Short communication

Synergistic Extraction of Europium and Americium into Nitrobenzene by Using Hydrogen Dicarbollylcobaltate and Dodecaethylene Glycol

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Abstract

Extraction of microamounts of europium and americium by a nitrobenzene solution of hydrogen dicarbollylcobaltate (H^+B^-) in the presence of dodecaethylene glycol (DDEG, L) has been investigated. The equilibrium data have been explained assuming that the species HL^+, H_2L^{2+}, ML^{3+} and $MH_{-1}L^{2+}$ ($M^{3+} = Eu^{3+}$, Am^{3+}; L = DDEG) are extracted into the organic phase. The values of extraction and stability constants of the complex species in nitrobenzene saturated with water have been determined. It was found that in this nitrobenzene medium, the stability constant of the EuL^{3+} complex is comparable with that of AmL^{3+}.

Keywords: Europium, americium, hydrogen dicarbollylcobaltate, dodecaethylene glycol, complexation, water–nitrobenzene system, extraction and stability constants

1. Introduction

The dicarbollylcobaltate anion[1] 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,[2–20] and on the technological scale for the separation of some high-activity isotopes in the reprocessing of spent nuclear fuel and acidic radioactive waste.[21–23]

Solvent extraction of microamounts of Sr^{2+} and Ba^{2+} by a nitrobenzene solution of hydrogen dicarbollylcobaltate (H^+B^-) [1] in the presence of polyethylene glycols PEG 200, PEG 300 and PEG 400 has been studied. It has been found that the extraction of the protonated polyethylene glycol molecule HL^+ and the extraction of the complex ML^{2+} ($M^{2+} = Sr^{2+}$, Ba^{2+}; L = PEG 200, PEG 300, PEG 400) are predominant reactions in this water-nitrobenzene system. The respective equilibrium constants have been determined. The extraction and stability constants of the HL^+ and ML^{2+} complex cations in the organic phase increase in the cation order $H^+ < Sr^{2+} < Ba^{2+}$, whereas the hydration numbers decrease in the same sequence.[24]

Recently, the extractive properties of a synergistic mixture of hydrogen dicarbollylcobaltate (H^+B^-) [1] and dodecaethylene glycol (DDEG, L) toward Cs^+, Ca^{2+} and Sr^{2+} have been investigated in the water-nitrobenzene system.[25,26] On the other hand, in the current work, the solvent extraction of microamounts of Eu^{3+} and Am^{3+} into nitrobenzene by means of this synergistic mixture was studied. We intended to find the composition of the species in the organic phase and to determine the corresponding equilibrium constants.

2. Experimental

Dodecaethylene glycol, $HO(CH_2CH_2O)_{12}H$ (abbrev. DDEG or L, respectively), was purchased from Fluka. Cesium dicarbollylcobaltate (Cs^+B^-) was synthesized by means of the method published by Hawthorne et al.[27] The other chemicals used (Lachema, Brno, Czech Republic) were of reagent grade purity. A nitrobenzene solution of hydrogen dicarbollylcobaltate (H^+B^-)[1] was prepared from Cs^+B^- by the procedure described elsewhere.[28] The radionuclides $^{152,154}Eu^{3+}$ and $^{241}Am^{3+}$ were supplied by Polatom, Poland; their radionuclidic purities were 99.9%.

The extraction experiments in the two–phase water–HCl–DDEG–M^{3+}(microamounts; $M^{3+} = Eu^{3+}$, Am^{3+}) –nitrobenzene– H^+B^- systems 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(TI) scintillation detector connected to a γ-analyzer NK 350 (Gamma, Budapest, Hungary).

The equilibrium distribution ratios of europium and americium, D, were determined as the ratios of the corresponding measured radioactivities of $^{152,154}Eu^{3+}$ and $^{241}Am^{3+}$ in the nitrobenzene and aqueous samples.

3. Results and Discussion

The dependences of the logarithm of the europium and americium distribution ratios (log D) on the logarithm of the numerical value of the total (analytical) concentration of the ligand DDEG in the initial aqueous phase, log c(L), are given in Figures 1 and 2, respectively. The initial concentration of hydrogen dicarbollylcobaltate in the organic phase, $c_B = 0.01$ mol/L, as well as the initial concentration of HCl in the aqueous phase, c(HCl) = 0.05 mol/L, are always related to the volume of one phase.

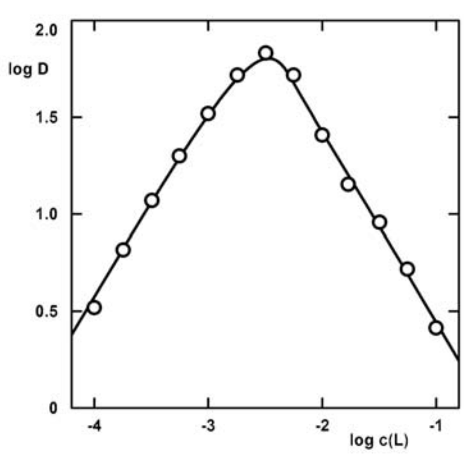

Figure 1. Log D as a function of log c(L), where L is DDEG, for the water– HCl– DDEG–Eu^{3+}(microamounts)–nitrobenzene–H^+B^- system; c(HCl) = 0.05 mol/L, c_B = 0.01 mol/L. The curve was calculated using the constants given in Table 3.

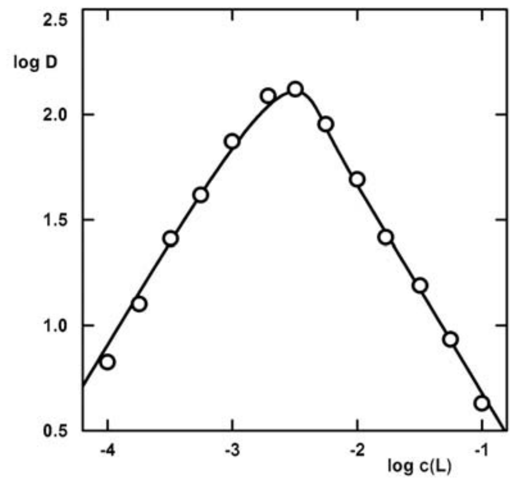

Figure 2. Log D as a function of log c(L), where L is DDEG, for the water– HCl– DDEG–Am^{3+}(microamounts)–nitrobenzene–H^+B^- system; c(HCl) = 0.05 mol/L, c_B = 0.01 mol/L. The curve was calculated using the constants given in Table 4.

With respect to previous results,[1,4–6,13–20,29–33] the considered water–HCl– DDEG (L) – M^{3+}(microamounts; $M^{3+} = Eu^{3+}$, Am^{3+})–nitrobenzene–H^+B^- systems can be described by the set of reactions:

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

$$H^+_{org} + L_{aq} \Leftrightarrow HL^+_{org} \quad (2)$$

$$2H^+_{org} + L_{aq} \Leftrightarrow H_2L^{2+}_{org} \quad (3)$$

$$M^{3+}_{aq} + 3H^+_{org} \Leftrightarrow M^{3+}_{org} + 3H^+_{aq} \quad (4)$$

$$M^{3+}_{aq} + L_{aq} + (r+3)H^+_{org} \Leftrightarrow MH_rL^{(r+3)+}_{org} + 3H^+_{aq} \quad (5)$$

to which the following equilibrium constants correspond:

$$K_D = \frac{[L_{org}]}{[L_{aq}]} \quad (6)$$

$$K_{ex}(HL^+_{org}) = \frac{[HL^+_{org}]}{[H^+_{org}][L_{aq}]} \quad (7)$$

$$K_{ex}(H_2L^{2+}_{org}) = \frac{[H_2L^{2+}_{org}]}{[H^+_{org}]^2[L_{aq}]} \quad (8)$$

$$K_{ex}(M^{3+}_{org}) = \frac{[M^{3+}_{org}][H^+_{aq}]^3}{[M^{3+}_{aq}][H^+_{org}]^3} \quad (9)$$

$$K_{ex}(MH_rL^{(r+3)+}_{org}) = \frac{[MH_rL^{(r+3)+}_{org}][H^+_{aq}]^3}{[M^{3+}_{aq}][L_{aq}][H^+_{org}]^{(r+3)}} \quad (10)$$

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 DDEG ligand and the electroneutrality conditions in both phases of the system under study, was formulated [24,34] and introduced into a more general least-squares minimizing program LETAGROP [35] used for determination of the "best" values of the extraction constants $K_{ex}(MH_rL_{org}^{(r+3)+})$ ($M^{3+} = Eu^{3+}, Am^{3+}$; L = DDEG). The minimum of the sum of errors in log D, i.e., the minimum of the expression

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

was sought.

The values $\log K_D = -3.0$ (see Table 3, footnote *a*), $\log K_{ex}(HL^+_{org}) = 4.12$ (see Table 3, footnote *b*), $\log K_{ex}(H_2L^{2+}_{org}) = 7.67$ (see Table 3, footnote *b*), $\log K_{ex}(Eu^{3+}_{org}) = 1.3$ [38] and $\log K_{ex}(Am^{3+}_{org}) = 1.5$ [38] were used for the respective calculations. The results are given in Tables 1 and 2. From these tables it is evident that the extraction data can be best explained assuming the species ML^{3+} and $MH_{-1}L^{2+}$ ($M^{3+} = Eu^{3+}, Am^{3+}$; L = DDEG) 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 DDEG.

Europium complexes in the organic phase	**log K_{ex}** [a]	**U** [b]
EuL^{3+}	11.17 (11.63)	11.41
$EuHL^{4+}$	14.29 (15.01)	57.62
$EuH_{-1}L^{2+}_{org}$	8.06 ± 0.22	0.82
EuL^{3+}, $EuHL^{4+}$	Transformed to EuL^{3+}	
EuL^{3+}, $EuH_{-1}L^{2+}_{org}$	10.23 ± 0.06, 7.78 ± 0.04	0.01

[a] The values of the extraction constants are given for each complex. The reliability interval of the constants is given as 3σ(K), where σ(K) is the standard deviation of the constant K.[35] These values are given in the logarithmic scale using the approximate expression log K ± {log[K + 1.5σ(K)] – log[K – 1.5σ(K)]} . For σ(K)>0,2K, the previous expression is not valid and then only the upper limit is given in the parentheses in the form of K(log [K + 3σ(K)].[35]
[b] The error-square sum $U = \sum(\log D_{calc} \log K - \log D_{exp})^2$.

Table 2. Comparison of various models of americium extraction from aqueous solution of HCl by nitrobenzene solution of H^+B^- in the presence of DDEG.

Americium complexes in the organic phase	**log K_{ex}** [a]	**U** [b]
AmL^{3+}	11.46 (11.91)	1.04
$AmHL^{4+}$	14.58 (15.29)	55.31
$AmH_{-1}L^{4+}_{org}$	8.35 ± 0.26	1.13
AmL^{3+}, $AmHL^{4+}$	Transformed to AmL^{3+}	
AmL^{3+}, $AmH_{-1}L^{2+}_{org}$	10.59 ± 0.08, 8.01 ± 0.06	0.01

[a] See Table 1, footnote *a*; [b] See Table 1, footnote *b*.

Knowing the values $\log K_D = -3.0$ (see Table 3, footnote *a*), $\log K_{ex}(HL^+_{org}) = 4.12$ (see Table 3, footnote *b*), $\log K_{ex}(H_2L^{2+}_{org}) = 7.67$ (see Table 3, footnote *b*), $\log K_{ex}(Eu^{3+}_{org}) = 1.3$ [38] and $\log K_{ex}(Am^{3+}_{org}) = 1.5$,[38] as well as the extraction constants $\log K_{ex}(Eu^{3+}_{org}) = 10.23$ and $\log K_{ex}(AmL^{3+}_{org}) = 10,59$ determined here (see Tables 1 and 2), the stability constants of the complexes HL^+_{org}, $H_2L^{2+}_{org}$ and ML^{3+}_{org} ($M^{3+} = Eu^{3+}, Am^{3+}$; L = DDEG) in the nitrobenzene phase defined as

$$\beta(HL^+_{org}) = \frac{[HL^+_{org}]}{[H^+_{org}][L_{org}]} \quad (12)$$

$$\beta(H_2L^{2+}_{org}) = \frac{[H_2L^{2+}_{org}]}{[H^+_{org}]^2[L_{org}]} \quad (13)$$

$$\beta(ML^{3+}_{org}) = \frac{[ML^{3+}_{org}]}{[M^{3+}_{org}][L_{org}]} \quad (14)$$

can be evaluated applying the simple relations:

$$\log\beta(HL^+_{org}) = \log K_{ex}(HL^+_{org}) - \log K_D \quad (15)$$

$$\log\beta(H_2L^{2+}_{org}) = \log K_{ex}(H_2L^{2+}_{org}) - \log K_D \quad (16)$$

$$\log\beta(ML^{3+}_{org}) = \log K_{ex}(ML^{3+}_{org}) - \log K_{ex}(M^{3+}_{org}) - \log K_D \quad (17)$$

Similarly, the protonation constant of the complex cation $MH_{-1}L^{2+}$ ($M^{3+} = Eu^{3+}, Am^{3+}$; L = DDEG) in nitrobenzene saturated with water, i. e., the equilibrium constant of the following general reaction

$$MH_{-1}L^{2+}_{org} + H^+_{org} \Leftrightarrow ML^{3+}_{org} \quad (18)$$

defined as

$$K(ML^{3+}_{org}) = \frac{[ML^{3+}_{org}]}{[MH_{-1}L^{2+}_{org}][H^+_{org}]} \quad (19)$$

was determined on the basis of Relation (20):

$$\log K(ML^{3+}_{org}) = \log K_{ex}(ML^{3+}_{org}) - \log K_{ex}(MH_{-1}L^{2+}_{org}) \quad (20)$$

The respective equilibrium constants are summarized in Tables 3 and 4.

Moreover, Figure 3 depicts the contributions of the species H^+_{org}, HL^+_{org} and $H_2L^{2+}_{org}$ to the total hydrogen cation concentration in the equilibrium nitrobenzene phase, whereas Figures 4 and 5 show the contributions of the cations Eu^{3+}_{org}, EuL^{3+}_{org}, $EuH_{-1}L^{2+}_{org}$ and Am^{3+}_{org}, AmL^{3+}_{org}, $AmH_{-1}L^{2+}_{org}$,

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

Equilibrium	log K
$L_{aq} \Leftrightarrow L_{org}$	–3.0 [a]
$H^+_{org} + L_{aq} \Leftrightarrow HL^+_{org}$	4.12 [b]
$2H^+_{org} + L_{aq} \Leftrightarrow H_2L^{2+}_{org}$	7.67 [b]
$Eu^{3+}_{aq} + 3H^+_{org} \Leftrightarrow Eu^{3+}_{aq} + 3H^+_{aq}$	1.3 [c]
$Eu^{3+}_{aq} + L_{aq} + 3H^+_{org} \Leftrightarrow EuL^{3+}_{org} + 3H^+_{aq}$	10.23
$Eu^{3+}_{aq} + L_{aq} + 2H^+_{org} \Leftrightarrow EuH_{-1}L^{2+}_{org} + 3H^+_{aq}$	7.78
$H^+_{org} + L_{org} \Leftrightarrow HL^+_{org}$	7.12
$2H^+_{org} + L_{org} \Leftrightarrow H_2L^{2+}_{org}$	10.67
$Eu^{3+}_{org} + L_{org} \Leftrightarrow EuL^{3+}_{org}$	11.93
$EuH_{-1}L^{2+}_{org} + H^+_{org} \Leftrightarrow EuL^{3+}_{org}$	2.45

[a] Determined by the method of the concentration dependent distribution.[36]
[b] Determined by the method described in detail in Ref. 37.
[c] Ref. 38.

Table 4. Equilibrium constants in the water–HCl–DDEG–Am^{3+} (microamounts)–nitrobenzene –H^+B^- system.

Equilibrium	log K
$L_{aq} \Leftrightarrow L_{org}$	–3.0 [a]
$H^+_{org} + L_{aq} \Leftrightarrow HL^+_{org}$	4.12 [b]
$2H^+_{org} + L_{aq} \Leftrightarrow H_2L^{2+}_{org}$	7.67 [b]
$Am^{3+}_{aq} + 3H^+_{org} \Leftrightarrow Am^{3+}_{aq} + 3H^+_{aq}$	1.5 [c]
$Am^{3+}_{aq} + L_{aq} + 3H^+_{org} \Leftrightarrow AmL^{3+}_{org} + 3H^+_{aq}$	10.59
$Am^{3+}_{aq} + L_{aq} + 2H^+_{org} \Leftrightarrow AmH_{-1}L^{2+}_{org} + 3H^+_{aq}$	8.01
$H^+_{org} + L_{org} \Leftrightarrow HL^+_{org}$	7.12
$2H^+_{org} + L_{org} \Leftrightarrow H_2L^{2+}_{org}$	10.67
$Am^{3+}_{org} + L_{org} \Leftrightarrow AmL^{3+}_{org}$	12.09
$AmH_{-1}L^{2+}_{org} + H^+_{org} \Leftrightarrow AmL^{3+}_{org}$	2.58

[a] Determined by the method of the concentration dependent distribution.[36]
[b] Determined by the method described in detail in Ref. 37.
[c] Ref. 38.

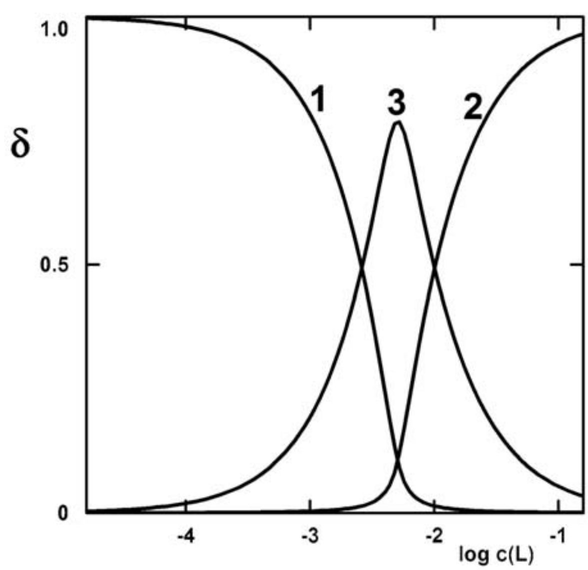

Figure 3. Distribution diagram of hydrogen cation in the equilibrium nitrobenzene phase of the water–HCl–DDEG–Eu^{3+}(microamounts)–nitrobenzene– H^+B^- extraction system in the forms of H^+, HL^+ and H_2L^{2+};
$c(HCl) = 0.05$ mol/L, $c_B = 0.01$ mol/L.
1 $\delta(H^+) = [H^+_{org}]/c(H^+)_{org}$,
2 $\delta(HL^+) = [HL^+_{org}]/c(H^+)_{org}$,
3 $\delta(H_2L^{2+}) = 2[H_2L^{2+}_{org}]/c(H^+)_{org}$,
where $c(H^+)_{org} = [HL^+_{org}] + 2[H_2L^{2+}_{org}]$
The distribution curves were calculated using the constants given in Table 3.

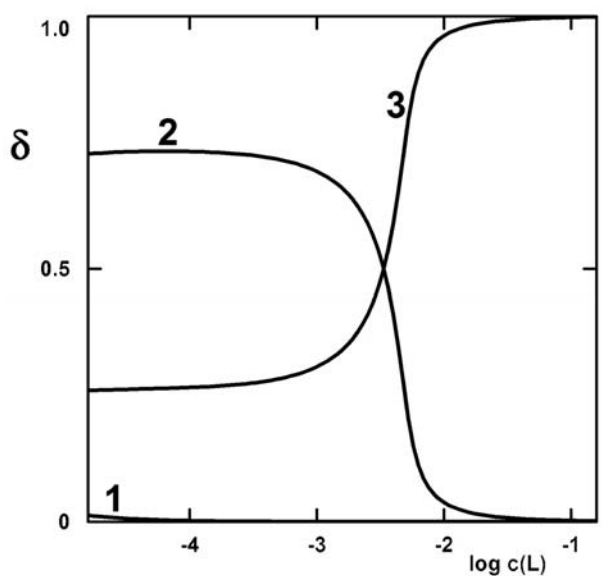

Figure 4. Distribution diagram of europium in the equilibrium nitrobenzene phase of the water–HCl–DDEG–Eu^{3+}(microamounts)–nitrobenzene–H^+B^- extraction system in the forms of Eu^{3+}, $EuL^{3+}$and $EuH_{-1}L^{2+}_{org}$;
$c(HCl) = 0.05$ mol/L, $c_B = 0.01$ mol/L.
1 $\delta(Eu^{3+}) = [Eu^{3+}_{org}]/c(Eu^{3+})_{org}$,
2 $\delta(EuL^{3+}) = [EuL^{3+}_{org}]/c(Eu^{3+})_{org}$,
3 $\delta(EuH_{-1}L^{2+}_{org}) = [EuH_{-1}L^{2+}_{org}]/c(Eu^{3+})_{org}$,
where $c(Eu^{3+})_{org} = [Eu^{3+}_{org}] + [EuL^{3+}_{org}] + [EuH_{-1}L^{2+}_{org}]$.
The distribution curves were calculated using the constants given in Table 3.

respectively, to the total trivalent metal cation concentration in the corresponding equilibrium organic phase.

Finally, it should be noted that the stability constants of the complex species EuL^{3+}_{org} and AmL^{3+}_{org}, where L is DDEG, in nitrobenzene saturated with water is log $\beta(EuL^{3+}_{org}) = 11.93$ and log $\beta(AmL^{3+}_{org}) = 12.09$, as given in Tables 3 and 4, respectively. Recently, the stability constants of the complexes CsL^+_{org}, CaL^{2+}_{org} and SrL^{2+}_{org} (L = DDEG) in water-saturated nitrobenzene were determined as log $\beta(CsL^+_{org}) = 6.83$,[25] log $\beta(CaL^{2+}_{org}) = 11.87$[26] and log $\beta(SrL^{2+}_{org}) = 13.19$.[26] Thus, in this nitrobenzene medium, the stability of the considered cationic complex species CsL^+_{org}, CaL^{2+}_{org}, SrL^{2+}_{org}, EuL^{3+}_{org} and AmL^{3+}_{org} increases in the series of $Cs^+ < Ca^{2+} \approx Eu^{3+} \approx Am^{3+} < Sr^{2+}$.

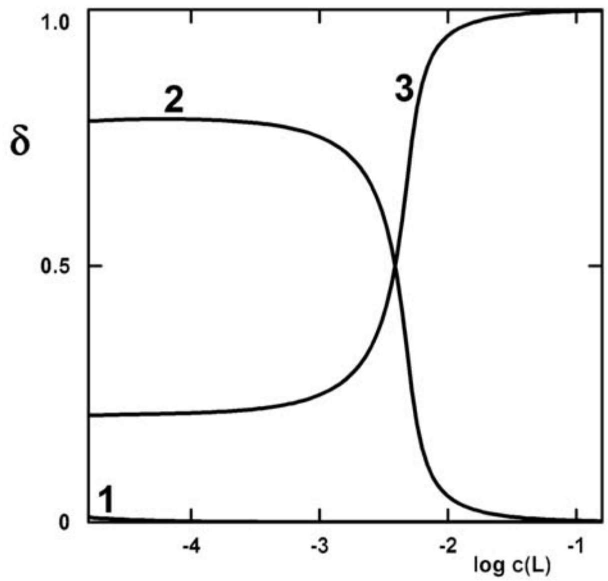

Figure 5. Distribution diagram of americium in the equilibrium nitrobenzene phase of the water–HCl–DDEG–Am^{3+}(microamounts)–nitrobenzene–H^+B^- extraction system in the forms of Am^{3+}, AmL^{3+} and $AmH_{-1}L^{2+}_{org}$;
c(HCl) = 0.05 mol/L, c_B = 0.01 mol/L.
1 $\delta(Am^{3+}) = [Am^{3+}_{org}]/c(Am^{3+})_{org}$,
2 $\delta(AmL^{3+}) = [AmL^{3+}_{org}]/c(Am^{3+})_{org}$
3 $\delta(AmH_{-1}L^{2+}_{org} = [AmH_{-1}L^{2+}_{org}]/c(Am^{3+})_{org}$,
where $c(Am^{3+})_{org} = [Am^{3+}_{org}] + [AmL^{3+}_{org}] + [AmH_{-1}L^{2+}_{org}]$.
The distribution curves were calculated using the constants given in Table 4.

4. Acknowledgements

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Povzetek

Proučevali smo ekstrakcijo mikrokoličin evropija in americija z raztopino hidrogen dikarbolilkobaltata (H^+B^-) v nitrobenzenu ob prisotnosti dodekaetilen glikola (DDEG, L). Ravnotežja smo pojasnili s pomočjo predpostavke, da se kompleksi HL^+, H_2L^{2+}, ML^{3+} in $MH_{-1}L^{2+}$ ($M^{3+} = Eu^{3+}$, Am^{3+}; L = DDEG) ekstrahirajo v organsko fazo. Določili smo konstante ekstrakcije in konstante stabilnosti kompleksov v nitrobenzenu, nasičenem z vodo. Ugotovili smo, da so konstante stabilnosti kompleksov evropija, EuL^{3+}, primerljive s konstantami stabilnosti kompleksov z americijem, AmL^{3+}.