Short communication

Synergistic Extraction of Europium into Nitrobenzene by Using Hydrogen Dicarbollylcobaltate and 2,6 – (Diphenylphosphino)Pyridine Dioxide

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Abstract

Solvent extraction of microamounts of europium by a nitrobenzene solution of hydrogen dicarbollylcobaltate (H⁺B⁻) in the presence of 2,6 – (diphenylphosphino)pyridine dioxide (DPPPDO, L) has been investigated. The equilibrium data have been explained assuming that the species HL⁺, HL⁺₂, EuL³⁺₃ and EuL³⁺₄ are extracted into the organic phase. The values of extraction and stability constants of the species in nitrobenzene saturated with water have been determined.

Keywords: Europium, hydrogen dicarbollylcobaltate, 2,6 – (diphenylphosphino)pyridine dioxide, water-nitrobenzene system, extraction and stability constants

1. Introduction

Bidentate phosphonates, phosphine oxides and malonamides have been intensively studied for the extraction of trivalent lanthanides and actinides from acidic media.¹⁻³ A process using octyl-phenyl-*N*,*N*-diisobutylcarbamoylmethyl phosphine oxide (i.e. "classical" CMPO) and called TRUEX was apparently used in the United States,¹ while malonic diamides (RR´NCO)₂CHR´´ (DIAMEX) were proposed in France.²

The dicarbollylcobaltate anion⁴ and some of its halogen derivatives are very useful reagents for the extraction of various metal cations (especially Cs^+ , Sr^{2+} , Ba^{2+} , Eu^{3+} and Am^{3+}) from aqueous solutions into a polar organic phase, both under laboratory conditions for purely theoretical or analytical purposes, ^{5–42} and on the technological scale for the separation of some high-activity isotopes in the reprocessing of spent nuclear fuel and acidic radioactive waste. ^{43,44}

In the current work, the solvent extraction of microamounts of europium by using a synergistic mixture of hydrogen dicarbollylcobaltate $(H^+B^-)^4$ and 2,6 – (diphenylphosphino)pyridine dioxide (DPPPDO, L; see Scheme 1) in nitrobenzene was studied. We intended to find the composition of the complex species in the organic phase and to determine the corresponding equilibrium constants.

2. Experimental

Preparation of 2,6 – (diphenylphosphino)pyridine dioxide (DPPPDO, L; see Scheme 1) was presented in Ref. 45. Cesium dicarbollylcobaltate, Cs^+B^- , was synthesized by means of the method published by Hawthorne et al.⁴⁶ A nitrobenzene solution of hydrogen dicarbollylcobaltate (H⁺B⁻)⁴ was prepared from Cs^+B^- by the procedure described elsewhere.⁴⁷ The other chemicals used (Lac-

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Scheme 1. Structural formula of 2,6 – (diphenylphosphino)pyridine dioxide (abbrev. DPPPDO or L, respectively).

hema, Brno, Czech Republic) were of reagent grade purity. The radionuclide ^{152,154}Eu³⁺ (Polatom, Poland) was of standard radiochemical purity.

The extraction experiments in the two–phase water–HCl–Eu³⁺ (microamounts)–nitrobenzene–DPPPDO– H⁺B⁻ system were performed in 10 mL glass test-tubes with polyethylene stoppers, using 2 mL of each phase. The test-tubes filled with the solutions were shaken for 2 h at 25 ± 1 °C, using a laboratory shaker. Under these conditions, the equilibria in the systems under study were established after approximately 20 min of shaking. Then the phases were separated by centrifugation. Afterwards, 1 mL samples were taken from each phase and their γ -activities were measured by means of a well-type NaI(T1) scintillation detector connected to a γ -analyzer NK 350 (Gamma, Budapest, Hungary).

The equilibrium distribution ratios of europium, D, were determined as the ratios of the corresponding measured radioactivities of ^{152,154}Eu³⁺ in the nitrobenzene and aqueous samples.



Figure 1. Log D as a function of log c(L), where L is DPPPDO, for the water–HCl–Eu³⁺ (microamounts)–nitrobenzene–DPPPDO– H⁺B⁻ system; c(HCl) = 0.10 mol/L, c_B = 0.001 mol/L. The curve was calculated using the constants given in Table 2.

3. Results and Discussion

The dependence of the logarithm of the europium distribution ratio (log D) on the logarithm of the numerical value of the total (analytical) concentration of the DPPPDO ligand in the initial nitrobenzene phase, log c(L), is given in Figure 1. The initial concentration of hydrogen dicarbollylcobaltate in the nitrobenzene phase, $c_B = 0.001$ mol/L, as well as the initial concentration of HCl in the aqueous phase, c(HCl) = 0.10 mol/L, are always related to the volume of one phase.

With respect to the results of previous papers, 9,25,29,35,38 the considered water-HCl-Eu³⁺ (microa-mounts)-nitrobenzene-DPPPDO(L)-H⁺B⁻ system can be described by the set of reactions:

$$L_{aq} \Leftrightarrow L_{org}$$
 (1)

$$H^{+}_{\rm org} + L^{-}_{\rm org} \Leftrightarrow HL^{+}_{\rm org}$$
(2)

$$H_{org}^{+} + 2L_{org} \Leftrightarrow HL_{2,org}^{+}$$
 (3)

$$Eu_{aq}^{3+} + 3H_{org}^{+} \Leftrightarrow Eu_{org}^{3+} + 3H_{aq}^{+}$$
(4)

$$\mathrm{Eu}_{\mathrm{aq}}^{3+} + \mathrm{nL}_{\mathrm{org}} + 3\mathrm{H}_{\mathrm{org}}^{+} \Leftrightarrow \mathrm{EuL}_{\mathrm{n,org}}^{3+} + 3\mathrm{H}_{\mathrm{aq}}^{+} \tag{5}$$

to which the following equilibrium constants correspond:

$$K_{\rm D} = \frac{[L_{\rm org}]}{[L_{\rm aq}]} \tag{6}$$

$$B(HL_{org}^{+}) = \frac{[HL_{org}^{+}]}{[H_{org}^{+}][L_{org}]}$$
(7)

$$\beta(\text{HL}_{2,\text{org}}^{+}) = \frac{[\text{HL}_{2,\text{org}}^{+}]}{[\text{H}_{\text{org}}^{+}][\text{L}_{\text{org}}]^{2}}$$
(8)

$$K_{ex}(Eu_{org}^{3+}) = \frac{[Eu_{org}^{3+}][H_{aq}^{+}]^{3}}{[Eu_{aq}^{3+}][H_{org}^{+}]^{3}}$$
(9)

$$K_{ex}(EuL_{n,org}^{3+}) = \frac{[EuL_{n,org}^{3+}][H_{aq}^{+}]^{3}}{[Eu_{aq}^{3+}][L_{org}]^{n}[H_{org}^{+}]^{3}}$$
(10)

The subscripts "aq" and "org" denote the aqueous and organic phases, respectively.

A subroutine UBBE, based on the relations given above, the mass balance of the DPPPDO ligand and the electroneutrality conditions in both phases of the system under consideration, was formulated^{48,49} and introduced into a more general least-squares minimizing program LETAGROP⁵⁰ used for determination of the "best" values of the extraction constants $K_{ex}(EuL_{n,org}^{3+})$ (L = DPPPDO). The minimum of the sum of errors in log D, i.e., the minimum of the expression

$$U = \sum (\log D_{calc} - \log D_{exp})^2$$
(11)

was sought.

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The values log $K_D = 2.38$ (see Table 2, footnote *a*), log $\beta(HL_{2,org}^+) = 9.45$ (see Table 2, footnote *b*), log $\beta(HL_{2,org}^+) = 11.99$ (see Table 2, footnote *b*) and log $K_{ex}(Eu_{org}^{3+}) = 1.3^{53}$ were used for the respective calculations. The results are listed in Table 1. From this table it is evident that the extraction data can be best explained assuming the complexes EuL_3^{3+} and EuL_4^{3+} (L = DPPPDO) to be extracted into the nitrobenzene phase.

Table 1. Comparison of various models of europium extraction from aqueous solution of HCl by nitrobenzene solution of H^+B^- in the presence of DPPPDO.

| Europium complexe | s log K _{ex} ^a | U ^{<i>b</i>} |
|--|---|-----------------------|
| in the organic phase | | |
| EuL ³⁺ | 31.37 (32.41) | 151.25 |
| $\operatorname{EuL}_{3}^{\overline{3}+}$ | 37.63 (38.05) | 3.95 |
| $\operatorname{EuL}_{4}^{3+}$ | 43.55 (44.33) | 124.16 |
| $EuL_{2}^{3+}, EuL_{3}^{3+}$ | Transformed to EuL_3^{3+} | |
| $EuL_{3}^{\tilde{3}+}, EuL_{4}^{\tilde{3}+}$ | 37.10 (37.38), 40.86 (41.20) | 0.04 |
| $EuL_{2}^{3+}, EuL_{3}^{3+}, EuL_{4}^{3+}$ | Transformed to EuL_3^{3+}, EuL_4^{3+} | |

^{*a*} The values of the extraction constants are given for each complex. The reliability interval of the constants is given as $3\sigma(K)$, where $\sigma(K)$ is the standard deviation of the constant K.⁵⁰ These values are given in the logarithmic scale using the approximate expression log K ± {(log [K + 1.5 $\sigma(K)$]) –log [K – 1.5 $\sigma(K)$]}. For $\sigma(K) > 0.2$ K, the previous expression is not valid and then only the upper limit is given in the parentheses in the form of log K (log [K + $3\sigma(K)$]).⁵⁰ ^{*b*} The error-square sum U = Σ (log D_{ealc} – log D_{eap}².

Table 2. Equilibrium constants in the water- $HCl-Eu^{3+}$ (microamounts)-nitrobenzene-DPPPDO- H^+B^- system.

| Equilibrium | log K | |
|--|--------------------|--|
| $L_{aq} \Leftrightarrow L_{org}$ | 2.38 ^a | |
| $H_{\text{org}}^{+} + L_{\text{org}}^{+} \Leftrightarrow HL_{\text{org}}^{+}$ | 9.45 ^b | |
| $H_{\text{org}}^+ + 2L_{\text{org}}^+ \Leftrightarrow HL_{2\text{ org}}^+$ | 11.99 ^b | |
| $Eu_{aq}^{3+} + 3H_{org}^{++} \Leftrightarrow Eu_{org}^{3+} + 3H_{aq}^{+}$ | 1.3 ^c | |
| $Eu_{aq}^{3+} + 3L_{org}^{+} + 3H_{org}^{+} \Leftrightarrow EuL_{3 org}^{3+} + 3H_{aq}^{+}$ | 37.10 | |
| $\operatorname{Eu}_{\operatorname{aq}}^{3+} + 4L_{\operatorname{org}}^{4+} + 3H_{\operatorname{org}}^{++} \Leftrightarrow \operatorname{Eu}_{\operatorname{4,org}}^{3+} + 3H_{\operatorname{aq}}^{++}$ | 40.86 | |
| $Eu_{org}^{3+} + 3L_{org}^{3+} \Leftrightarrow EuL_{3,org}^{3+}$ | 35.80 | |
| $Eu_{\text{org}}^{3+} + 4L_{\text{org}}^{3+} \Leftrightarrow EuL_{4,\text{org}}^{3+}$ | 39.56 | |

^{*a*} Determined by the method of the concentration dependent distribution.⁵¹ ^{*b*} Determined by the method described in detail in Ref. 52. ^{*c*} Ref. 53.

Knowing the values $\log K_{ex}(Eu_{org}^{3+}) = 1.3$,⁵³ as well as the extraction constants $\log K_{ex}(EuL_{3,org}^{3+}) = 37.10$ and $\log K_{ex}(EuL_{4,org}^{3+}) = 40.86$ determined here (Table 1), the stability constants of the complexes EuL_3^{3+} and EuL_4^{3+} (L = DPPPDO) in the nitrobenzene phase defined as

$$\beta(\text{EuL}_{3,\text{org}}^{3+}) = \frac{[\text{EuL}_{3,\text{org}}^{3+}]}{[\text{Eu}_{\text{org}}^{3+}][\text{L}_{\text{org}}]^3}$$
(12)

$$\beta(\text{EuL}_{4,\text{org}}^{3+}) = \frac{[\text{EuL}_{4,\text{org}}^{3+}]}{[\text{Eu}_{\text{org}}^{3+}][\text{L}_{\text{org}}]^4}$$
(13)

can be evaluated applying the simple relations:

$$log \beta(EuL_{3,org}^{3+}) = log K_{ex}(EuL_{3,org}^{3+}) - log K_{ex}(Eu_{org}^{3+})$$
(14)

$$\log \beta(\text{EuL}_{4,\text{org}}^{3+}) = \log K_{\text{ex}}(\text{EuL}_{4,\text{org}}^{3+}) - \log K_{\text{ex}}(\text{Eu}_{\text{org}}^{3+})$$
(15)

The respective equilibrium constants are summarized in Tables 3.

Table 3. Stability constants of the complex species HL^+ , HL_2^+ and EuL_n^{3+} [n = 2, 3, 4; L = octyl-phenyl-*N*,*N*-diisobutylcarbamoylmethyl phosphine oxide ("classical" CMPO), 2,6 – (diphenylphosphino)pyridine dioxide (DPPPDO)] in nitrobenzene saturated with water at 25 °C.

| | L | | |
|--------------------------------------|-------------------------------|---------------------|--|
| Quantity | "classical" CMPO ^a | DPPPDO ^b | |
| $\log \beta(HL_{org}^+)$ | 6.16 | 9.45 | |
| $\log \beta(HL_{2,org}^+)$ | 9.29 | 11.99 | |
| $\log \beta(EuL_{2,org}^{3+\sigma})$ | 18.13 | _ | |
| $\log \beta(EuL_{3,org}^{3+})$ | 24.26 | 35.80 | |
| $\log \beta(EuL_{4,org}^{3+})$ | 29.13 | 39.56 | |

^a Ref. 9. ^b This work.

Moreover, Figure 2 presents the contributions of the species H_{org}^+ , HL_{org}^+ and $HL_{2,org}^+$ to the total hydrogen cation concentration in the equilibrium nitrobenzene phase, whereas Figure 3 depicts the contributions of the cationic complexes $EuL_{3,org}^{3+}$ and $EuL_{4,org}^{3+}$ to the total trivalent europium cation concentration in the equilibrium organic phase.

In conclusion, Table 3 summarizes the stability constants of the complex species HL⁺, HL⁺₂ and EuL³⁺_n (n = 2, 3, 4) with two electroneutral ligands L, denoted by the symbols "classical" CMPO and DPPPDO (see Schemes 1 and 2), in nitrobenzene saturated with water. From the data reviewed in this table it follows that in the considered nitrobenzene medium, the stability constants of the cationic species HL⁺, HL⁺₂, EuL³⁺₃ and EuL³⁺₄, where L = DPPP-DO, are substantially higher than those of the corresponding species HL⁺, HL⁺₂, EuL³⁺₃ and EuL³⁺₄ involving the "classical" CMPO ligand.

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Figure 2. Distribution diagram of hydrogen cation in the equilibrium organic phase of the water- HCl-Eu³⁺ (microamounts) - nitrobenzene-DPPPDO-H+B- extraction system in the forms of H+, HL^+ and HL_2^+ ;

 $c(HCl) = 0.10 \text{ mol/L}, c_B = 0.001 \text{ mol/L}.$

 $1 \delta(H^+) = [H^+_{org}]/c(H^+)$

 $2 \,\delta(\mathrm{HL}^{+}) = [\mathrm{H}\tilde{\mathrm{L}}_{\mathrm{org}}^{+}]/c(\mathrm{H}^{+})_{\mathrm{org}},$

 $3 \, \delta(HL_2^+) = [HL_{2,org}^{+}]/c(H^+)_{org}^{+},$ where $c(H^+)_{org} = [H_{org}^+] + [HL_{org}^+] + [HL_{2,org}^+].$

The distribution curves were calculated using the constants given in Table 2.



Figure 3. Distribution diagram of europium in the equilibrium organic phase of the water- HCl-Eu³⁺ (microamounts)-nitrobenzene–DPPDO–H⁺B⁻ extraction system in the forms of EuL_{3}^{3+} and $EuL_{4}^{3+};$

 $c(HCl) = 0.10 \text{ mol/L}, c_B = 0.001 \text{ mol/L}.$

 $I \ \delta(\text{EuL}_{3}^{3+}) = [\text{EuL}_{3,\text{org}}^{3+}]/c(\text{Eu}^{3+})_{\text{org}}, 2 \ \delta(\text{EuL}_{4}^{3+}) = [\text{EuL}_{4,\text{org}}^{3+}]/c(\text{Eu}^{3+})_{\text{org}},$

where
$$c(Eu^{3+})_{org} = [EuL_{3,org}^{3+}] + [EuL_{4,org}^{3+}]$$
.

The distribution curves were calculated using the constants given in Table 2.



Scheme 2. Structural formula of octyl-phenyl-N,N-diisobutylcarbamoylmethyl phosphine oxide (abbrev. "classical" CMPO).

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Povzetek

Proučevali smo ekstrakcijo evropija z raztopino hydrogen dikarbolilkobaltata (H⁺B⁻) v nitrobenzen ob prisotnosti 2,6 – (difenilfosfino)piridin dioksida (DPPPDO, L). Eksperimentalne podatke o ravnotežni koncentraciji smo obravnavali ob predpostavki, da se ionske zvrsti HL⁺, HL⁺₂, EuL³⁺₃ in EuL³⁺₄ in extrahirajo v organsko fazo. Določili smo vrednosti ravnotežnih konstant za ekstrakcijo in konstant stabilnosti za omenjene ionske zvrsti v nitrobenzenu, nasičenem z vodo.