

Short communication

Synergistic Extraction of Strontium into Nitrobenzene by Using Hydrogen Dicarbolylcobaltate and *N,N,N',N'*-Tetracyclohexyl-oxybis(o-phenyleneoxy) diacetamide

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Abstract

Extraction of microamounts of strontium by a nitrobenzene solution of hydrogen dicarbolylcobaltate (H^+B^-) in the presence of *N,N,N',N'*-tetracyclohexyl-oxybis(o-phenyleneoxy)diacetamide (abbrev. barium ionophore I, L) has been investigated. The equilibrium data have been explained assuming that the species HL^+ , SrL^{2+} and SrL_2^{2+} are extracted into the organic phase. The values of extraction and stability constants of the cationic complexes in nitrobenzene saturated with water have been determined.

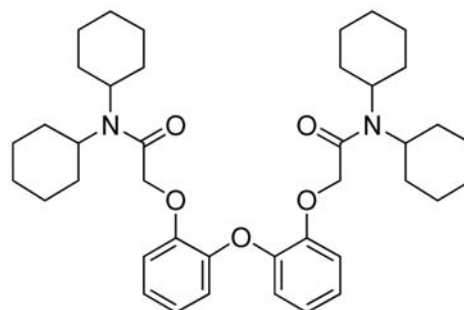
Keywords: Strontium, hydrogen dicarbolylcobaltate, barium ionophore I, water-nitrobenzene system, extraction and stability constants

1. Introduction

Electrically neutral, lipophilic organic complexing agents are widely used as components for ion-selective electrodes.^{1–3} Sensors for lithium,⁴ sodium,⁵ potassium,^{1,2,6–8} ammonium,^{9,10} calcium,¹¹ strontium¹² and barium^{13–16} are based on such molecules. Because of their analytical potential, considerable effort had been directed towards the design of selective and stable electrodes for barium.^{13–17}

The dicarbolylcobaltate anion¹⁸ 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,^{19–33} and on the technological scale for the separation of some high-activity isotopes in the reprocessing of spent nuclear fuel and acidic radioactive waste.^{34,35}

In the current work, the solvent extraction of microamounts of strontium by a nitrobenzene solution of hydrogen dicarbolylcobaltate (H^+B^-)¹⁸ in the presence of *N,N,N',N'*-tetracyclohexyl-oxybis(o-phenyleneoxy)diacetamide (abbrev. barium ionophore I, L) (see Scheme 1)



Scheme 1. Structural formula of *N,N,N',N'*-tetracyclohexyl-oxybis(o-phenyleneoxy)diacetamide (abbrev. barium ionophore I or L, respectively).

was studied. We intended to find the composition of the species in nitrobenzene phase and to determine the corresponding equilibrium constants.

2. Experimental

N,N,N',N'-Tetracyclohexyl-oxybis(o-phenyleneoxy)diacetamide, called also barium ionophore I (Scheme 1), was supplied by Fluka. Cesium dicarbollycobaltate, Cs^+B^- , was synthesized by the method published by Hawthorne et al.³⁶ A nitrobenzene solution of hydrogen dicarbollycobaltate (H^+B^-)¹⁸ was prepared from Cs^+B^- by the procedure described elsewhere.³⁷ The other chemicals used (Lachema, Brno, Czech Republic) were of reagent grade purity. The radionuclide $^{85}\text{Sr}^{2+}$ (DuPont, Belgium) was of standard radiochemical purity.

The extraction experiments in the two-phase water–HCl– Sr^{2+} (microamounts)–nitrobenzene–barium ionophore I– 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(Tl) scintillation detector connected to a γ -analyzer NK 350 (Gamma, Budapest, Hungary).

The equilibrium distribution ratios of strontium, D , were determined as the ratios of the measured radioactivities of $^{85}\text{Sr}^{2+}$ in the nitrobenzene and aqueous samples.

3. Results and Discussion

The dependence of the logarithm of the strontium distribution ratio ($\log D$) on the logarithm of the numerical value of the total (analytical) concentration of the barium ionophore I ligand in the initial nitrobenzene phase, $\log c(L)$, is given in Figure 1. The initial concentration of hydrogen dicarbollycobaltate in the organic phase, $c_B = 0.0002$ mol/L, as well as the initial concentration of HCl in the aqueous phase, $c(\text{HCl}) = 0.01$ mol/L, are always related to the volume of one phase.

With respect to the results of previous papers,^{18,27,29,31,38–48} the considered water–HCl– Sr^{2+} (microamounts)–nitrobenzene–barium ionophore I (L)– H^+B^- system can be described by the set of reactions

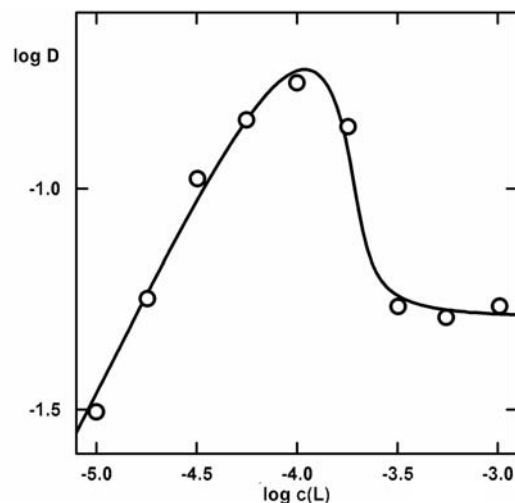
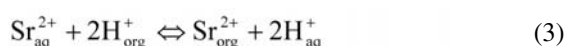
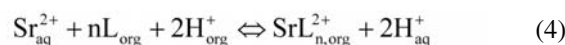


Figure 1. $\log D$ as a function of $\log c(L)$, where L is barium ionophore I, for the water–HCl– Sr^{2+} (microamounts)–nitrobenzene–barium ionophore I– H^+B^- system; $c(\text{HCl}) = 0.01$ mol/L, $c_B = 0.0002$ mol/L. The curve was calculated using the constants given in Table 2.



to which the following equilibrium constants correspond:

$$K_D = \frac{[\text{L}_{\text{org}}]}{[\text{L}_{\text{aq}}]} \quad (5)$$

$$\beta(\text{HL}^+_{\text{org}}) = \frac{[\text{HL}^+_{\text{org}}]}{[\text{H}^+_{\text{org}}][\text{L}_{\text{org}}]} \quad (6)$$

$$K_{\text{ex}}(\text{Sr}^{2+}_{\text{org}}) = \frac{[\text{Sr}^{2+}_{\text{org}}][\text{H}^+_{\text{aq}}]^2}{[\text{Sr}^{2+}_{\text{aq}}][\text{H}^+_{\text{org}}]^2} \quad (7)$$

$$K_{\text{ex}}(\text{SrL}^{2+}_{n,\text{org}}) = \frac{[\text{SrL}^{2+}_{n,\text{org}}][\text{H}^+_{\text{aq}}]^2}{[\text{Sr}^{2+}_{\text{aq}}][\text{L}_{\text{org}}]^n[\text{H}^+_{\text{org}}]^2} \quad (8)$$

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 barium ionophore I ligand and the electroneutrality conditions in both phases of the system under consideration, was formulated^{39,44} and introduced into a more general least-squares minimizing program LETAGROP⁴⁹ used for determination of the “best” values of the extraction constants $K_{\text{ex}}(\text{SrL}^{2+}_{n,\text{org}})$ (L = barium ionophore I). The minimum of the sum of errors in $\log D$, i.e., the minimum of the expression

$$U = \sum (\log D_{\text{calc}} - \log D_{\text{exp}})^2 \quad (9)$$

was sought.

The values $\log K_D = 3.6$ (see Table 2, footnote a),⁵¹ $\log \beta(\text{HL}^+_{\text{org}}) = 5.8$ ⁵¹ and $\log K_{\text{ex}}(\text{Sr}^{2+}_{\text{org}}) = 0.7$ ³⁹ 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 species SrL^{2+} and SrL_2^{2+} (L = barium ionophore I) to be extracted into the nitrobenzene phase.

Table 1. Comparison of three different models of strontium extraction from aqueous solution of HCl by nitrobenzene solution of H^+B^- in the presence of barium ionophore I.

Strontium complexes in the organic phase	$\log K_{\text{ex}}^a$	U^b
SrL^{2+}	9.18 (9.42)	0.47
SrL_2^{2+}	14.95 (15.50)	3.98
$\text{SrL}^{2+}, \text{SrL}_2^{2+}$	$9.04 \pm 0.06, 13.63 \pm 0.16$	0.01

^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 .⁴⁹ These values are given in the logarithmic scale using the approximate expression $\log K \pm \{\log [K + 1.5\sigma(K)] - \log [K - 1.5\sigma(K)]\}$. For $\sigma(K) > 0.2K$, the previous expression is not valid and then only the upper limit is given in the parentheses in the form of $\log K(\log [K + 3\sigma(K)])$.⁴⁹

^b The error-square sum $U = \sum(\log D_{\text{calc}} - \log D_{\text{exp}})^2$.

Knowing the values $\log K_{\text{ex}}(\text{Sr}_{\text{org}}^{2+}) = 0.7$,³⁹ as well as the extraction constants $\log K_{\text{ex}}(\text{SrL}_{\text{org}}^{2+}) = 9.04$ and $\log K_{\text{ex}}(\text{SrL}_{2,\text{org}}^{2+}) = 13.63$ determined here (see Table 1), the stability constants of the complexes SrL^{2+} and SrL_2^{2+} (L = barium ionophore I) in the nitrobenzene phase defined as

$$\beta(\text{SrL}_{\text{org}}^{2+}) = \frac{[\text{SrL}_{\text{org}}^{2+}]}{[\text{Sr}_{\text{org}}^{2+}][\text{L}_{\text{org}}]} \quad (10)$$

$$\beta(\text{SrL}_{2,\text{org}}^{2+}) = \frac{[\text{SrL}_{2,\text{org}}^{2+}]}{[\text{Sr}_{\text{org}}^{2+}][\text{L}_{\text{org}}]^2} \quad (11)$$

can be evaluated applying the following simple relations:

$$\log \beta(\text{SrL}_{\text{org}}^{2+}) = \log K_{\text{ex}}(\text{SrL}_{\text{org}}^{2+}) - \log K_{\text{ex}}(\text{Sr}_{\text{org}}^{2+}) \quad (12)$$

Table 2. Equilibrium constants in the water–HCl– Sr^{2+} (microamounts)–nitrobenzene–barium ionophore I – H^+B^- system.

Equilibrium	$\log K$
$\text{L}_{\text{aq}} \rightleftharpoons \text{L}_{\text{org}}$	3.6 ^a
$\text{H}_{\text{org}}^+ + \text{L}_{\text{org}} \rightleftharpoons \text{HL}_{\text{org}}^+$	5.8 ^b
$\text{Sr}_{\text{org}}^{2+} + 2\text{H}_{\text{org}}^+ \rightleftharpoons \text{Sr}_{\text{org}}^{2+} + 2\text{H}_{\text{org}}^+$	0.7 ^c
$\text{Sr}_{\text{aq}}^{2+} + \text{L}_{\text{org}} + 2\text{H}_{\text{org}}^+ \rightleftharpoons \text{SrL}_{\text{org}}^{2+} + 2\text{H}_{\text{aq}}^+$	9.04
$\text{Sr}_{\text{aq}}^{2+} + 2\text{L}_{\text{org}} + 2\text{H}_{\text{org}}^+ \rightleftharpoons \text{SrL}_{2,\text{org}}^{2+} + 2\text{H}_{\text{aq}}^+$	13.63
$\text{Sr}_{\text{org}}^{2+} + \text{L}_{\text{org}} \rightleftharpoons \text{SrL}_{\text{org}}^{2+}$	8.34
$\text{Sr}_{\text{org}}^{2+} + 2\text{L}_{\text{org}} \rightleftharpoons \text{SrL}_{2,\text{org}}^{2+}$	12.93

^a Determined by the method of the concentration dependent distribution.⁵⁰ ^b Ref. 51. ^c Ref. 39.

$$\log \beta(\text{SrL}_{2,\text{org}}^{2+}) = \log K_{\text{ex}}(\text{SrL}_{2,\text{org}}^{2+}) - \log K_{\text{ex}}(\text{Sr}_{\text{org}}^{2+}) \quad (13)$$

The respective equilibrium constants are summarized in Table 2.

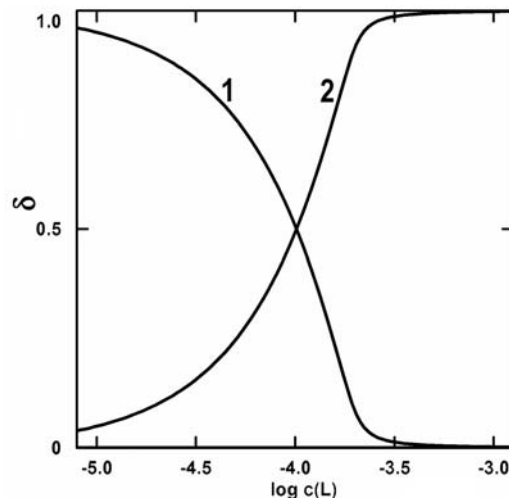


Figure 2. Distribution diagram of hydrogen cation in the equilibrium nitrobenzene phase of the water–HCl– Sr^{2+} (microamounts) –nitrobenzene– barium ionophore I – H^+B^- extraction system in the forms of H^+ and HL^+ ; $c(\text{HCl}) = 0.01$ mol/L, $c_{\text{B}} = 0.0002$ mol/L. $1 \delta(\text{H}^+) = [\text{H}_{\text{org}}^+]/c(\text{H}^+)_{\text{org}}$, $2 \delta(\text{HL}^+) = [\text{HL}_{\text{org}}^+]/c(\text{H}^+)_{\text{org}}$, where $c(\text{H}^+)_{\text{org}} = [\text{H}_{\text{org}}^+] + [\text{HL}_{\text{org}}^+]$. The distribution curves were calculated using the constants given in Table 2.

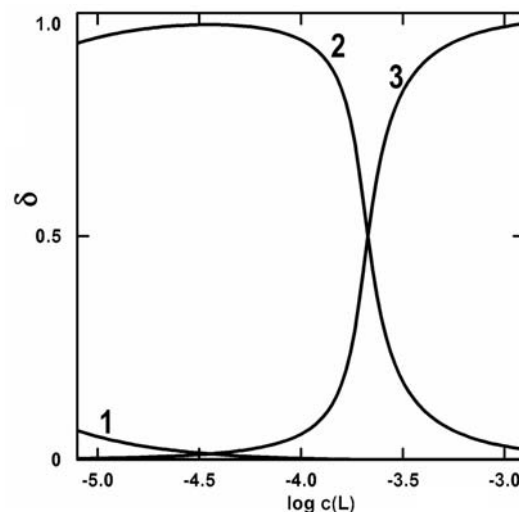


Figure 3. Distribution diagram of strontium in the equilibrium nitrobenzene phase of the water–HCl– Sr^{2+} (microamounts) –nitrobenzene– barium ionophore I – H^+B^- extraction system in the forms of Sr^{2+} , SrL^{2+} and SrL_2^{2+} ; $c(\text{HCl}) = 0.01$ mol/L, $c_{\text{B}} = 0.0002$ mol/L. $1 \delta(\text{Sr}^{2+}) = [\text{Sr}_{\text{org}}^{2+}]/c(\text{Sr}^{2+})_{\text{org}}$, $2 \delta(\text{SrL}^{2+}) = [\text{SrL}_{\text{org}}^{2+}]/c(\text{Sr}^{2+})_{\text{org}}$, $3 \delta(\text{SrL}_2^{2+}) = [\text{SrL}_{2,\text{org}}^{2+}]/c(\text{Sr}^{2+})_{\text{org}}$, where $c(\text{Sr}^{2+})_{\text{org}} = [\text{Sr}_{\text{org}}^{2+}] + [\text{SrL}_{\text{org}}^{2+}] + [\text{SrL}_{2,\text{org}}^{2+}]$. The distribution curves were calculated using the constants given in Table 2.

Moreover, Figure 2 depicts the contributions of the species H_{org}^+ and HL_{org}^+ to the total hydrogen cation concentration in the equilibrium nitrobenzene phase, whereas Figure 3 shows the contributions of the cations Sr_{org}^{2+} , SrL_{org}^{2+} and $SrL_{2,org}^{2+}$ to the total strontium concentration in the equilibrium organic phase. From Figure 3 it follows that the cationic complex species $SrL_{2,org}^{2+}$ is present in significant concentrations only at relatively high amounts of the barium ionophore I ligand in the system under consideration.

Finally, Table 3 presents the stability constants of the ML^+ complexes, where $M^+ = Li^+, Na^+, H_3O^+, NH_4^+, Ag^+, K^+, Rb^+, Tl^+, Cs^+$ and L is barium ionophore I, determined previously in water-saturated nitrobenzene.⁵¹ Thus, from the data given in Tables 2 and 3 it follows that in the mentioned nitrobenzene medium, the stability constants of the considered complexes ML^+ and SrL^{2+} (L = barium ionophore I) increase in the order: $Cs^+ < Rb^+ < NH_4^+, K^+ < H_3O^+ < Na^+ < Ag^+, Tl^+ < Li^+ < Sr^{2+}$.

Table 3. Stability constants of the complexes ML^+ ($M^+ = Li^+, Na^+, H_3O^+, NH_4^+, Ag^+, K^+, Rb^+, Tl^+, Cs^+$; L = barium ionophore I) in nitrobenzene saturated with water at 25 °C.

M^+	Li^+	Na^+	H_3O^+	NH_4^+	Ag^+	K^+	Rb^+	Tl^+	Cs^+
$\log \beta(ML_{org}^+)^a$	6.6	5.9	5.8	4.8	6.0	4.8	4.2	6.0	4.0

^a Ref. 51.

Table 4. Stability constants of the complexes SrL^{2+} and SrL_2^{2+} [L = diglyme, triglyme, tetraglyme, PEG 200, PEG 300, PEG 400, Slovafofol 909, 15-crown-5 (15C5), benzo-15-crown-5 (B15C5), 2,3-naphtho-15-crown-5 (N15C5), 18-crown-6 (18C6), benzo-18-crown-6 (B18C6), dibenzo-18-crown-6 (DB18C6), dicyclohexano-18-crown-6 (DCH18C6), dibenzo-21-crown-7 (DB21C7), dibenzo-24-crown-8 (DB24C8), dicyclohexano-24-crown-8 (DCH24C8), dibenzo-30-crown-10 (DB30C10), barium ionophore I] in nitrobenzene saturated with water at 25 °C.

L	$\log \beta(SrL_{org}^{2+})$	$\log \beta(SrL_{2,org}^{2+})$	Ref.
diglyme	3.06	–	38
triglyme	4.34	6.77	38
tetraglyme	4.90	7.52	38
PEG 200	9.06	–	39
PEG 300	10.41	–	39
PEG 400	11.03	–	39
Slovafofol 909	10.22	14.52	40
15C5	–	14.89	41
B15C5	–	13.20	42
N15C5	–	11.28	43
18C6	11.50	16.24	44
B18C6	9.29	13.68	27
DB18C6	6.38	8.94	29
DCH18C6	11.19	14.74	31
DB21C7	6.61	10.00	45
DB24C8	8.15	12.77	46
DCH24C8	9.99	14.44	47
DB30C10	9.28	–	48
barium ionophore I	8.34	12.93	This work

In conclusion, Table 4 summarizes the stability constants of the complexes SrL_{org}^{2+} and $SrL_{2,org}^{2+}$ with 19 oxethylene ligands L, denoted by the symbols diglyme, triglyme, tetraglyme, PEG 200, PEG 300, PEG 400, Slovafofol 909, 15C5, B15C5, N15C5, 18C6, B18C6, DB18C6, DCH18C6, DB21C7, DB24C8, DCH24C8, DB30C10 and barium ionophore I, in nitrobenzene saturated with water. From the data reviewed in this table it follows that in this nitrobenzene medium, the stability constants of the complexes SrL_{org}^{2+} increase in the series of diglyme < triglyme < tetraglyme < DB18C6 < DB21C7 < DB24C8 < barium ionophore I < PEG 200 < DB30C10 \approx B18C6 < DCH24C8 < Slovafofol 909 < PEG 300 < PEG 400 < DCH18C6 < 18C6, whereas the stability of the cationic complex species $SrL_{2,org}^{2+}$ increases in the following sequence: triglyme < tetraglyme < DB18C6 < DB21C7 < N15C5 < DB24C8 < barium ionophore I < B15C5 < B18C6 < DCH24C8 < Slovafofol 909 < DCH18C6 < 15C5 < 18C6.

4. Acknowledgements

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Povzetek

Raziskovali smo ekstrakcijo mikrokoličn stroncija v benzenovo raztopino hidrogen dikarbolilkobaltata (H^+B^-) v prisotnosti *N,N,N',N'*-tetracikloheksil-oksibis (o-phenilenoksi)diacetamida (barijev ionopfor I, L). Eksperimentalne podatke smo obravnavali s pomočjo modela, da se kompleksi HL^+ , SrL^{2+} in SrL_2^{2+} ekstrahirajo v organsko fazo. Ocenili smo koeficiente porazdelitve ter konstante stabilnosti kompleksov v nitrobenzenu, nasičenem z vodo.