

## Extraction of Microamounts of Barium in the System: Water – CsCl – 18-Crown-6 – Nitrobenzene – Cesium Dicarbolylcobaltate

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Extraction of microamounts of barium with a nitrobenzene solution of cesium dicarbolylcobaltate ( $\text{Cs}^+\text{B}^-$ ) in the presence of 18-crown-6 (18C6, L) has been investigated. Equilibrium data have been explained assuming that the complexes  $\text{CsL}^+$ ,  $\text{CsL}_2^+$ ,  $\text{BaL}^{2+}$  and  $\text{BaL}_2^{2+}$  are extracted into the organic phase. Values of extraction and stability constants of the species in nitrobenzene saturated with water have been determined.

*Key words:* barium, 18-crown-6, dicarbolylcobaltate, water – nitrobenzene extraction system.

### INTRODUCTION

In our previous works,<sup>1–4</sup> we have studied the extraction of  $\text{Sr}^{2+}$  and  $\text{Ba}^{2+}$  ions using a nitrobenzene solution of  $\text{H}^+$  dicarbolylcobaltate ( $\text{H}^+\text{B}^-$ ) in the presence of polyoxyethylene ligands. These ions ( $\text{M}^{2+}$ ) have been found to be extracted in the form of the  $\text{ML}_{n,\text{org}}^{2+}$  complexes. The occurrence of maxima on the plot of metal ion distribution ratio ( $D$ ) vs. the total polyoxyethylene compound concentration,  $c(\text{L})$ , has been explained in terms of competi-

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tion between the charged complexes  $ML_{n,org}^{2+}$  and  $HL_{org}^{+}$  during the balancing of the dicarbollylcobaltate electrostatic charge in the organic phase.

The aim of this paper was to study the extraction of microamounts of barium with the nitrobenzene solution of cesium dicarbollylcobaltate ( $Cs^{+}B^{-}$ ) in the presence of 18-crown-6 (18C6, L). We intended to find the composition of the species present in the organic phase and to determine their respective equilibrium constants.

## EXPERIMENTAL

18-crown-6 (Merck, Darmstadt, Germany) was used without purification. Cesium salt of dicarbollylcobaltate,  $Cs^{+}B^{-}$ , was synthesized in the Institute of Inorganic Chemistry, Řež, Czech Republic, using the method published by Hawthorne *et al.*<sup>5</sup> The other chemicals used (Lachema, Brno, Czech Republic) were of reagent grade purity. The radionuclide  $^{133}Ba$  (Polatom, Poland) was of standard radiochemical purity.

Extraction experiments in the system water –  $CsCl$  –  $Ba^{2+}$  (microamounts) – 18C6 – nitrobenzene –  $Cs^{+}B^{-}$  (water and nitrobenzene were not preequilibrated) 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 hours at  $25 \pm 2$  °C using a laboratory shaker. Under these conditions, an equilibrium in the system under study was established after approximately 30 min of shaking. Then, the phases were separated by centrifugation (5 min, 2500 rpm). After centrifugation, 1 ml samples were taken from each phase and their  $\gamma$ -activities were measured using a well-type NaI(Tl) scintillation detector connected with a single channel  $\gamma$ -analyzer NK 350 (Gamma, Budapest, Hungary).

The equilibrium distribution ratio of barium,  $D$ , was determined as a ratio of the measured radioactivities of  $^{133}Ba$  in the nitrobenzene and aqueous samples.

## RESULTS AND DISCUSSION

Dependences of the logarithm of the barium distribution ratios ( $\log D$ ) on the logarithm of the total (analytical) concentration of the ligand 18C6 in the initial aqueous phase,  $\log c(L)$ , were measured for three concentrations of cesium dicarbollylcobaltate in the nitrobenzene phase ( $c_B = 0.01, 0.03$  and  $0.05 \text{ mol dm}^{-3}$ ) in the presence of 18C6 [ $c(L) = 5.6 \times 10^{-4}$  to  $0.10 \text{ mol dm}^{-3}$ ]; the initial concentration of cesium chloride in the aqueous phase,  $c(CsCl)$ , was  $0.05 \text{ mol dm}^{-3}$ . The mentioned initial concentrations  $c(CsCl)$ ,  $c_B$  and  $c(L)$  are always related to the volume of one phase. The results are given in Table I and Figure 1.

Dependences of  $\log D$  vs.  $\log c(L)$  with characteristic maxima (see Figure 1) can be explained – analogously to previous papers<sup>1–4</sup> – by the competition between charged complexes of barium and cesium during balancing of the dicarbollylcobaltate electrostatic charge in the nitrobenzene phase.

TABLE I

Log  $D$  as a function of  $\log c(L)^a$  for barium extraction from aqueous solution of cesium chloride<sup>b</sup> by nitrobenzene solutions of  $Cs^+B^-$

$c_B = 0.01 \text{ mol dm}^{-3}$		$c_B = 0.03 \text{ mol dm}^{-3}$		$c_B = 0.05 \text{ mol dm}^{-3}$	
$\log c(L)$	$\log D$	$\log c(L)$	$\log D$	$\log c(L)$	$\log D$
-3.252	-1.919	-3.252	-1.289	-2.495	-0.207
-3.000	-1.702	-3.000	-1.076	-2.252	0.102
-2.699	-1.350	-2.699	-0.652	-2.000	0.308
-2.495	-1.188	-2.495	-0.448	-1.699	0.479
-2.252	-0.998	-2.252	-0.170	-1.495	0.507
-2.000	-2.464	-2.000	-0.021	-1.252	-1.763
-1.699	-3.279	-1.699	-0.039	-1.000	-2.931
		-1.495	-2.030		
		-1.252	-3.228		

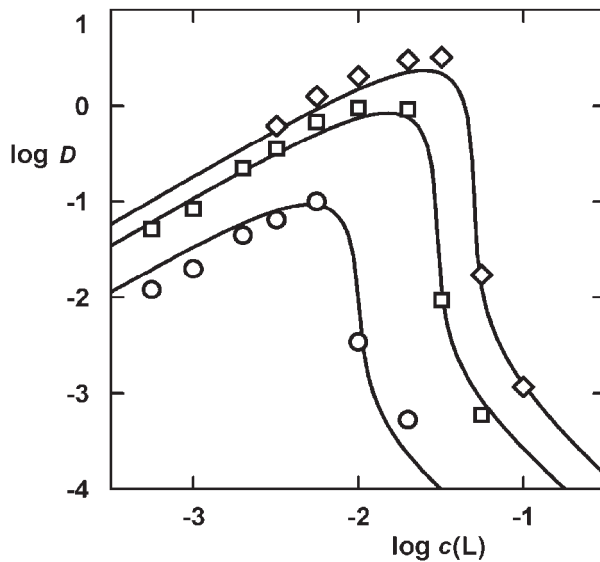
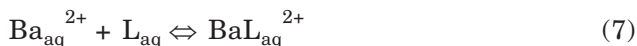
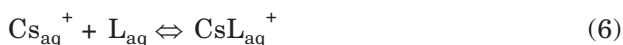
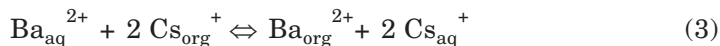
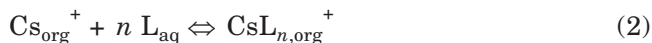


Figure 1. Log  $D$  as a function of  $\log c(L)$  for the system: water – CsCl –  $Ba^{2+}$  (microamounts) – 18C6 – nitrobenzene –  $Cs^+B^-$ , where  $c(CsCl) = 0.05 \text{ mol dm}^{-3}$ .  
 $\circ c_B = 0.01 \text{ mol dm}^{-3}$ ;  $\square c_B = 0.03 \text{ mol dm}^{-3}$ ;  $\diamond c_B = 0.05 \text{ mol dm}^{-3}$ . The curves were calculated using the constants given in Table III.

With regard to the results of previous papers,<sup>1-4</sup> the system water – CsCl – Ba<sup>2+</sup> (microamounts) – 18C6 – nitrobenzene – Cs<sup>+</sup>B<sup>-</sup> can be described by the set of reactions



with the following equilibrium constants:

$$K_D(L) = \frac{[L_{\text{org}}]}{[L_{\text{aq}}]} \quad (8)$$

$$K_{\text{ex}}(CsL_{n,\text{org}}^+) = \frac{[CsL_{n,\text{org}}^+]}{[Cs_{\text{org}}^+][L_{\text{aq}}]^n} \quad (9)$$

$$K_{\text{ex}}(Ba_{\text{org}}^{2+}) = \frac{[Ba_{\text{org}}^{2+}][Cs_{\text{aq}}^+]^2}{[Ba_{\text{aq}}^{2+}][Cs_{\text{org}}^+]^2} \quad (10)$$

$$K_{\text{ex}}(BaL_{\text{org}}^{2+}) = \frac{[BaL_{\text{org}}^{2+}][Cs_{\text{aq}}^+]^2}{[Ba_{\text{aq}}^{2+}][L_{\text{aq}}][Cs_{\text{org}}^+]^2} \quad (11)$$

$$K_{\text{ex}}(BaL_{2,\text{org}}^{2+}) = \frac{[BaL_{2,\text{org}}^{2+}][Cs_{\text{aq}}^+]^2}{[Ba_{\text{aq}}^{2+}][L_{\text{aq}}]^2[Cs_{\text{org}}^+]^2} \quad (12)$$

$$\beta(\text{CsL}_{\text{aq}}^+) = \frac{[\text{CsL}_{\text{aq}}^+]}{[\text{Cs}_{\text{aq}}^+][\text{L}_{\text{aq}}]} \quad (13)$$

$$\beta(\text{BaL}_{\text{aq}}^{2+}) = \frac{[\text{BaL}_{\text{aq}}^{2+}]}{[\text{Ba}_{\text{aq}}^{2+}][\text{L}_{\text{aq}}]} \quad (14)$$

Subscripts (aq) and (org) denote the aqueous and organic phases, respectively.

A subroutine UBBE, based on the relations given above, mass balance of the crown ligand and the electroneutrality conditions of both phases, was formulated<sup>1,4</sup> and introduced into a more general least-squares minimizing program LETAGROP<sup>6</sup> used for determination of the »best« values of the constants  $K_{\text{ex}}(\text{CsL}_{n,\text{org}}^+)$ . The minimum of the sum of errors in  $\log D$ , *i.e.*, the minimum of the expression

$$U = \sum (\log D_{\text{calc}} - \log D_{\text{exp}})^2 \quad (15)$$

was sought.

Values  $K_{\text{D}}(\text{L}) = 0.10$ ,<sup>7</sup>  $\log K_{\text{ex}}(\text{Ba}_{\text{org}}^{2+}) = -5.1$  (inferred from Refs. 8 and 9),  $\log K_{\text{ex}}(\text{BaL}_{\text{org}}^{2+}) = 6.37$  (inferred from Refs. 4 and 7–9),  $\log K_{\text{ex}}(\text{BaL}_{2,\text{org}}^{2+}) = 10.68$  (inferred from Refs. 4 and 7–9),  $\log \beta(\text{CsL}_{\text{aq}}^+) = 0.99$  (Ref. 10) and  $\log \beta(\text{BaL}_{\text{aq}}^{2+}) = 3.87$  (Ref. 10) were used for the respective calculations. The re-

TABLE II

Comparison of three different models of barium extraction from aqueous solution of cesium chloride by nitrobenzene solutions of  $\text{Cs}^+\text{B}^-$  in the presence of 18-crown-6

Cesium complexes in the organic phase	$\log K_{\text{ex}}^{\text{a}}$	$U^{\text{b}}$
$\text{CsL}^+$	$5.53 \pm 0.11$	1.21
$\text{CsL}_2^+$	10.67 (10.90)	19.15
$\text{CsL}^+, \text{CsL}_2^+$	$5.53 \pm 0.11, 6.78 (7.35)$	1.10

<sup>a</sup> The values of 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 constant  $K$ .<sup>6</sup> These values are expressed on the logarithmic scale using the approximate relation  $\log K \pm \{\log [K + 1.5 \sigma(K)] - \log [K - 1.5 \sigma(K)]\}$ . For  $\sigma(K) > 0.2K$ , the previous relation is not valid and then only the upper limit is given in the parentheses in the form of  $\log K (\log [K + 3 \sigma(K)])$ .<sup>6</sup>

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

sults are listed in Table II, from which it is evident that the extraction data can be explained assuming the cesium complex species  $\text{CsL}^+$  and  $\text{CsL}_2^+$  to be extracted into the nitrobenzene phase.

Figure 2 presents the contributions of the species  $\text{Cs}_{\text{org}}^+$ ,  $\text{CsL}_{\text{org}}^+$  and  $\text{CsL}_{2,\text{org}}^+$  to the total cesium concentration in the equilibrium nitrobenzene phase while Figure 3 depicts the contributions of particles  $\text{BaL}_{\text{org}}^{2+}$  and  $\text{BaL}_{2,\text{org}}^{2+}$  to the total barium concentration in the equilibrium organic phase. It follows from both of these figures that the »sandwich« type complexes  $\text{CsL}_{2,\text{org}}^+$  and  $\text{BaL}_{2,\text{org}}^{2+}$  are present in significant concentrations only at relatively high amounts of the 18C6 ligand in the system under study. Moreover, it is noteworthy that practically only the cesium species  $\text{CsL}^+$  exists in the system under consideration and at the same time,  $[\text{BaL}_{\text{org}}^{2+}] \approx [\text{BaL}_{2,\text{org}}^{2+}]$ , if  $\log c(\text{L}) \approx -1.5$  (see Figures 2 and 3).

Knowing the value  $K_{\text{D}}(\text{L}) = 0.10$ ,<sup>7</sup> and the extraction constants  $\log K_{\text{ex}}(\text{CsL}_{\text{org}}^+) = 5.53$  and  $\log K_{\text{ex}}(\text{CsL}_{2,\text{org}}^+) = 6.78$  determined here, the stability constants of the complexes  $\text{CsL}^+$  and  $\text{CsL}_2^+$  in the nitrobenzene phase defined as

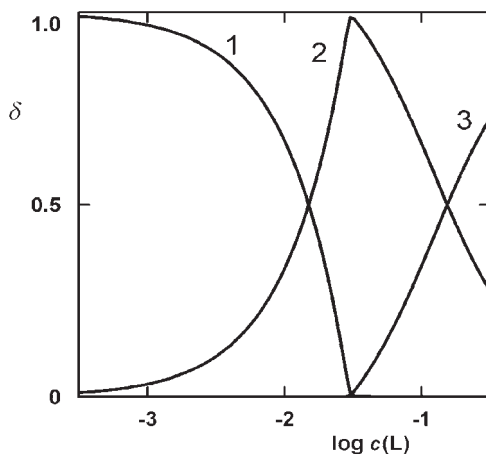


Figure 2. Distribution diagram of cesium present in the equilibrium nitrobenzene phase of the system: water –  $\text{CsCl}$  –  $\text{Ba}^{2+}$  (microamounts) – 18C6 – nitrobenzene –  $\text{Cs}^+\text{B}^-$  in the forms of  $\text{Cs}^+$ ,  $\text{CsL}^+$  and  $\text{CsL}_2^+$ .  $c(\text{CsCl}) = 0.05 \text{ mol dm}^{-3}$ ,  $c_{\text{B}} = 0.03 \text{ mol dm}^{-3}$ . 1)  $\delta(\text{Cs}^+) = [\text{Cs}_{\text{org}}^+] / c(\text{Cs}^+)_{\text{org}}$ , 2)  $\delta(\text{CsL}^+) = [\text{CsL}_{\text{org}}^+] / c(\text{Cs}^+)_{\text{org}}$ , 3)  $\delta(\text{CsL}_2^+) = [\text{CsL}_{2,\text{org}}^+] / c(\text{Cs}^+)_{\text{org}}$ , where  $c(\text{Cs}^+)_{\text{org}} = [\text{Cs}_{\text{org}}^+] + [\text{CsL}_{\text{org}}^+] + [\text{CsL}_{2,\text{org}}^+]$ . The distribution curves were calculated using the constants  $\log K_{\text{D}}(\text{L}) = -1.0$ ,  $\log \beta(\text{CsL}_{\text{aq}}^+) = 0.99$ ,  $\log K_{\text{ex}}(\text{CsL}_{\text{org}}^+) = 5.53$  and  $\log K_{\text{ex}}(\text{CsL}_{2,\text{org}}^+) = 6.78$ .

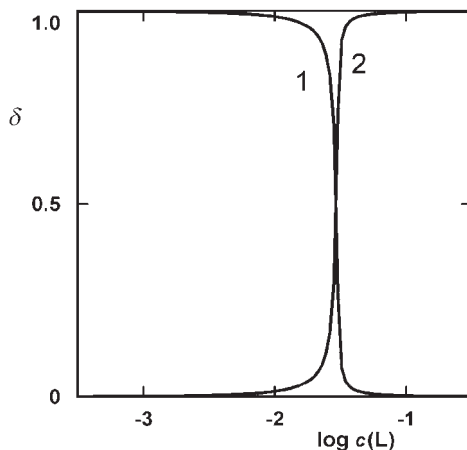


Figure 3. Distribution diagram of barium present in the equilibrium nitrobenzene phase of the system: water – CsCl – Ba<sup>2+</sup> (microamounts) – 18C6 – nitrobenzene – Cs<sup>+</sup>B<sup>-</sup> in the forms of BaL<sup>2+</sup> and BaL<sub>2</sub><sup>2+</sup>.  $c(\text{CsCl}) = 0.05 \text{ mol dm}^{-3}$ ,  $c_{\text{B}} = 0.03 \text{ mol dm}^{-3}$ . 1)  $\delta(\text{BaL}^{2+}) = [\text{BaL}_{\text{org}}^{2+}] / c(\text{Ba}^{2+})_{\text{org}}$ , 2)  $\delta(\text{BaL}_2^{2+}) = [\text{BaL}_{2,\text{org}}^{2+}] / c(\text{Ba}^{2+})_{\text{org}}$ , where  $c(\text{Ba}^{2+})_{\text{org}} = [\text{Ba}_{\text{org}}^{2+}] + [\text{BaL}_{\text{org}}^{2+}] + [\text{BaL}_{2,\text{org}}^{2+}]$ . The distribution curves were calculated using the constants given in Table III.

$$\beta(\text{CsL}_{\text{org}}^+) = \frac{[\text{CsL}_{\text{org}}^+]}{[\text{Cs}_{\text{org}}^+][\text{L}_{\text{org}}]} \quad (16)$$

$$\beta(\text{CsL}_{2,\text{org}}^+) = \frac{[\text{CsL}_{2,\text{org}}^+]}{[\text{Cs}_{\text{org}}^+][\text{L}_{\text{org}}]^2} \quad (17)$$

can be evaluated using the simple relations:

$$\log \beta(\text{CsL}_{\text{org}}^+) = \log K_{\text{ex}}(\text{CsL}_{\text{org}}^+) - \log K_{\text{D}}(\text{L}) \quad (18)$$

$$\log \beta(\text{CsL}_{2,\text{org}}^+) = \log K_{\text{ex}}(\text{CsL}_{2,\text{org}}^+) - 2 \log K_{\text{D}}(\text{L}) \quad (19)$$

Finally, the individual extraction constants of cations CsL<sup>+</sup> and BaL<sup>2+</sup>, denoted  $K_i(\text{CsL}^+)$  and  $K_i(\text{BaL}^{2+})$ , respectively, in the water – nitrobenzene system corresponding to the transfer of these complex cations from the aqueous into the nitrobenzene phase <sup>8</sup>



TABLE III

The equilibrium constants for the system: water – CsCl – Ba<sup>2+</sup>  
(microamounts) – 18C6 – nitrobenzene – Cs<sup>+</sup>B<sup>-</sup>

Equilibrium	log <i>K</i>
$L_{\text{aq}} \Leftrightarrow L_{\text{org}}$	-1.0 <sup>a</sup>
$\text{Cs}_{\text{aq}}^+ + L_{\text{aq}} \Leftrightarrow \text{CsL}_{\text{aq}}^+$	0.99 <sup>b</sup>
$\text{Ba}_{\text{aq}}^{2+} + L_{\text{aq}} \Leftrightarrow \text{BaL}_{\text{aq}}^{2+}$	3.87 <sup>b</sup>
$\text{Cs}_{\text{org}}^+ + L_{\text{aq}} \Leftrightarrow \text{CsL}_{\text{org}}^+$	5.53
$\text{Cs}_{\text{org}}^+ + 2L_{\text{aq}} \Leftrightarrow \text{CsL}_{2,\text{org}}^+$	6.78
$\text{Ba}_{\text{aq}}^{2+} + 2\text{Cs}_{\text{org}}^+ \Leftrightarrow \text{Ba}_{\text{org}}^{2+} + 2\text{Cs}_{\text{aq}}^+$	-5.1 <sup>c</sup>
$\text{Ba}_{\text{aq}}^{2+} + L_{\text{aq}} + 2\text{Cs}_{\text{org}}^+ \Leftrightarrow \text{BaL}_{\text{org}}^{2+} + 2\text{Cs}_{\text{aq}}^+$	6.37 <sup>d</sup>
$\text{Ba}_{\text{aq}}^{2+} + 2L_{\text{aq}} + 2\text{Cs}_{\text{org}}^+ \Leftrightarrow \text{BaL}_{2,\text{org}}^{2+} + 2\text{Cs}_{\text{aq}}^+$	10.68 <sup>d</sup>
$\text{Cs}_{\text{org}}^+ + L_{\text{org}} \Leftrightarrow \text{CsL}_{\text{org}}^+$	6.53
$\text{Cs}_{\text{org}}^+ + 2L_{\text{org}} \Leftrightarrow \text{CsL}_{2,\text{org}}^+$	8.78
$\text{Ba}_{\text{org}}^{2+} + L_{\text{org}} \Leftrightarrow \text{BaL}_{\text{org}}^{2+}$	12.47 <sup>e</sup>
$\text{Ba}_{\text{org}}^{2+} + 2L_{\text{org}} \Leftrightarrow \text{BaL}_{2,\text{org}}^{2+}$	17.78 <sup>e</sup>
$\text{CsL}_{\text{aq}}^+ \Leftrightarrow \text{CsL}_{\text{org}}^+$	1.8
$\text{BaL}_{\text{aq}}^{2+} \Leftrightarrow \text{BaL}_{\text{org}}^{2+}$	-2.9

<sup>a</sup> Ref. 7.

<sup>b</sup> Ref. 10.

<sup>c</sup> Inferred from Refs. 8, 9.

<sup>d</sup> Inferred from Refs. 4 and 7–9.

<sup>e</sup> Ref. 4.



were evaluated using the following relationships

$$\log K_i(\text{CsL}^+) = \log K_{\text{ex}}(\text{CsL}_{\text{org}}^+) - \log \beta(\text{CsL}_{\text{aq}}^+) + \log K_i(\text{Cs}^+) \quad (22)$$

$$\log K_i(\text{BaL}^{2+}) = \log K_{\text{ex}}(\text{BaL}_{\text{org}}^{2+}) - \log \beta(\text{BaL}_{\text{aq}}^{2+}) + 2 \log K_i(\text{Cs}^+) \quad (23)$$

where  $\log \beta(\text{CsL}_{\text{aq}}^+) = 0.99$ ,<sup>10</sup>  $\log \beta(\text{BaL}_{\text{aq}}^{2+}) = 3.87$ <sup>10</sup> and  $\log K_i(\text{Cs}^+) = -2.7$ .<sup>8</sup> The respective equilibrium constants are summarized in Table III.

In conclusion, it should be noted that the stability constants of the complexes CsL<sup>+</sup> and CsL<sub>2</sub><sup>+</sup> (L = 18-crown-6) in water saturated nitrobenzene, evaluated in the water – HNO<sub>3</sub> – Cs<sup>+</sup> (microamounts) – 18C6 – nitrobenzene



–  $H^+B^-$  system, are  $\log \beta(CsL_{org}^+) = 6.54$ ,<sup>11</sup> and  $\log \beta(CsL_{2,org}^+) = 8.64$ ,<sup>11</sup> which are in very good agreement with the values  $\log \beta(CsL_{org}^+) = 6.53$  and  $\log \beta(CsL_{2,org}^+) = 8.78$  determined in this work (see also Table III). This fact confirms our results quite unambiguously.

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## SAŽETAK

### **Ekstrakcija mikrokoličina barija u sustavu: voda – CsCl – 18-kruna-6 – nitrobenzen – cezijev dikarbolilkobaltat**

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Ekstrakcija mikrokoličina barija otopinom cezijeva dikarbolilkobaltata ( $Cs^+B^-$ ) u nitrobenzenu proučavana je u prisutnosti 18-kruna-6 (18C6, L). Pretpostavljajući da se u organsku fazu ekstrahiraju kompleksi  $CsL^+$ ,  $CsL_2^+$ ,  $BaL^{2+}$  i  $BaL_2^{2+}$  objašnjeni su podatci o ravnoteži. Određene su konstante stabilnosti specija ekstrahiranih u nitrobenzen zasićen vodom.