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Synergistic Extraction of Pu(IV) by Mixtures of HTTA with TBP and DBBP

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Abstract

The synergistic extraction of Pu(IV) from perchloric acid medium by mixtures of thenoyltrifluoroacetone (HTTA) and the neutral donors tri-*n*-butylphosphate (TBP) and dibutyl butylphosphonate (DBBP) using benzene and cyclohexane as diluents was investigated by different solvent extraction methods. With TBP, the adduct responsible for synergism was found to be $Pu(TTA)_4 \cdot TBP$. With DBBP, in addition to $Pu(TTA)_4 \cdot DBBP$, it was indicated that more species containing perchlorate ion are involved in the extraction. The adduct formation of $Pu(TTA)_4$ with DBBP in benzene was also investigated by spectrophotometry. The equilibrium constants for several equilibria involved were obtained.

INTRODUCTION

The data on synergistic extraction of tetravalent actinides are limited (*1–4*). We have initiated a program on the synergistic extraction of tetravalent actinides from different aqueous media by mixtures of HTTA and different neutral donors. The results obtained for the extraction of Th(IV) (*5*), U(IV) (*6–8*), Np(IV) (*9*, *10*), and Pu(IV) (*11*) were reported earlier. In continuation, the extraction of Pu(IV) from aqueous perchloric acid by mixtures of thenoyltrifluoroacetone (HTTA) with tri-*n*-butylphosphate (TBP) and dibutyl butylphosphonate (DBBP) using benzene and cyclohexane as the diluents was investigated and the results are reported here. An attempt was also made to substantiate the solvent extraction results by absorption spectral studies.

EXPERIMENTAL

Materials

The make and purity of most of the materials used were described earlier (*11*). DBBP, obtained from K & K Labs, USA, was purified by the same method as that used for TBP (*12*, *13*). Laboratory reagent-grade cyclohexane, obtained from B.D.H., England, was used as such. The cyclohexane solution of HTTA was pre-equilibrated for about 3 hr with dilute perchloric acid.

Procedure

Plutonium in nitric acid medium was adjusted to Pu(IV) by adding a few drops of hydrogen peroxide, the excess of which was destroyed by heating. The Pu(IV) thus formed was extracted into 0.5 *M* HTTA in benzene and the extracted Pu(IV) was stripped back with 8 *M* nitric acid. The solution of Pu(IV) in 8 *M* nitric acid was scrubbed with benzene to remove the dissolved HTTA. This procedure, besides ensuring the oxidation state of Pu(IV), removed any ^{241}Am present in the initial plutonium solution formed by decay of ^{241}Pu. A small aliquot of this stock solution of Pu(IV) was added to ~100 to 150 ml of 1 *M* $HClO_4$ containing $\sim 10^{-4}$ *M* vanadium(V) as the holding oxidant for Pu(IV). The nitrate ion concentration in this stock was $\sim 5 \times 10^{-4}$ *M*, the influence of which is considered negligible. Equal volumes (3.0 mL each) of Pu(IV) in the aqueous stock were thus made, and the organic phase (benzene or cyclohexane) containing mixtures of HTTA and TBP or DBBP of the desired composition were equilibrated for 1 hr in a thermostated water bath kept at 25 ± 0.1 °C. The equilibrium concentration of Pu in both phases was measured by alpha liquid scintillation counting (*11*). All the experiments were done in triplicate, and the agreement in the distribution ratio values was usually better than ±2%. It is known that when cyclohexane is used as the diluent, the distribution ratio of HTTA is quite low (*14*, *15*). In all the experiments with cyclohexane, the equilibrium HTTA concentration in the aqueous phase was measured spectrophotometrically (*16*) and the organic phase equilibrium HTTA concentration was corrected accordingly. When cyclohexane was used as the diluent, the distribution ratio values of Pu(IV) were corrected for aqueous phase complexing of Pu(IV) by HTTA by assuming that $Pu(TTA)^{3+}$ is the dominant species with formation constant 83 (*17*).

For spectrophotometric studies, macroconcentrations of Pu(IV) in nitric acid were extracted into 0.1 *M* HTTA in benzene. The benzene solution of

Pu(TTA)$_4$ thus obtained was subsequently diluted with DBBP in benzene to give several solutions with the same concentration of Pu(IV) (1.91×10^{-3} *M*) but various concentrations of DBBP. The absorption spectra of these solutions were measured against a benzene blank by using a Cary-14 recording spectrophotometer.

RESULTS AND DISCUSSION

The equilibria involved in the extraction of Pu(IV) from perchloric acid by mixtures of a neutral donor and HTTA are presented below. When D is the overall distribution ratio for Pu(IV) obtained with the mixture of extractants, it can be represented by

$$D = D_A + D_B + D_{AB} \tag{1}$$

where D_A is the distribution ratio when HTTA alone was used, D_B is the distribution ratio when the neutral donor (S) alone was used, and D_{AB} is the distribution ratio that accounts for the other species extracted, which are responsible for the synergism. The composition of such species and the equilibrium quotients of their formation can be obtained by studying the variation of D_{AB} as a function of the several variables involved.

Extraction of Pu(IV) by Mixtures of HTTA and DBBP in Benzene

Results obtained for the extraction of Pu(IV) at constant HTTA concentration and various DBBP concentrations are given in Table 1. In all the tables and figures the concentrations of HTTA, TBP, and DBBP refer to the equilibrium organic phase concentrations. The values of D_B, at the concentrations of DBBP used, are negligible. The D_{AB} values thus obtained are plotted in Fig. 1 as a function of DBBP concentration. Because the

TABLE 1

Variation of the Distribution Ratio of Pu(IV) with DBBP concentration (diluent: benzene)

[DBBP] (*M*)	[HTTA] = 0.01 *M*			[HTTA] = 0.0125 *M*		
	D	D_{AB}	β_{AB}	D	D_{AB}	β_{AB}
0	0.206	—	—	0.555	—	—
0.002	0.479	0.273	663	1.25	0.695	626
0.004	0.750	0.544	660	2.04	1.49	669
0.006	1.03	0.825	668	2.86	2.31	692
0.008	1.36	1.15	700	3.65	3.10	697
0.010	1.66	1.45	704	4.27	3.72	669

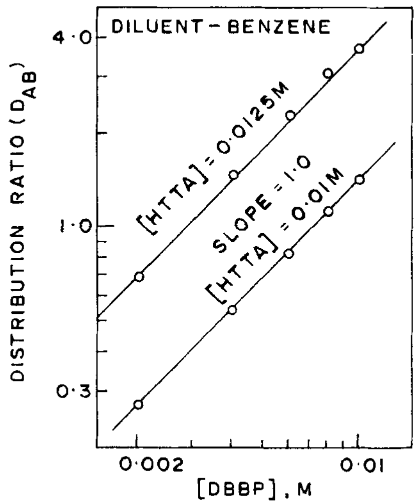

FIG. 1. Variation of distribution ratio (D_{AB}) of Pu(IV) with DBBP concentration in the presence of HTTA.

slope of the log-log plot obtained is unity, it follows that one molecule of DBBP is associated with the extractable complex responsible for synergism. Assuming that this complex is the adduct of $Pu(TTA)_4$ with DBBP, the extraction of the adduct can be given by the equilibrium

$$Pu^{4+}_{(aq)} + 4HTTA_{(org)} + DBBP_{(org)} \overset{K_{AB}}{\rightleftharpoons} Pu(TTA)_4 \cdot DBBP_{(org)} + 4H^{+}_{(aq)} \tag{2}$$

and its equilibrium constant by

$$K_{AB} = \frac{[Pu(TTA)_4DBBP][H^+]^4}{[Pu^{4+}][HTTA]^4[DBBP]} = \frac{D_{AB}[H^+]^4}{[HTTA]^4[DBBP]} \tag{3}$$

As the extraction of $Pu(TTA)_4$ is governed by the equilibrium shown in

$$Pu^{4+}_{(aq)} + 4HTTA_{(org)} \overset{K_A}{\rightleftharpoons} Pu(TTA)_{4(org)} + 4H^{+}_{(aq)} \tag{4}$$

one obtains, by substracting Eq. (4) from Eq. (2), the organic phase adduct formation equilibrium as shown by

$$Pu(TTA)_{4(org)} + DBBP_{(org)} \overset{\beta_{AB}}{\rightleftharpoons} Pu(TTA)_4 \cdot DBBP_{(org)} \tag{5}$$

Thus

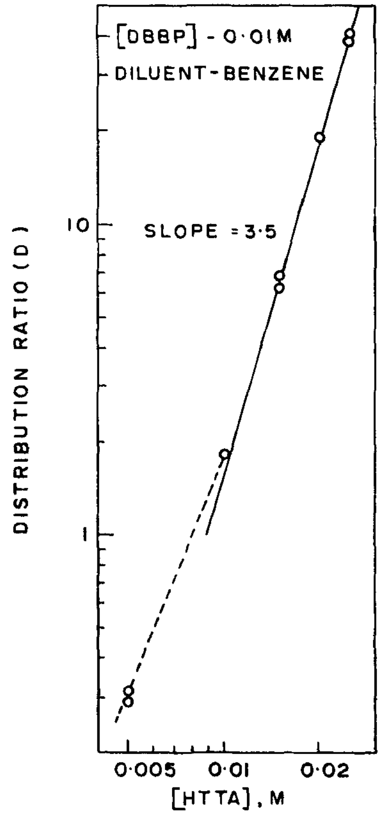

FIG. 2. Variation of distribution ratio (D) of Pu(IV) with HTTA concentration in the presence of DBBP.

$$\beta_{AB} = \frac{K_{AB}}{K_A} = \frac{D_{AB}}{D_A} \times \frac{1}{[\mathrm{DBBP}]} \tag{6}$$

Using the relation given in Eq. (6) and the data given in Table 1, the β_{AB} values were calculated and are also included in Table 1. It is seen that all the values obtained are fairly close to each other, giving an average value of $\beta_{AB} = 675$.

Results obtained on the variation of D at constant DBBP concentration as a function of HTTA concentration are shown in Fig. 2. The log-log plot gave a slope ~ 3.5 when [HTTA] $\geqslant$ [DBBP] and an even smaller slope when [HTTA] $<$ [DBBP]. This suggests that species other than the suggested adduct are also involved in the extraction when [HTTA] $<$ [DBBP]. As these species contain less than four ions of TTA, it is reasonable to infer that they are associated with perchlorate ions so as to make the species electrically neutral.

In order to corroborate these results, the extraction was also investigated by Job's method of continuous variations as applied to solvent extraction systems by Gal and Nikolic (*18*). In this method the distribution ratio data are obtained by keeping the total concentration of HTTA + DBBP constant and varying the fraction of each component. When [HTTA] + [DBBP] = C and [HTTA] = xC, it follows that [DBBP] = $(1 - x)C$. By substituting these values for [HTTA] and [DBBP] in Eq. (3), it follows that

$$K_{AB} = \frac{D_{AB}}{x^4(1-x)} \frac{[H^+]^4}{C^5} \tag{7}$$

The distribution ratio data obtained as a function of x with $C = 0.02$ M and 0.01 M are given in Tables 2 and 3, respectively. The values of D_{AB} were calculated by using Eq. (1). The D_A and D_B values for different values of x were calculated from the values of D when $x = 1$ and 0, respectively, assuming $D_A \propto [HTTA]^4$ and $D_B \propto [DBBP]^2$. It is seen that the values of $D_{AB}/x^4(1 - x)$ for different values of x do not remain constant as demanded by Eq. (7). This suggests that more species besides $Pu(TTA)_4 \cdot DBBP$ are involved in the extraction. Assuming that when [HTTA] ≫ [DBBP] the predominant synergistic species is $Pu(TTA)_4 \cdot DBBP$, the corresponding value of $D_{AB}/x^4(1 - x)$ was obtained by extraporating the values of $D_{AB}/x^4(1 - x)$ to $x = 1$. Extrapolation from the data in Tables 2 and 3 gave $D_{AB}/x^4(1 - x) = 40$ and 1.8, respectively. The K_{AB} values estimated by putting these values in Eq. (7) are $\log K_{AB} = 10.10$ and 10.26, respectively.

TABLE 2

Variation of the Distribution Ratio of Pu(IV) with Simultaneous Variation in the Concentration of HTTA and DBBP (diluent: benzene)
[HTTA] + [DBBP] = 0.02 M

x	D	D_{AB}	$D_{AB}/x^4(1-x)$
1.0	3.44	—	—
0.9	4.42	2.16	32.9
0.8	4.80	3.39	41.4
0.7	3.90	3.07	42.6
0.6	2.79	2.34	45.1
0.5	1.93	1.71	54.7
0.4	1.16	1.06	69.0
0.3	0.614	0.578	102
0.2	0.287	0.271	212
0.1	0.108	0.095	1056
0	0.0161	—	—

TABLE 3

Variation of the Distribution Ratio of Pu(IV) with Simultaneous Variation in the Concentration of HTTA and DBBP (diluent: benzene)
[HTTA] + [DBBP] = 0.01 M

x	D	D_{AB}	$D_{AB}/x^4(1-x)$
1.0	0.236	—	—
0.9	0.278	0.123	1.88
0.8	0.266	0.169	2.06
0.7	0.243	0.186	2.58
0.6	0.192	0.160	3.09
0.5	0.153	0.136	4.35
0.4	0.113	0.105	6.84
0.3	0.0818	0.0767	13.5
0.2	0.0464	0.0418	32.7
0.1	0.0193	0.0140	156
0	0.0065	—	—

TABLE 4

Variation of the Distribution Ratio of Pu(IV) with DBBP Concentration (diluent: cyclohexane)
[HTTA] = 0.01 M

[DBBP] (M)	D	D_{AB}	$\beta_{AB} \times 10^{-4}$
0	0.487	—	—
0.0002	2.29	1.80	1.85
0.0004	4.25	3.76	1.93
0.0006	5.89	5.40	1.85
0.0008	7.64	7.15	1.84
0.0010	10.1	9.61	1.97

Extraction of Pu(IV) by Mixtures of HTTA and DBBP in Cyclohexane

Results obtained for the extraction of Pu(IV) at constant HTTA concentration and various DBBP concentrations are given in Table 4. The D_{AB} values obtained using Eq. (1) from these data are shown in Fig. 3. The unit value of the slope obtained from the plot again suggests that one molecule of DBBP is associated with the complex responsible for synergism. The β_{AB} values obtained using Eq. (5) are included in Table 4. Thus the average value of β_{AB} (1.89×10^4) is obtained for the equilibrium shown in Eq. (5) with cyclohexane as the diluent.

To verify the equilibrium shown in Eq. (4) for the cyclohexane medium, the distribution ratio values were obtained as a function of HTTA con-

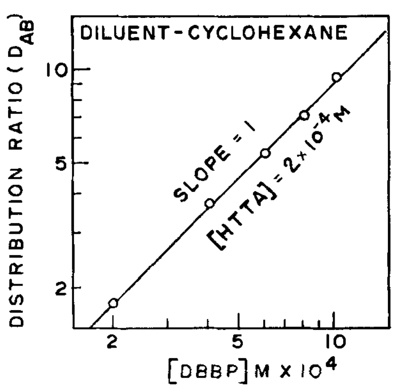

FIG. 3. Variation of distribution ratio (D_{AB}) of Pu(IV) with DBBP concentration in the presence of HTTA.

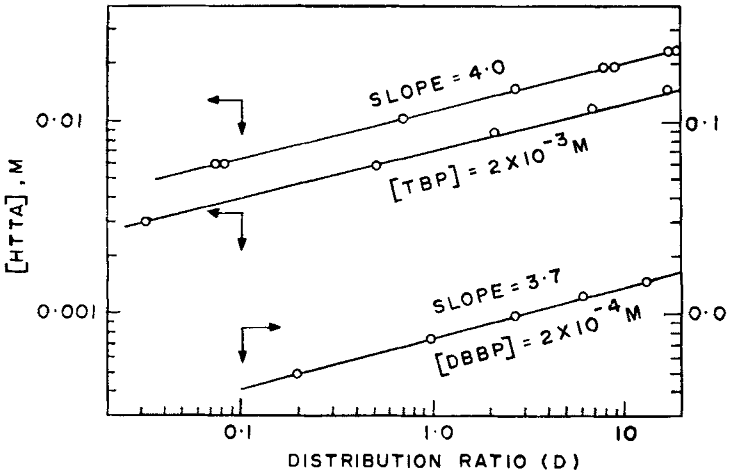

FIG. 4. Variation of distribution ratio (D) of Pu(IV) with HTTA concentration.

centration and they are shown in Fig. 4. The log-log plot gave a slope of +4, in agreement with Eq. (4). Similar data were then obtained in the presence of DBBP, and they are included in Fig. 4. The slightly lower slope value (+3.7) obtained in this case suggests that additional extractable species, as in the case of the benzene medium, may be involved, though to a lesser extent, in this medium also. The results obtained for this extraction by Gal and Nikolic's method are given in Table 5. Although the maximum D_{AB} value is again in agreement with the extractable species $Pu(TTA)_4 \cdot DBBP$, the $D_{AB}/x_4(1 - x)$ values suggest that more species are present in the extraction. Following the extrapolation procedure adopted earlier, the value for $D_{AB}/x^4(1 - x) = 3.6$ was estimated for $x = 1$. Assuming that the predominant synergistic species is $Pu(TTA)_4 \cdot DBBP$ under these conditions, log K_{AB} was calculated to be 12.06.

Extraction of Pu(IV) by Mixtures of HTTA and TBP in Cyclohexane

Variation of the distribution ratio of Pu(IV) was studied at constant HTTA concentration as a function of TBP concentration. The results are given in Table 6. The D_{AB} values obtained from these data using Eq. (1) are plotted in Fig. 5 against TBP concentration. The unit value of the slope of the bilogarithmic plot suggests that the synergistic species contains one molecule of TBP. The β_{AB} value for the equilibrium shown by

$$Pu(TTA)_{4(org)} + TBP_{(org)} \overset{\beta_{AB}}{\rightleftharpoons} Pu(TTA)_4 \cdot TBP_{(org)} \qquad (8)$$

was calculated by an equation similar to Eq. (6) and is included in Table 6.

TABLE 5

Variation of the Distribution Ratio of Pu(IV) with Simultaneous Variation in the Concentration of HTTA and DBBP (diluent: cyclohexane) [HTTA] + [DBBP] = 0.005 *M*

x	D	D_{AB}	$D_{AB}/x^4(1-x)$
1.0	0.0354	—	—
0.9	0.258	0.235	3.58
0.8	0.309	0.294	3.59
0.7	0.277	0.268	3.72
0.6	0.215	0.209	4.03
0.5	0.146	0.143	4.58
0.4	0.0957	0.0928	6.04
0.3	0.0586	0.0556	9.81
0.2	0.0357	0.0320	25.0
0.1	0.0194	0.0149	166
0	0.0056	—	—

TABLE 6

Variation of the Distribution Ratio of Pu(IV) with TBP Concentration (diluent: cyclohexane)

[TBP] (M)	[HTTA] = 0.0105 M			[HTTA] = 0.0150 M		
	D	D_{AB}	$\beta_{AB} \times 10^{-3}$	D	D_{AB}	$\beta_{AB} \times 10^{-3}$
0	0.592	—	—	2.18	—	—
0.002	3.28	2.68	2.27	12.0	9.85	2.26
0.004	5.74	5.15	2.18	20.1	17.9	2.05
0.006	7.88	7.29	2.05	27.3	25.1	1.92
0.008	10.9	10.3	2.18	34.4	32.3	1.85
0.010	12.5	11.9	2.02	41.7	39.5	1.81

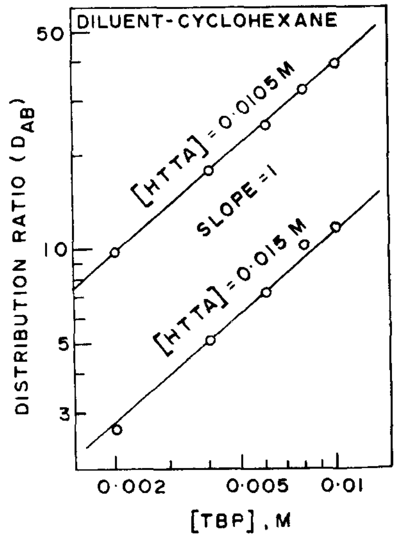

FIG. 5. Variation of distribution ratio (D_{AB}) of Pu(IV) with TBP concentration in the presence of HTTA.

It is seen that the values obtained are reasonably constant, giving an average value of $\beta_{AB} = 2.06 \times 10^3$.

Results obtained on the variation of D at constant TBP concentration as a function of HTTA concentration are included in Fig. 4. The log-log plot gave a slope value of 4.0, in agreement with

$$\mathrm{Pu}^{4+}_{(aq)} + 4\mathrm{HTTA}_{(org)} + \mathrm{TBP}_{(org)} \overset{K_{AB}}{\rightleftharpoons} \mathrm{Pu(TTA)_4 \cdot TBP}_{(org)} + 4\mathrm{H}^{+}_{(aq)} \quad (9)$$

Thus no species other than $\mathrm{Pu(TTA)_4 \cdot TBP}$ seems to be involved in this system. To substantiate this conclusion, data were also obtained by Gal

TABLE 7

Variation of the Distribution Ratio of Pu(IV) with Simultaneous Variation in the Concentration of HTTA and TBP (diluent: cyclohexane) [HTTA] + [TBP] = 0.01 *M*

x	D	D_{AB}	$D_{AB}/x^4(1-x)$
1.0	0.550	—	—
0.9	1.32	0.960	14.6
0.8	1.36	1.14	13.9
0.7	1.09	0.958	13.3
0.6	0.774	0.703	13.6
0.5	0.471	0.437	14.0
0.4	0.232	0.218	14.2
0.3	0.0878	0.0833	14.7
0.2	0.0203	0.0194	15.2
0.1	0.00143	0.00138	15.3
0	0.00007	—	—

and Nikolic's method and are given in Table 7. The data presented in this table clearly show that the maximum D_{AB} value is in accordance with the adduct composition. The constancy of the $D_{AB}/x^4(1-x)$ value for all values of x rules out the possibility of any additional species. Thus, in cyclohexane as well as in the benzene medium studied earlier (*11*), it is clear that $Pu(TTA)_4 \cdot TBP$ is the only synergistic species formed. The K_{AB} value calculated from the average value of $D_{AB}/x^4(1-x)$ was found to be $\log K_{AB} = 11.16$.

Spectrophotometric Study of the Formation of Adducts of $Pu(TTA)_4$ with DBBP in Benzene Medium

The absorption spectrum of $Pu(TTA)_4$ and the changes caused in it by the addition of different amounts of DBBP are shown in Fig. 6. The well-defined isosbestic points observed at several wavelengths clearly suggest that there are only two absorbing species present in the system; viz., $Pu(TTA)_4$ and its adduct with DBBP. By analogy with similar studies carried out earlier (*11*) with TBP, it is reasonable to assume that the adduct formed is $Pu(TTA)_4 \cdot DBBP$. More work is in progress to obtain the adduct formation constant by spectrophotometry.

The values of the equilibrium constants obtained in this work by different methods are given in Table 8 along with earlier values for comparison. It is seen that the values obtained by the two methods are in reasonable agreement.

The present studies reveal that mixed ligand complexes, with the

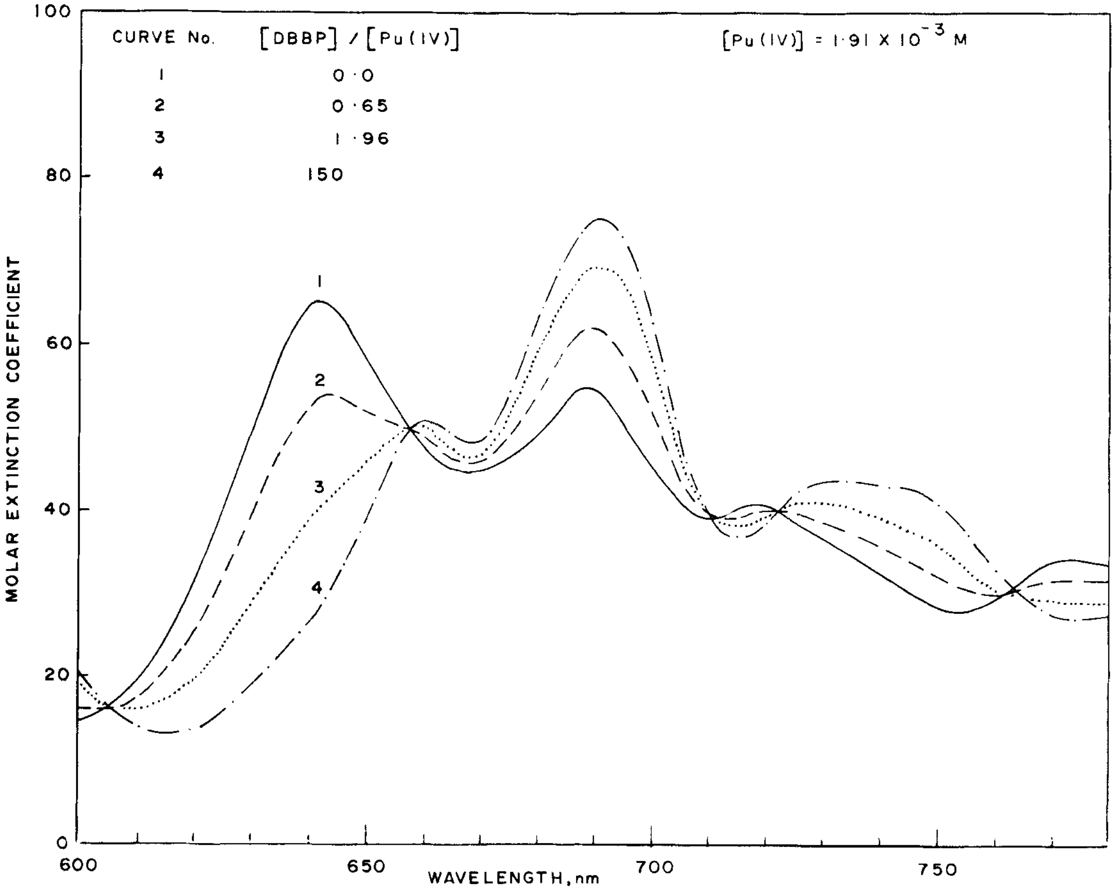

FIG. 6. Changes in the absorption of HTTA extract of Pu(IV) with DBBP concentration.

TABLE 8

Summary of the Equilibrium Constant Values: Adduct, $Pu(TTA)_4 \cdot S_1$; Aqueous Phase, 1.0 *M* $HClO_4$

Source of the data	Neutral donor(s)	Diluent	log K_A	log K_{AB}	log β_{AB}
Ref. *11*	TBP	Benzene	7.34	9.06	1.72
Table 1	DBBP	"	"	10.17	2.83
Table 2	"	"	"	10.10	2.76
Table 3	"	"	"	10.26	2.92
Table 4	"	Cyclohexane	7.74	12.02	4.28
Table 5	"	"	"	12.06	4.32
Table 6	TBP	"	"	11.05	3.31
Table 7	"	"	"	11.16	3.42

participation of the perchlorate ion, are involved in the synergistic extraction of Pu(IV) when DBBP was used as the neutral donor. It is felt that participation of the anion from the aqueous medium in the synergistic mixed ligand complexes depends primarily on the extractibility of the metal ion by the neutral donor alone from the aqueous medium used. It is known that with neutral organophosphorous extractants the extractibility of Pu(IV) follows the order phosphate < phosphonate < phosphinate < phosphine oxide. The absence of a mixed ligand species involving the perchlorate ion, with TBP as the neutral donor, is apparently due to poor extractibility of Pu(IV) from perchlorate medium by TBP alone at the concentrations used. It is anticipated that the participation of perchlorate ion in the extractable synergistic species would be more pronounced when phosphine oxides are used as the neutral donors. This was, in fact, found to be true in the extraction of Np(IV) from aqueous perchloric acid with mixtures of HTTA and TOPO (*19*).

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