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Extraction of Rare-Earth Elements(III) from Nitric Acid Solutions with Diethyl 2-[(Diphenylphosphoryl)methoxy]-5-ethylphenylphosphonate

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Abstract—Extraction of micro amounts of rare earth elements(III) (REE) from nitric acid solutions with diethyl 2-[(diphenylphosphoryl)methoxy]-5-ethylphenylphosphonate (I) in dichloroethane and ionic liquid, 1-butyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imide has been studied. The stoichiometry of extracted complexes has been determined, the effect of HNO_3 concentration in aqueous phase on the efficiency of metal ion recovery into organic phase has been considered. It has been found that compound **I** in dichloroethane extracts REE(III) less efficiently than its analogs with phenyl and butyl substituents at the phosphorus atom. It has been shown that the efficiency of REE(III) extraction with compound **I** from nitric acid solutions increases considerably in the presence of ionic liquid in organic phase.

Keywords: extraction, rare earth elements(III), organophosphorus compounds, ionic liquids

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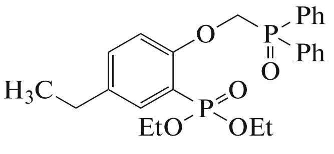
Extraction methods are widely used for the preconcentration and separation of rare earth elements (REE) [1]. The study of extraction properties of new polyfunctional organophosphorus compounds is of interest due to the promise of their use in radiochemical and analytical practice. Polydentate neutral organophosphorus compounds show high extraction ability toward REE(III) [2, 3], the most studied compounds among them are methylenediphosphine dioxides [4], (dialkylcarbamoylmethyl)diarylphosphine oxides (CMPO) [5] and their derivatives [6], *N*-diphenylphosphoryl-*N*'-alkylureas [7], phosphorylated calixarenes [8–12], phosphorylated pyridine N-oxides [13, 14], as well as acyclic analogs of crown ethers: podands with amide [15–20] or phosphoryl [21–23] terminal groups.

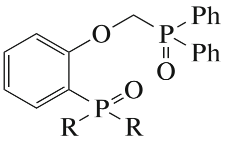
The extraction ability and selectivity of phosphoryl-containing podands (PP) is considerably affected by the length of polyether chain, the structure of bridge between the ethereal oxygen atom and the PO group, and the nature of substituent at the phosphorus atom [21–23]. In certain cases, the replacement of alkylene bridge in the molecules of tetraphenylalkylenediphosphine dioxides by ethylene glycol fragments leads to increase in the extraction ability of the corresponding PP toward REE(III) [23]. Among these compounds, tetraaryl-substituted (*o*-phenyleneoxymethylene)diphosphine dioxides show the highest extraction ability toward actinide and REE(III) ions [23], which is superior to that for CMPO. The replacement of aryl substituents at the phosphorus atom in these compounds by alkyl ones leads to the sharp drop of extraction ability of PP toward actinides and REE in nitric acid solutions due to "anomalous aryl strengthening" (AAS) effect for extracted complexes [24]. Extraction properties of PP with alkoxy groups at the phosphorus atom were studied in much less extent [25].

In recent time, there is growing interest in the use of ionic liquids (ILs) in the processes of extraction preconcentration and separation of organic and inorganic compounds [26–29]. REE(III) extraction with bidentate neutral organophosphorus compounds [30–32] and substituted diglycolamides [33–35] considerably increases in the presence of ILs, 1-alkyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imides. In these systems, the quantitative recovery of REE(III) requires no considerable excess of NO_3^- ions in aqueous phase, which is necessary condition for extraction with these extractants in common organic solvents. There are no literature data on the effect of IL on metal ion extraction with PP.

The aim of this work is to study effect of IL on the efficiency of REE(III) extraction from nitric acid solutions with solutions of compound **I**. Therefore, we considered certain features of REE(III) distribution between aqueous HNO_3 solutions and solutions of compound **I** in 1,2-dichloroethane and IL, 1-butyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imide. We compared the data on REE(III) extraction with compound **I** and its structural analogs with phenyl (**II**) and butyl (**III**) substituents at the phosphoryl group.



I



II–III

II, R = Ph; **III**, R = Bu

EXPERIMENTAL

Diethyl 2-[(diphenylphosphoryl)methoxy]-5-ethylphenylphosphonate (I) was obtained by alkylation of diethyl (2-hydroxy-5-ethylphenyl)phosphonate with (diphenylphosphoryl)methyl benzenesulfonate in the presence of sodium hydride in boiling dioxane by the previously described procedure [36]. Yield of compound **I** was 69%, mp = 89–91°C (benzene–hexane).

For $C_{25}H_{30}O_5P_2$ anal. calcd. (%): C, 63.56 H, 6.40; P, 16.93.

Found (%): C, 63.49, 63.15; H, 6.05, 6.32; P, 16.41, 16.67.

^{1}H NMR ($CDCl_3$, δ, ppm): 2.18 m (9H, $2C\underline{H}_3CH_2O$ + Ar–$CH_2C\underline{H}_3$), 3.65 q (2H, Ar–$C\underline{H}_2CH_3$), 5.01 m (4H, $2CH_3C\underline{H}_2O$), 5.82 d (2H, $^2J_{H-P}$ = 16 Hz, $OC\underline{H}_2P(O)Ph_2$), 7.96 t (1H, $^3J_{H-H}$ = 7.3 Hz, Ar–H), 8.30 t (1H, $^3J_{H-H}$ = 4.6 Hz, Ar–H), 8.52 m (6H, Ar–H), 8.76 d (1H, $^2J_{H-P}$ = 32 Hz, P(O)Ar–H), 9.04 m (4H, Ar–H). ^{31}P NMR ($CDCl_3$, δ, ppm): 18.55, 27.96.

Organic diluents used were 1,2-dichloroetane of reagent grade without additional purification and 1-butyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imide (C_4mimTf_2N), prepared and purified by the known procedure [37]. Extractant solutions were prepared from precisely weighed sample.

Initial REE(III) aqueous solutions were prepared by dissolution of the corresponding nitrates in water followed by addition of HNO_3. Initial metal ions concentration was 2×10^{-6} mol/L. Phases were contacted at ambient temperature on a rotary stirring apparatus with stirring rate 60 rpm for 1 h. It was preliminary established that this time is sufficient to reach constant values of distribution ratios (D_{Ln}) in extraction systems.

REE(III) concentration in initial and equilibrium aqueous solutions was determined by inductively coupled plasma mass spectrometry (ICP-MS) using a Thermo Elemental X-7 mass spectrometer (USA). Element content in organic phase was determined after back extraction with 0.1 M solution of hydroxyethylidenediphosphonic acid. Distribution ratio of elements was calculated as the ratio of their concentrations in organic and aqueous phases. Determination error for distribution ratio was not larger than 5%.

The concentration of Tf_2N^- ions in equilibrium aqueous phases was determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) using a Thermo Jarrel Ash ICAP-61 spectrometer (USA).

The concentration of HNO_3 in equilibrium aqueous phase was determined by potentiometric titration with NaOH solution, that in organic phase was determined by the same method after back extraction of HNO_3 with water. The content of HNO_3 in organic phase was determined by acid extraction with pure dichloroethane (blank test). The result of blank test was taken into account in computation of total concentration of HNO_3 complexes with extractant in organic phase.

RESULTS AND DISCUSSION

The process of metal ion extraction from nitric acid solutions with neutral organophosphorus compounds is accompanied by HNO_3 reaction with extractant. Therefore, we preliminary studied HNO_3 extraction with solution of compound **I**. The data on HNO_3 distribution between its aqueous solutions and compound **I** solution in dichloroethane (Fig. 1) indicate that at $[HNO_3] > 2.5$ mol/L, the ratio of HNO_3 concentration bound in complexes with the extractant to initial extractant concentration in organic phase is larger 1. Assuming that extractant (L) complexes with one or two nitric acid molecules pass into organic phase, the process of HNO_3 extraction can be described by the equation:

$$i\left(H^+ + NO_3^-\right) + L_{(o)} \rightleftarrows (HNO_3)_i\,L_{(o)}, \qquad (1)$$

where symbols (o) indicate the components of organic phase, i = 1 and 2 are the number of HNO_3 molecules in the extracted solvate. Efficient extraction constants for HNO_3 (K_1 and K_2) are expressed as

$$K_i = \left[(HNO_3)_i\,L\right] \big/ \left([L]a^i\right), \qquad (2)$$

where a is HNO_3 activity in equilibrium aqueous phase ($a = [H^+][NO_3^-]\gamma_\pm^2$), [L] is equilibrium concentration of free extractant in organic phase. The values

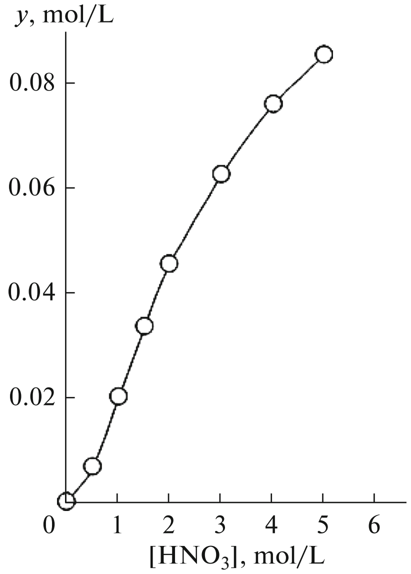


Fig. 1. Extraction of nitric acid with 0.05 M solution of compound **I** in dichloroethane.

of K_1 and K_2 calculated by non-linear least squares using equation:

$$y/[\mathrm{L}]_{\mathrm{ini}} = \left(K_1a + 2K_2a^2\right)/\left(1 + K_1a + K_2a^2\right), \quad (3)$$

where y is the total concentration of HNO_3 complexes with extractant in organic phase, $[\mathrm{L}]_{\mathrm{ini}}$ is the initial extractant concentration, are shown in Table 1. For comparison, the table also displays K_1 and K_2 values for compounds **II** and **III**. Increase in the extraction ability toward HNO_3 in the series of compounds **I** < **II** < **III** is due to increase in the donor ability of the P=O group as the electronegativity of the substituents at the phosphorus atom decreases [38].

We considered the effect of HNO_3 concentration in equilibrium aqueous phase on the alteration of distribution ratios of REE(III) upon extraction with solution of compound **I** in dichloroethane (Fig. 2). Extraction of Ho(III)–Lu(III) increases with $[HNO_3]$, while the extraction of La(III)–Tb(III) shows $\log D_{\mathrm{Ln}}$–$[HNO_3]$ dependences with maximum, which is owing to the salting out effect of NO_3^- ions and extractant binding with nitric acid, as well as change in the activity coefficients of REE(III) nitrates depending on $[HNO_3]$. The position of maximum is shifted to the region of higher acidity of aqueous phase as atomic number (Z) of REE increases, which is caused by the growth of hydration energy of Ln^{3+} ions because of decrease of their ionic radii when Z rises. This leads to increase in Lu/La separation factor ($\beta_{\mathrm{Lu/La}} = D_{\mathrm{Lu}}/D_{\mathrm{La}}$) with HNO_3 concentration (Fig. 2). The similar character of D_{Ln}–$[HNO_3]$ dependence was noted for the extraction of REE(III) with solutions of compounds **II** and **III** as coordination-solvated nitrates [23].

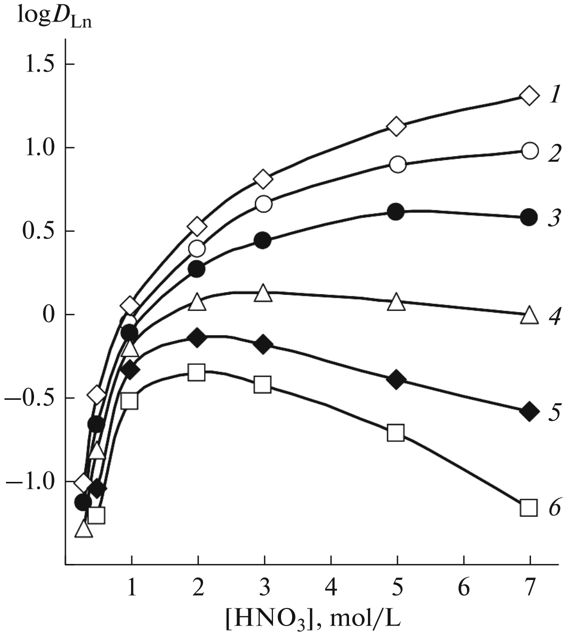


Fig. 2. Dependences of distribution ratios of (*1*) Lu(III), (*2*) Ho(III), (*3*) Tb(III), (*4*) Eu(III), (*5*) Nd(III), and (*6*) La(III) on HNO_3 concentration in equilibrium aqueous phase upon extraction with 0.1 M solutions of compound **I** in dichloroethane.

Metal : extractant stoichiometric ratio in the extracted complexes was determined by equilibrium shift method. Obtained data (Fig. 3) showed that REE(III) are extracted with this compound as a mixture of mono- and disolvates. REE(III) are extracted with solutions of compounds **II** and **III** in dichloroethane as complexes of the same stoichiometry [21]. The obtained data indicate that the nature of substituent at the phosphorus atom in compounds **I**–**III** causes no considerable change in the stoichiometry of extracted REE(III) complexes. On the basis of obtained data, the process of REE(III) extraction from HNO_3 solutions of moderate concentration with compound **I** can be described by the equations:

$$\mathrm{Ln}^{3+} + 3\mathrm{NO}_3^- + \mathrm{L}_{(\mathrm{o})} \rightleftarrows \mathrm{LnL}\left(\mathrm{NO}_3\right)_{3(\mathrm{o})}, K_1^{\mathrm{Ln}}, \quad (4)$$

$$\mathrm{Ln}^{3+} + 3\mathrm{NO}_3^- + 2\mathrm{L}_{(\mathrm{o})} \rightleftarrows \mathrm{LnL}_2\left(\mathrm{NO}_3\right)_{3(\mathrm{o})}, K_2^{\mathrm{Ln}}. \quad (5)$$

Table 1. Efficient constants of HNO_3 extraction with solutions of compounds **I**–**III** in dichloroethane

Compound	K_1	K_2
I	1.15 ± 0.06	0.09 ± 0.005
II	2.70 [21]	0.23 [21]
III	8.03 [21]	1.03 [21]

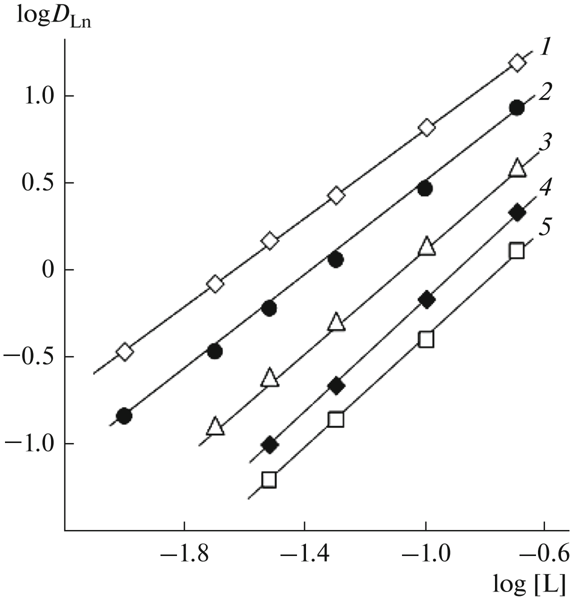


Fig. 3. Dependences of distribution ratios of (*1*) Lu(III), (*2*) Tb(III), (*3*) Eu(III), (*4*) Nd(III), and (*5*) La(III) on compound **I** concentration in dichloroethane upon extraction from 3 M HNO_3 solutions.

Table 2 shows the efficient constants of REE(III) extraction (K_1^{Ln} and K_2^{Ln}) calculated by nonlinear least squares using equation

$$D_{Ln} = \left[NO_3^-\right]^3 \gamma_\pm^4 \left(K_1^{Ln}[L]_{ini} f^{-1} + K_2^{Ln}[L]_{ini}^2 f^{-2}\right), \quad (6)$$

where $\gamma_\pm$ is activity coefficient of the corresponding REE(III) nitrate [39], f is a correction for extractant binding with nitric acid ($f = 1 + K_1a + K_2a^2$). The table also shows K_2^{Ln} values for compounds **II** and **III**. The presented data display that the extraction ability of PP toward REE(III) increases in the series of compounds **I** < **III** < **II**. The high extraction ability of compound **II** with phenyl substituents at the phosphorus atom toward REE(III) is due to appearance of AAS effect in the systems with PP.

Table 2. Efficient constants of REE(III) extraction from HNO_3 solutions with solutions of compounds **I–III** in dichloroethane

Ln(III)	K_1^{Ln} (I)	K_2^{Ln} (I)	K_2^{Ln} (II) [22]	K_2^{Ln} (III) [22]
La	2.02	4.40	6.96	5.01
Ce	2.12	4.52	7.16	5.10
Pr	2.18	4.62	7.40	5.25
Nd	2.21	4.67	7.68	5.49
Sm	2.46	4.83	7.86	5.66
Eu	2.53	4.92	8.01	5.76
Gd	2.51	4.94	7.98	5.56
Tb	2.89	5.07	8.38	5.80
Dy	2.93	5.06	8.49	5.84
Ho	2.92	5.01	8.58	5.84
Er	2.87	4.94	8.66	5.91
Tm	2.86	4.92	8.64	5.86
Yb	2.87	4.91	8.62	5.81
Lu	2.88	4.90	8.62	5.68

REE(III) extraction with compounds **I–III** from solutions with moderate HNO_3 concentration shows a trend to decrease REE(III) extraction efficiency when Z increases. This is caused by enhancement in the stability of REE(III) complexes with hard (according to Pearson) ligands when charge density of Ln^{3+} ions rises owing to decrease of their ionic radii as Z increases [40].

The efficiency of REE(III) extraction with solutions of compound **I** from nitric acid solutions considerably increases in the presence of IL. This may be due to the growth of hydrophobicity of extracted complexes on account of displacement of NO_3^- ions by more hydrophobic Tf_2N^- ions [41]. The character of $\log D_{Ln}$–$[HNO_3]$ dependence changes in the presence of IL in organic phase (Fig. 4). The decrease of D_{Ln} when $[HNO_3]$ rises up to 3 mol/L is caused by the drop of free extractant concentration in organic phase due to reaction of compound **I** with both HNO_3 and HTf_2N present in the system owing to marked transfer of C_4mim^+ and Tf_2N^- ions in aqueous phase [42]. Extraction of HTf_2N with compound **I** can be described by the equation:

$$H^+ + Tf_2N^- + L_{(o)} \rightleftarrows HTf_2NL_{(o)}, \quad (7)$$

while extraction constant can be expressed as

$$K_{HTf2N} = [HTf_2NL] \big/ \left([L]\left[H^+\right]\left[Tf_2N^-\right]\right). \quad (8)$$

The concentration constant of HTf_2N extraction with compound **I** in dichloroethane was calculated from the dependence of ion Tf_2N^- distribution on compound **I** concentration in organic phase and Tf_2N^- and H^+ ions in aqueous phase (Fig. 5). It was preliminary established that Li^+ and Cl^- ions do not transfer into organic phase under experimental conditions. The magnitude of $K_{HTf_2N} = 2690 \pm 80$ is much higher than the constant of HNO_3 extraction with this compound (Table 1), which is due to much higher hydrophobicity of Tf_2N^- anion as compared with NO_3^-.

The value of synergic effect $S = D/D_0$ (D and D_0 are distribution ratios of REE(III) in the presence and in the absence of IL in organic phase) decreases when $[HNO_3]$ rises. Thus, upon extraction of Eu(III), the value of S decreases from 2150 to 2.2 when $[HNO_3]$

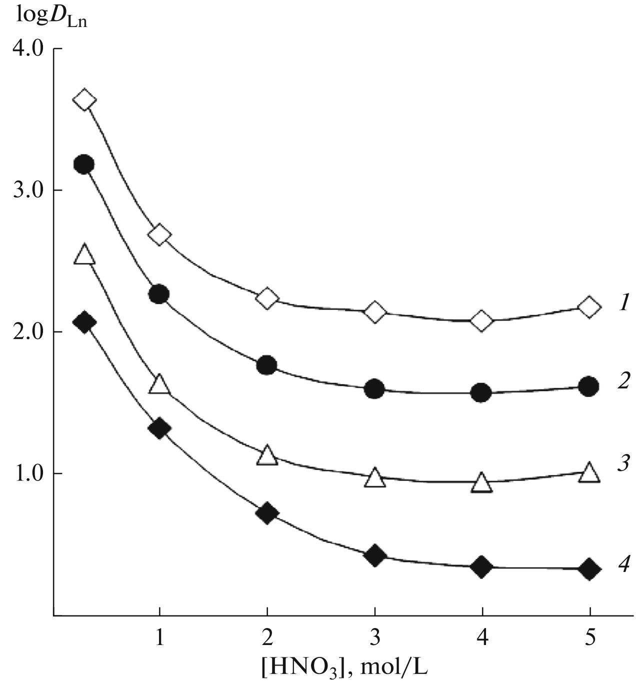


Fig. 4. Dependences of distribution ratios for (*1*) Lu(III), (*2*) Tm(III), (*3*) Ho(III), and (*4*) La(III) on HNO_3 concentration in equilibrium aqueous phase upon extraction with 0.1 M solutions of compound **I** in IL.

increases from 0.3 to 3 mol/L. The character of D_{Ln}–Z dependence for REE(III) extraction from nitric acid solutions with solutions of compound **I** in IL and dichloroethane considerably differs (Fig. 6). One can see that the extraction selectivity of heavy REE(III) in the system with IL is higher than on the use of dichloroethane as a solvent: the values of $\beta_{Lu/Sm}$ in these systems are 70.5 and 6.0, respectively.

Stoichiometric ratio REE(III) : **I** in the complexes extracted in the presence of IL vary from 1 : 3 to 1 : 2 as HNO_3 concentration in aqueous phase increases (Fig. 7), i.e., the system with IL displays the growth of solvation numbers in extracted complexes as compared with extraction with solutions of compound **I** in dichloroethane (Fig. 3). This is due to low coordination ability of Tf_2N^- ions [43], which seem to occupy outer coordination sphere of extracted complex, whereas REE(III) ions in the absence of IL are extracted with compounds **I–III** as coordination-solvated nitrates $LnL_s(NO_3)_3$ ($s = 1, 2$) where NO_3^- ions coordinate to Ln^{3+} ions [36].

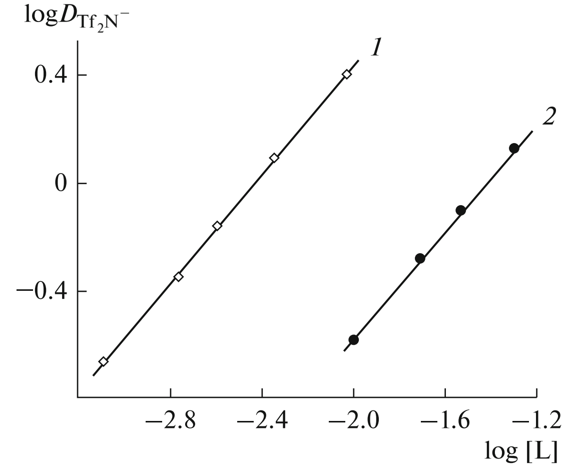


Fig. 5. Dependences of distribution ratio of Tf_2N^- ion on compound **I** concentration in dichloroethane upon extraction from 0.001 M $LiTf_2N$ solutions containing (*1*) 0.1 or (*2*) 0.01 mol/L HCl.

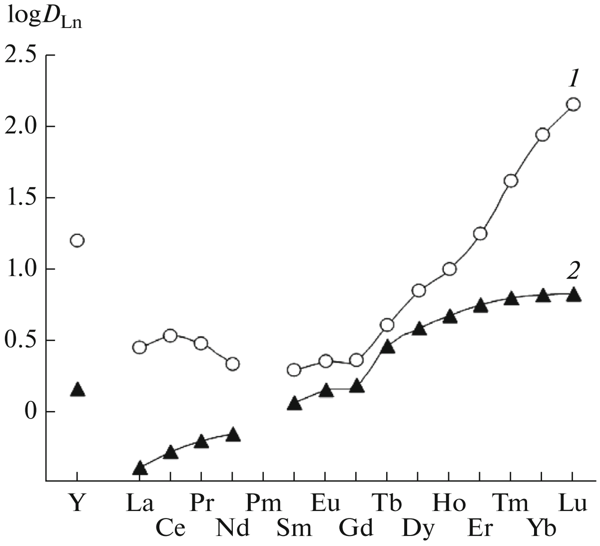


Fig. 6. Distribution ratios of REE(III) upon extraction from 3 M HNO_3 solutions with 0.1 M solutions of compound **I** in (*1*) IL and (*2*) dichloroethane.

CONCLUSIONS

The presented data shows that the extraction ability of PP toward REE(III) increases in the series of compounds **I** < **III** < **II**. The efficiency and selectivity of REE(III) extraction with diethyl 2-[(diphenylphosphoryl)methoxy]-5-ethylphenylphosphonate from nitric acid solutions considerably rises in the presence of ionic liquid, 1-butyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imide, in organic phase.

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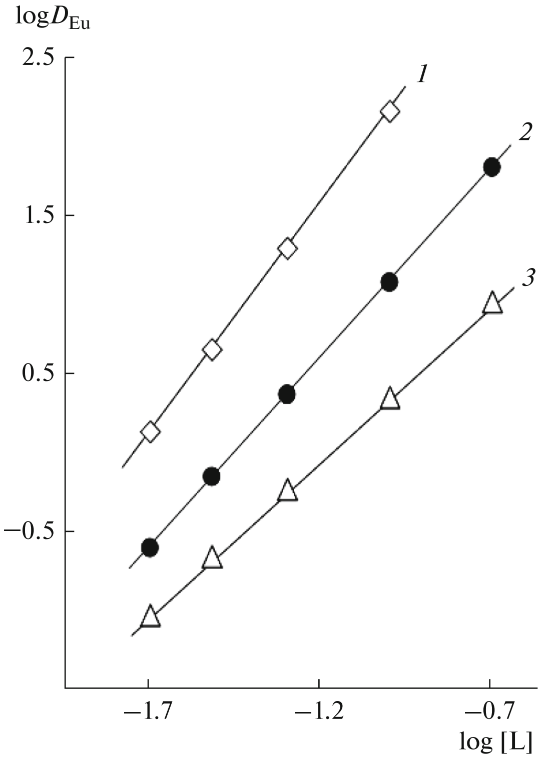

Fig. 7. Dependences of Eu(III) distribution ratios on compound **I** concentration in IL upon extraction from (*1*) 0.1, (*2*) 1.0, and (*3*) 3.0 M HNO_3 solutions.

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