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Synergistic Extraction of Trivalent Actinides by Mixtures of Thenoyltrifluoroacetone and Neutral Oxo Donors

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Abstract

The synergistic extraction of trivalent actinides Am, Cm, Bk, and Cf has been studied by mixtures of HTTA and TOPO as well as DOSO in xylene at 30°C. HTTA-S (TOPO, DOSO) interaction corrections have been applied to calculate the "free" S concentrations in the organic phase. In the extraction of trivalent actinides, the third-power dependence on $[\mathrm{HTTA}]_{\mathrm{org}}$ at a fixed $[\mathrm{S}]_{\mathrm{org}}$ has been observed only after applying this correction. The synergistic species $\mathrm{M(TTA)_3.S}$ and $\mathrm{M(TTA)_3.2S}$ were found to be extracted into the organic phase whose stability constants (β_1, β_2, and K_2) have been evaluated. Extraction by HTTA + S (S = TOPO, DOSO, TBP, TBTP) shows the order of extraction to be Tm > Cf > Bk > Eu > Pm > Am > Cm for the trivalent ions. The Am/Cm separation factor with the synergistic mixtures is ~3 whereas with HTTA alone it is ~6 when they are extracted from the chloroacetate buffer.

Synergistic extraction of metal ions by mixtures of chelating agents and neutral donors has been the subject of great interest, primarily because of the large enhancement of extraction obtained using various combinations of the extracting agents. Although a large number of papers (*1–7*) has appeared on the synergistic extraction of the trivalent lanthanides by mixtures of thenoyltrifluoroacetone (HTTA) and neutral donors, relatively little work (*1, 3, 7, 8*) has been carried out on the extraction of trivalent actinides by such mixtures. In our earlier communication (*9*), synergistic extraction of the trivalent actinides Am, Cm, Bk, and Cf by a mixture of HTTA and tri-*n*-butyl phosphate (TBP) in xylene was reported. In the present work, synergistic extraction of the trivalent actinides by mixtures of HTTA with di-*n*-octyl sulfoxide (DOSO) and tri-*n*-octyl phosphine oxide (TOPO) in xylene is studied. In addition, extraction of some trivalent actinides and lanthanides by synergistic mixtures of HTTA and several neutral donors was studied with a view to explore the possibility of separation of the trivalent actinides and lanthanides.

EXPERIMENTAL

Materials

Tracer solutions of ^{241}Am, ^{244}Cm, ^{249}Bk, Cf (mainly ^{252}Cf), ^{147}Pm, ^{152,154}Eu, and ^{170}Tm were prepared as described previously (*8*, *9*). HTTA was Merck's Pro analysis. TBP and tri-*n*-butyl thiophosphate (TBTP) were purified by standard methods, while TOPO (mp 53.5°C) was used as such. DOSO was kindly supplied in a purified state by Dr M. S. Subramanian of this laboratory. Chloroacetic acid (BDH AnalaR) was used for preparing the buffer solution ($\mu = 0.005$, pH 2.70) and was used as the aqueous phase throughout this work.

Radioassay of the Tracers

The trivalent actinides ^{241}Am, ^{244}Cm, ^{249}Bk, and Cf were assayed by liquid scintillation counting of their α or soft β (^{249}Bk) particles as described earlier (*9*). Of the lanthanides ^{147}Pm was assayed by liquid scintillation counting of its β particles while ^{152,154}Eu and ^{170}Tm were assayed by γ-scintillation counting using a well-type NaI (Tl) counter.

HTTA Variation Experiments

One milliliter of the aqueous phases spiked with the tracers of the actinides were equilibrated with an equal volume of xylene solutions of varying HTTA concentrations alone as well as with a fixed concentration of either DOSO or TOPO, respectively. These and subsequent equilibrations were done for 1 h by slow rotation in a thermostatted bath maintained at 30 ± 0.1°C. After settling for ½ h, aliquots of both phases were removed for radioassay. The distribution coefficient values (D) are not affected (*6*, *9*) in the presence of the low concentrations of chloroacetate buffer used, and no correction is necessary for the uptake of chloroacetic acid by the basic oxo donors.

Synergistic Extraction of the Trivalent Actinides and Lanthanides

Equilibrations were carried out with the trivalent actinides as described above, the organic phases containing HTTA at a fixed concentration (0.064

M for Am, Cm, and Cf and 0.024 M for Bk) and varying concentrations of DOSO (8×10^{-4} to 5×10^{-3} M) and TOPO (5×10^{-5} to 3×10^{-4} M). Extraction of the trivalent actinides and the lanthanides Pm(III), Eu(III), and Tm(III) was also studied with mixtures of HTTA (0.1 M) and the synergists TBP, DOSO, and TBTP (0.002 M) and TOPO (0.001 M).

Calculation of the Equilibrium Constants

The equilibrium constants (β_1, β_2, and K_2) for the organic phase addition reactions of $M(TTA)_3$ with S, where S is the synergist, may be represented by.

$$M(TTA)_3 + S \overset{\beta_1}{\rightleftharpoons} M(TTA)_3.S \quad (1)$$

$$M(TTA)_3 + 2S \overset{\beta_2}{\rightleftharpoons} M(TTA)_3.2S \quad (2)$$

$$M(TTA)_3.S + S \overset{K_2}{\rightleftharpoons} M(TTA)_3.2S \quad (3)$$

As described in detail in earlier work (*6, 9*), β_1, β_2, and K_2 may be calculated using the equation

$$\frac{D - D_{TTA}}{[S]_{org}} = K'_{syn_1} \frac{[HTTA]^3_{org}}{[H^+]^3_{aq}} + K'_{syn_2} \frac{[HTTA]^3_{org}}{[H^+]^3_{aq}} [S]_{org} \quad (4)$$

where D and D_{TTA} are the distribution coefficients in the presence and in the absence of the synergist S, respectively, and k'_{syn_1} and K'_{syn_2} are the two phase equilibrium constants for the extraction of the trivalent actinides by the synergistic mixture. As seen from Figs. 4 and 5, the plots of $(D - D_{TTA})/[S]_{org}$ vs $[S]_{org}$ for all the trivalent actinides gave straight lines, indicating the presence of $M(TTA)_3.S$ and $M(TTA)_3.2S$ as the only synergistic species in the organic phase. The presence of the $M(TTA)_3$ moiety in the synergistically extracted species is confirmed from the straight line plots with slopes equal to 3 obtained after applying the HTTA–neutral donor interaction correction as described in detail later. The intercepts (c) and the slopes (m) of the straight lines in Figs. 4 and 5 are related to k'_{syn_1} and K'_{syn_2} by the relationships

$$\text{Intercept } c = k'_{syn_1} \frac{[HTTA]^3_{org}}{[H^+]^3_{aq}}$$

$$\text{Slope } m = K'_{\text{syn}_2} \frac{[\text{HTTA}]^3_{\text{orb}}}{[\text{H}^+]^3_{\text{aq}}}$$

Values of k'_{syn_1} and K'_{syn_2} were calculated from the known values of $[\text{HTTA}]_{\text{org}}$ and $[\text{H}^+]_{\text{aq}}$. β_1 and β_2 were calculated from the relations

$$\beta_1 = k'_{\text{syn}_1}/K'_{\text{TTA}} \qquad (5)$$

$$\beta_2 = K'_{\text{syn}_2}/K'_{\text{TTA}} \qquad (6)$$

where K'_{TTA} is related to the equilibrium constant K_{TTA} of the reaction

$$\text{M}^{3+}_{\text{aq}} + 3\text{HTTA}_{\text{org}} \overset{K_{\text{TTA}}}{\rightleftharpoons} \text{M(TTA)}_{3_{\text{org}}} + 3\text{H}^+_{\text{aq}} \qquad (7)$$

by the relationship $K'_{\text{TTA}} = K_{\text{TTA}} \cdot X^{-1}$ (*9*). Values of K_2 were calculated from the relationship $K_2 = \beta_2/\beta_1$. The equilibrium constants so evaluated refer only to concentration quotients whose calculations are based on the assumption that the activity coefficients of the species involved do not change significantly under the experimental conditions employed.

RESULTS AND DISCUSSION

Plots of log D vs log $[\text{HTTA}]_{\text{org}}$ for the extraction of the trivalent actinides by HTTA are presented in Fig. 1. Straight line plots with slopes equal to 3 obtained for Am, Bk, and Cf indicate the extraction of M(TTA)_3 (M = Am, Bk, Cf) into the organic phase. In the case of Cm(III) the slope of 2 obtained at lower HTTA concentrations (0.024–0.14 *M*) indicates in addition the extraction of $\text{M(TTA)}_2.\text{Y}$ where Y is presumably the chloroacetate anion. The extraction behavior of Cm(III) shows that in the presence of chloroacetate it is complexed to a lesser extent by HTTA in comparison with other actinides studied, which is also reflected in the lower K'_{TTA} value for Cm(III) given in Table 3.

Figure 2 presents plots of log D vs log $[\text{HTTA}]_{\text{org}}$ for the trivalent actinides obtained using a fixed TOPO concentration and varying concentrations of HTTA in the organic phase. The experimental points of log D lie on curves (dotted lines) whose slopes vary from 3 to 1 as the HTTA concentration increases. Irving and Edgington (*10*) also found a trend of decreasing slope (from 3 to 1) with increasing HTTA concentrations at a fixed TBP

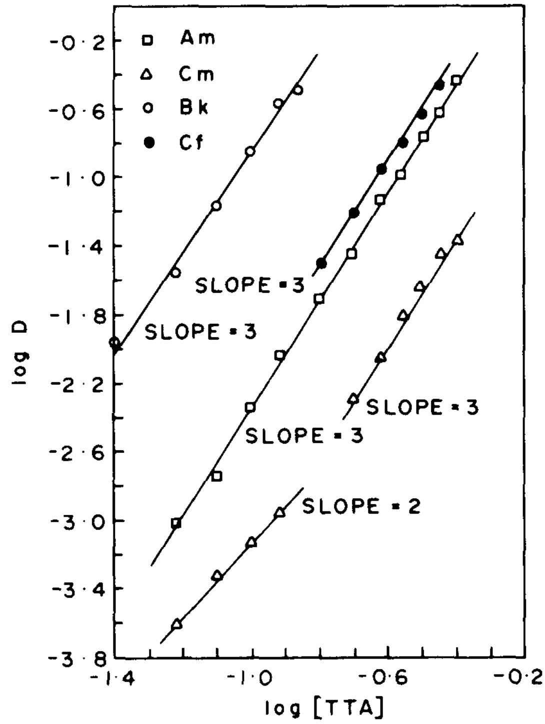

FIG. 1. Variation of distribution coefficient of trivalent actinides as a function of HTTA concentration.

concentration in their study of the Th(IV)/HTTA/TBP system which they have ascribed to the weak complexes formed, as also in the HTTA-TBP interaction. Similar extraction behavior was encountered in the M(III)/HTTA/TBP (M = Am, Cm, Bk, Cf) studied by us earlier (*9*) where the departure of slope from the expected value of 3 was shown to arise due to the decreasing TBP activity arising from the HTTA-TBP interaction as the HTTA concentration increases.

The organic phase interaction of HTTA and S can be represented by

$$\mathrm{HTTA} + n\mathrm{S} \rightleftharpoons \mathrm{HTTA}.n\mathrm{S} \tag{8}$$

the equilibrium constant K_{HT} of which is given by

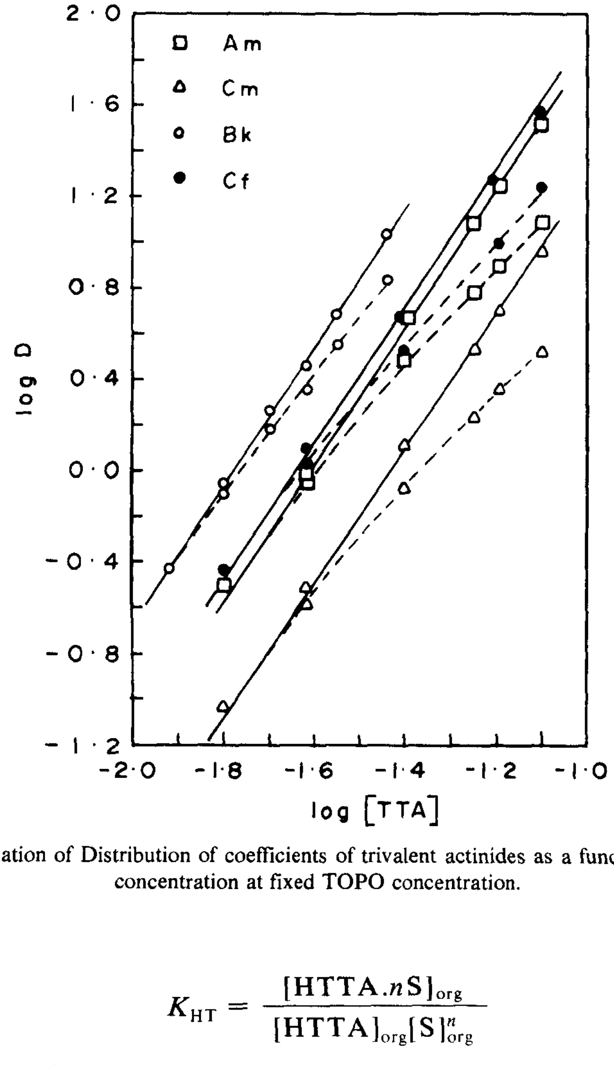

FIG. 2. Variation of Distribution of coefficients of trivalent actinides as a function of HTTA concentration at fixed TOPO concentration.

$$K_{\mathrm{HT}} = \frac{[\mathrm{HTTA}.n\mathrm{S}]_{\mathrm{org}}}{[\mathrm{HTTA}]_{\mathrm{org}}[\mathrm{S}]^{n}_{\mathrm{org}}} \qquad (9)$$

It has been found from an absorption spectrophotometric study of the distribution of HTTA in the presence of various synergists carried out in this laboratory (*11*) that with many synergists, including DOSO and TOPO, the value of n is unity. The values of K_{HT} obtained in this work are given in Table 1. Values of log D (Fig. 2) for the trivalent actinides are obtained with a fixed $[\mathrm{TOPO}]_{\mathrm{org}}$ and varying concentrations of HTTA. However, due to HTTA–TOPO interaction, the "free" $[\mathrm{TOPO}]_{\mathrm{org}}$ decreases progressively as the HTTA concentration increases, and log D is consequently reduced substantially. Correction for this can, however, be made by normalizing log D

TABLE 1

Equilibrium Constants of the Adducts Formed between HTTA and Neutral Oxo Donors in Xylene[a]

Oxo donor	K_{HT}	Log K_{HT}
TBP	2.6	0.41
DOSO	3.7	0.57
DBBP	8.2	0.91
TOPO	14.0	1.15

[a] Initial $[HTTA]_{org} = 0.02$ *M*, $[H^+] = 0.13$ *N*.

values for a constant "free" $[TOPO]_{org}$ using the K_{HT} value for TOPO (Table 1). The normalized log *D* values (Fig. 2) are derived from the plots of log *D* vs "free" $[TOPO]_{org}$ at a fixed manyfold higher HTTA concentration—which is not materially affected by the HTTA–TOPO interaction—given in Fig. 3. From the straight line plots of slopes 1.8 to 1.3 obtained for the trivalent actinides, empirical equations are derived for the normalization of the log *D* values given in Fig. 2. Thus for Am(III) the empirical equation

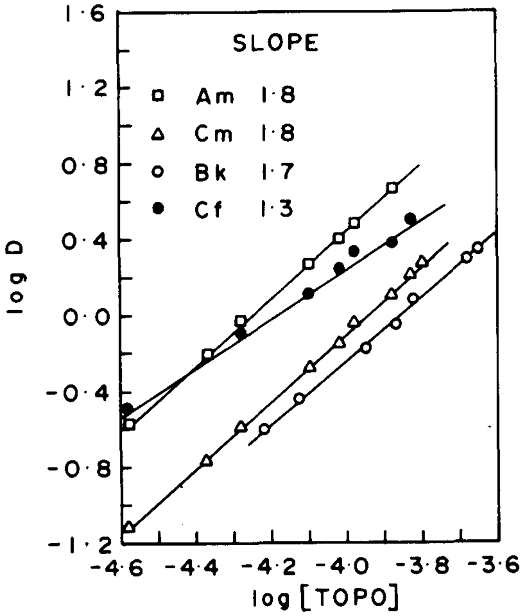

FIG. 3. Variation of distribution coefficient of trivalent actinides as a function of TOPO concentration at fixed HTTA (0.064 *M* for Am, Cm, Cf and 0.024 *M* for Bk) concentration.

TABLE 2

Distribution Coefficients of Am/TTA/TOPO System at Fixed TOPO Concentration and Varying HTTA Concentrations[a]

[HTTA] (M)	After correction [TOPO] ($M \times 10^{-4}$)	Log D experimental value	Log D^* corrected[b]	$\Delta \log D = \log D^* - \log D$
0.016	2.454	−0.512	−0.512	–
0.024	2.249	−0.047	−0.021	0.026
0.040	1.927	0.478	0.667	0.189
0.056	1.686	0.787	1.081	0.294
0.064	1.586	0.898	1.239	0.341
0.080	1.419	1.087	1.515	0.428

[a] Initial [TOPO] = 3.0×10^{-4} M.

[b] Log D values normalized for [TOPO] = 2.454×10^{-4} M.

$$\log D_1 - 1.8 \log [\text{TOPO}]_1 = \log D_2 - 1.8 \log [\text{TOPO}]_2$$

is derived, from which the normalized values of log D for Am(III) are calculated. These are presented in Table 2. The straight lines with slopes of 3 (unbroken lines) in Fig. 2 are obtained using the normalized log D values. Similar results are also obtained when DOSO is the synergist, showing that the moiety $M(TTA)_3$ is present in all the synergistic systems studied in this work.

Figures 4 and 5 show the plots of $(D - D_{TTA})/[\text{DOSO}]_{org}$ vs $[\text{DOSO}]_{org}$ and similar plots when TOPO is used as the synergist, respectively, the synergist concentration referring to the "free" [S]. The straight line plots indicate the extraction of $M(TTA)_3.S$ and $M(TTA)_3 2S$ into the organic phase. The values of K'_{syn_1} and K'_{syn_2} are evaluated from these plots and are presented in Table 3 along with the K'_{TTA} values. Stability constants of the organic phase addition reactions (β_1, β_2, and K_2) of the metal chelates with the synergists are presented in Table 4. Newman (*7*) described the formation of only one synergistic species $M(TTA)_3.2TOPO$ in benzene with log β_2 equal to 10.0 (Am) and 9.5 (Cm) while Healy (*12*) gives the values of 12.0 (Am) and 12.5 (Cm) for log β_2 for the same system. In the present work where two synergistic species are reported for this system, log β_2 values of 11.12 (Am) and 11.56 (Cm) are obtained which are intermediate between the values obtained by the other two workers. Recently Akiba et al. (*13*) and Reddy et al. (*14*) have studied the Am(III)/HTTA/TOPO and the Am(III)/HTTA/DPSO systems, respectively, in both of which two synergistic species are reported.

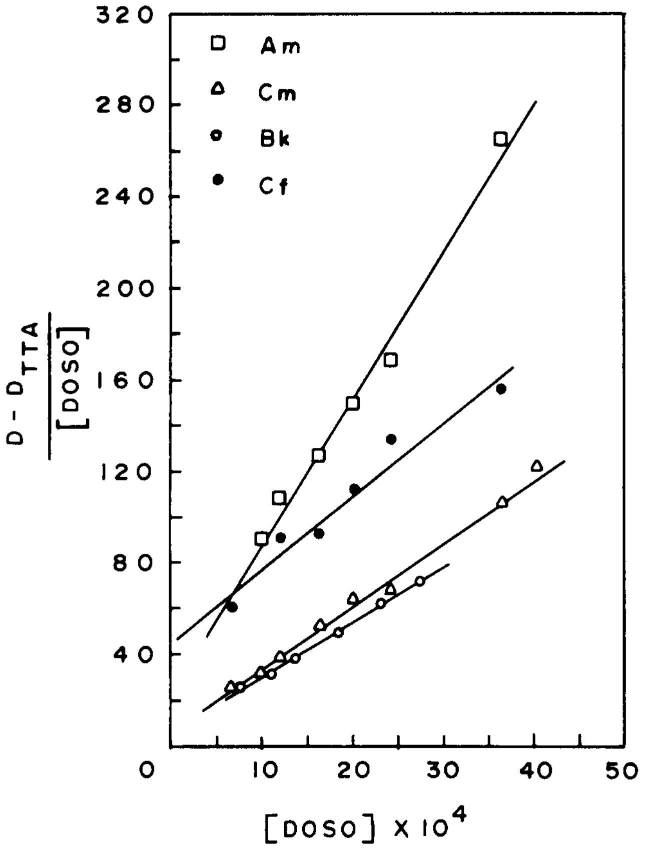

FIG. 4. Variation of $(D - D_{TTA})/[DOSO]_{org}$ vs $[DOSO]_{org}$ for the trivalent actinides.

The values of β_1, β_2, and K_2 obtained in the present work do not show the decreasing trend with increasing atomic number of the trivalent actinides observed in some other synergistic systems both for the trivalent actinides and the lanthanides (*14–16*). In our earlier work with the M(III)/HTTA/TBP system (*9*) β_1 etc. did not vary monotonically with the atomic number of the trivalent actinides. The values of the adduct stabilities, however, do show a decreasing trend with increasing stabilities of the metal chelates (Tables 3 and 4) as was also observed in several synergistic systems by other workers (*17*). The values of K'_{TTA} (Table 3) do not show a monotonic increase with increasing atomic numbers warranted by the decreasing ionic radii of the trivalent actinides. Thus K'_{TTA} for Cm(III) is actually appreciably smaller

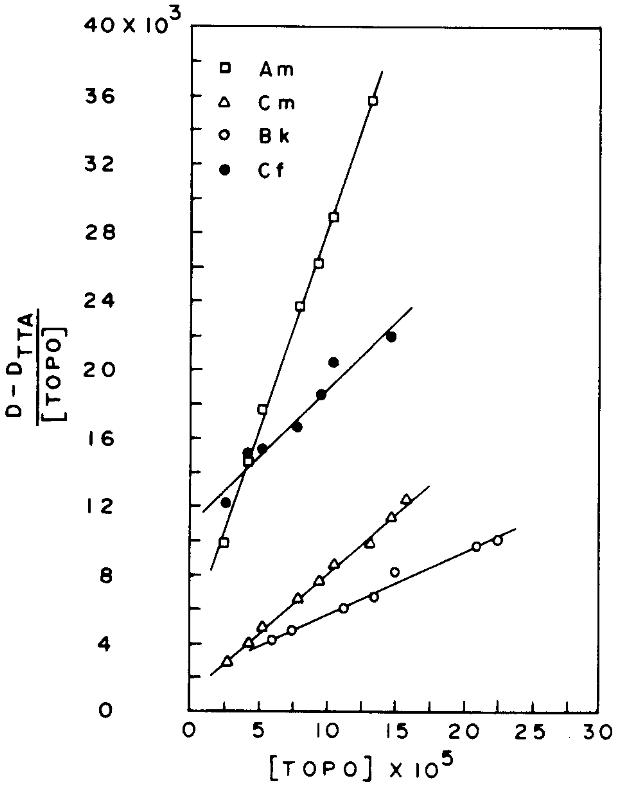

FIG. 5. Variation of $(D - D_{TTA})/[TOPO]_{org}$ vs $[TOPO]_{org}$ for the trivalent actinides.

TABLE 3

Two-Phase Equilibrium Constants of Trivalent Actinides in HTTA–Neutral Oxo Donors–Xylene Systems at 30°C

M(III)	Log K'_{syn_1}	Log K'_{syn_2}	Log K'_{TTA}
	HTTA + DOSO		
Am	−3.18 ± 0.07	0.29 ± 0.01	−7.27 ± 0.05
Cm	−3.78 ± 0.11	−0.07 ± 0.01	−8.22 ± 0.05
Bk	−2.51 ± 0.07	1.14 ± 0.01	−6.12 ± 0.10
Cf	−2.86 ± 0.05	−0.01 ± 0.02	−7.29 ± 0.03
	HTTA + TOPO		
Am	−0.87 ± 0.03	3.85 ± 0.01	
Cm	−1.49 ± 0.05	3.32 ± 0.01	
Bk	+0.09 ± 0.04	4.31 ± 0.02	
Cf	−0.48 ± 0.06	3.37 ± 0.03	

TABLE 4

Equilibrium Constants for the Organic Phase Synergistic Reaction of Trivalent Actinides–HTTA Chelate with Neutral Oxo Donors

M(III)	Log β_1	Log β_2	Log K_2
	HTTA + DOSO		
Am	4.09 ± 0.08	7.56 ± 0.05	3.47 ± 0.07
Cm	4.46 ± 0.11	8.16 ± 0.04	3.70 ± 0.11
Bk	3.61 ± 0.10	7.26 ± 0.09	3.65 ± 0.06
Cf	4.43 ± 0.05	7.27 ± 0.04	2.84 ± 0.06
	HTTA + TOPO		
Am	6.40 ± 0.06	11.12 ± 0.05	4.72 ± 0.05
Cm	6.75 ± 0.07	11.56 ± 0.05	4.81 ± 0.05
Bk	6.20 ± 0.11	10.43 ± 0.10	4.23 ± 0.05
Cf	6.81 ± 0.03	10.66 ± 0.04	3.85 ± 0.03
Eu	5.56 ± 0.02	8.60 ± 0.03	3.03 ± 0.04

TABLE 5

Equilibrium Constants of the Organic Phase Synergistic Reaction of Am(III)–HTTA with TBP, DOSO, and TOPO at 30°C

Neutral donor	Log β_1	Log β_2	Log K_2
TBP	4.19 ± 0.06	7.45 ± 0.05	3.26 ± 0.08
DOSO	4.09 ± 0.08	7.56 ± 0.05	3.47 ± 0.07
TOPO	6.40 ± 0.06	11.12 ± 0.05	4.72 ± 0.05

than that for Am(III). A nonmonotonic variation of K'_{TTA} values for the trivalent lanthanides has also been observed where the K'_{TTA} values for Eu(III) and Yb(III) are found to be higher than the neighboring lanthanides of a higher atomic numbers, which has been ascribed to their favorable redox potentials (*8*). Although the reasons for the comparatively higher K'_{TTA} values of Am(III) and Bk(III) cannot be unequivocally ascribed to any known causes, they are definitely reflected in the comparatively lower values [especially for Bk(III)] of their adduct stability constants. Table 5 gives the values of β_1, β_2, and K_2 for the Am(III)/HTTA/S system (S = TBP, TOPO, DOSO). TBP and DOSO adducts are similar in stability whereas those with TOPO are stabler by several orders of magnitude. This follows the order of the basicities of the neutral oxodonors.

Table 6 gives the distribution coefficients for the extraction of some trivalent actinides and lanthanides by mixtures of HTTA with the neutral

TABLE 6

Distribution Coefficients of Trivalent Actinides and Lanthanides Extracted by Mixtures of HTTA + Neutral Oxo Donors at 30°C[a]

	K_D			
M(III)	TBP (0.002 *M*)	TOPO (0.001 *M*)	DOSO (0.002 *M*)	TBTP (0.002 *M*)
Am	0.800	115.86	0.845	0.048
Cm	0.283	65.64	0.271	0.019
Bk	2.31	206.13	2.92	0.320
Cf	4.26	229.97	4.54	0.420
Pm	1.080	214.72	0.780	0.058
Eu	1.901	225.0	1.438	0.099
Tm	5.748	403.1	4.551	0.153

[a] [HTTA] = 0.1 *M*, pH = 2.7.

TABLE 7

Separation Factor of Am(III) from Cm(III) by Various Extracting Agents

Extracting agent	Aqueous phase	Diluent	Separation factor K_D Am/K_D Cm
HTTA + TBP	*pH* 2.7, buffer Cl acetate ($\mu = 0.005$)	Xylene	2.8
HTTA + TOPO	*pH* 2.7, buffer Cl acetate ($\mu = 0.005$)	Xylene	1.8
HTTA + DOSO	*pH* 2.7, buffer Cl acetate ($\mu = 0.005$)	Xylene	3.1
HTTA + TBTP	*pH* 2.7, buffer Cl acetate ($\mu = 0.005$)	Xylene	2.5
HDEHP[a]	*pH* 2.7, buffer Cl acetate ($\mu = 0.005$)	Xylene	0.82
NH_4DNNS[b]	*pH* 2.7, buffer Cl acetate + 1.0 *M* NaCl	Hexane	0.88
HTTA	*pH* 2.7, buffer Cl acetate ($\mu = 0.005$)	Xylene	6

[a] Di-2-ethylhexyl phosphoric acid.

[b] Ammonium salt of dinonylnaphthalene sulfonic acid.

donors TBP, TOPO, DOSO, and TBTP. It is observed that none of these mixtures could be used for a one-step separation of travalent actinides from trivalent lanthanides. The order of extraction (K_D) is Tm > Cf > Bk > Eu > Pm > Am > Cm. Davis et al. (*18*) have used a synergistic mixture of HTTA and TBP in kerosene for the partial separation of trivalent actinides and lanthanides. From the extraction order given above it seems likely that these synergistic systems could also be used for the separation of Am(III) and Cm(III) from the lanthanides with atomic numbers lower than that of Eu. Table 7 gives the values of the separation of factors (S.F) of Am(III) from Cm(III) by various extracting agents. The synergistic mixtures of HTTA with TBP and DOSO give S.F. ~ 3. The S.F. ~ 6 obtained when HTTA alone is used as an extractant from chloroacetate buffer looks promising for the separation of Am(III) and Cm(III). Extraction chromotographic work is in progress for attempting this separation.

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