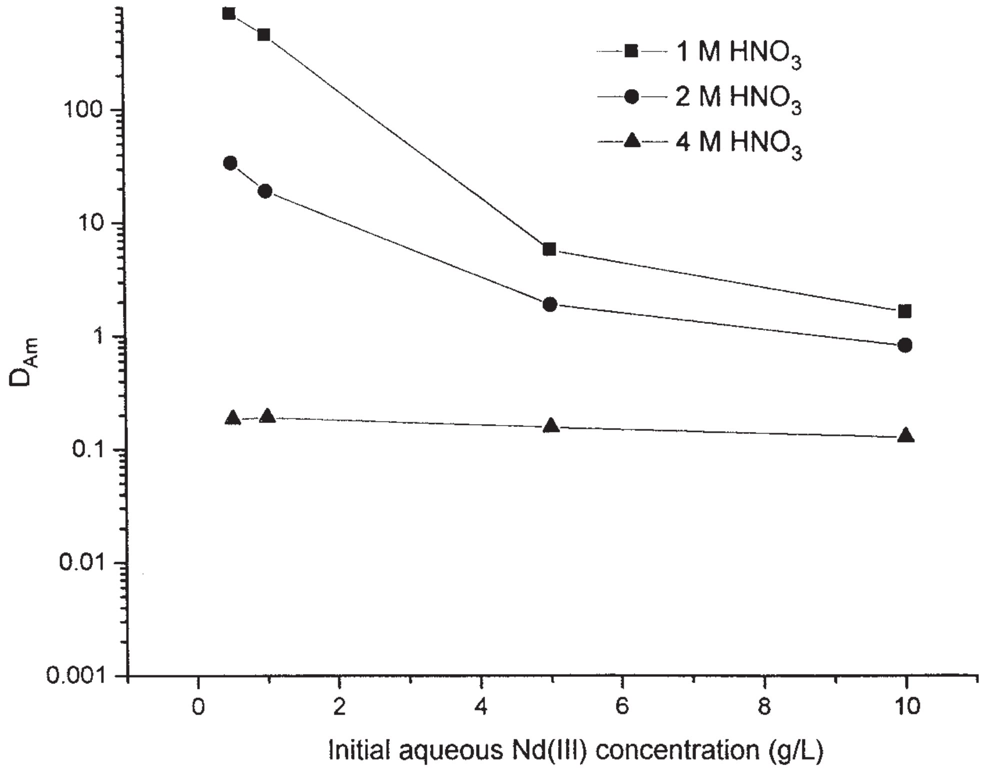


FIG. 6 Effect of Nd(III) loading on Am(III) extraction by 0.05 M PBI + 0.2 M TOPO in CCl_4.

TABLE 6
Composition of the Synthetic Waste Solution (in g/L) in 1 M HNO_3

Salt	Amount	Salt	Amount
$Fe(NO_3)_3 \cdot 9H_2O$	3.25	CrO_3	0.2
$NiCl_2 \cdot 6H_2O$	0.7	$NaNO_3$	1.9
Rb_2CO_3	0.07	$Sr(NO_3)_2$	0.22
$ZrOCl_2$	1.6	$CsNO_3$	0.34
$Na_2MoO_4 \cdot 2H_2O$	1.17	$Co(NO_3)_2 \cdot 6H_2O$	0.14
$Ba(NO_3)_2$	0.39	$Ce(SO_4)_2$	0.87
$SmCl_3$	0.09	$EuCl_3$	0.05
UO_3	1.28		

capable of extracting Am quantitatively only at relatively lower acidities, a synthetic waste solution containing various metal ions corresponding to HLW composition (Table 6) was prepared in 1 M HNO_3. ^{241}Am was spiked to the synthetic HLW, and by using the 0.05 M PBI + 0.2 M TOPO mixture, D_{Am} was found to be 31.2 ± 0.3. Thus, the PBI–TOPO mixture could remove all the actinides along with Am(III) in just two contacts if the acidity of the HLW solution was adjusted to 1 M. Am(III) in the organic phase could also be easily separated from other actinides by selectively stripping it using high acid (6–10 M HNO_3). There was no third-phase formation.

Behavior of Fission Products

The extraction of Cs^+ and Sr^{2+} by the PBI + TOPO mixture was investigated using ^{137}Cs and $^{85,89}Sr$ as tracers, respectively. In the entire range of 1–6 M HNO_3, no extraction of Cs or Sr was observed ($D < 10^{-3}$). Since the HLW is associated with a host of other fission products as well, their distribution behavior (collected on an aluminum catcher foil from a ^{252}Cf source, dissolved in 1 M HCl, evaporated to dryness, and adjusted to 1 M HNO_3) was evaluated in the two-phase system: 1 M HNO_3/0.05 M PBI + 0.2 M TOPO/CCl_4. The distribution ratio of different fission products obtained by measuring their gamma activity is shown in Table 7. It can be seen from the data that rare-earth elements (namely Ce, Nd, Sm, and La) are extracted quantitatively and Zr, Cd, and In are extracted up to 80–85%, while the extraction of Ru, I, Pd, and Te is marginal (<6%). Ba and Ag are not extracted at all.

CONCLUSIONS

PBI is the only β-diketone capable of quantitatively extracting Am(III) from 1 M HNO_3 when used along with TOPO in carbon tetrachloride. If U(VI)

TABLE 7
Distribution of Fission Products in the Two-Phase System
(1 M HNO_3–0.05 M PBI + 0.2 M TOPO/carbon tetrachloride)[a]

Fission product	$D_{f.p.}$	Fission product	$D_{f.p.}$
^{147}Nd	>300	^{112}Pd	0.045
^{153}Sm	500	^{97}Nb	21.7
^{141}Ce	>400	^{97}Zr	11.5
^{117}In	4.88	^{96}Nb	14.5
^{113}As	N.E.	^{132}Te	0.0095
^{115}Cd	13.5	^{140}La	>130
^{103}Ru	0.061	^{133}I	0.025
^{140}Ba	N.E.		

[a] N.E. denotes ‘‘no extraction.’’

and Pu(IV) are present in the aqueous solution, they will also be extracted quantitatively. From the organic extract containing all three actinides, Am(III) can be selectively stripped using 6 M HNO_3. Rare earths are expected to accompany Am(III). Quantitative Am(III) extraction can be achieved from a solution containing up to 5 g/L of U(VI) or 2 g/L of Nd(III).

ACKNOWLEDGMENTS

The authors are grateful to Dr. S. B. Manohar, Head, Radiochemistry Division, for his keen interest in this work. They are also thankful to Dr. Haldhar Naik for his help in γ-spectrometry work and Mr. P. N. Pathak for his help during the preparation of the manuscript.

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Received by editor September 17, 1997
Revision received February 1998

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