

# Separation of Rare Earths by Solvent Extraction with an Undiluted Nitrate Ionic Liquid

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**Abstract** A solvent extraction system based on the ionic liquid tricaprylmethylammonium nitrate, [A336][NO<sub>3</sub>], has been investigated for separation of mixtures of rare earth elements (REE) at high total REE concentrations (up to 2 M). The chelating agent EDTA was added to the aqueous nitrate feed solution to increase the separation factors (SFs). This improvement of the SFs is based on the fact that [A336][NO<sub>3</sub>] extracts the light REE (LREE) stronger than the heavy REE (HREE), whereas a chelating agent forms more stable complexes with the HREE than with the LREE. The combination of these two effects makes that the LREE are even more efficiently extracted than in the absence of a chelating agent. The most efficient separation of the LREE La–Nd from the other REE was obtained using a total initial REE concentration of 1 M, an EDTA concentration of 0.2 M, and a total nitrate concentration of 11 M. Stripping of the extracted REE from the ionic liquid phase could be done using water.

**Keywords** Ionic liquids · Hydrometallurgy · Lanthanides · Rare earths · Solvent extraction

## Introduction

The use of ionic liquids in solvent extraction could lead to environmentally friendlier and more efficient processes for the separation of mixtures of rare earth elements (REE) [1–3]. Ionic liquids are solvents that consist entirely of ions. They are typically organic salts with a melting point below 100 °C [4]. Ionic liquids have an extremely low vapor pressure. Replacing molecular diluents by ionic liquids in solvent extraction processes reduces the flammability of the organic phase and also eliminates the risk of air pollution by evaporation of organic solvents [1]. Many studies on ionic liquids in solvent extraction make use of ionic liquids with a fluorinated anion, such as bis(trifluoromethylsulfonyl)imide or hexafluorophosphate, because these ionic liquids are immiscible with water. However, fluorinated anions are known to be highly persistent and the hexafluorophosphate anion tends to hydrolyze in contact with water, releasing hydrofluoric acid. Therefore, the ionic liquids of choice for solvent extraction processes are those comprising hydrophobic quaternary ammonium or phosphonium cations with long alkyl chains and a nonfluorinated anion [5]. These ionic liquids are preferably used in undiluted form, i.e., not diluted in a molecular organic solvent.

The choice of the anion in these hydrophobic ionic liquids depends on the requirements of the solvent extraction system. The chloride anion is a potential choice since REE ions can be extracted by quaternary ammonium chloride at very high chloride concentrations in the aqueous feed solution, but extraction efficiencies are low [6, 7]. Low extraction efficiencies are also observed for extraction of REE ions from chloride feed solutions with neutral extractants [8]. The most obvious choice as the anion for extraction of REE ions by undiluted ionic liquids is the

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nitrate ion. Nitrates have historically been used for REE separation systems because nitrates form highly extractable complexes with REE ions. The use of tri-caprylmethylammonium nitrate in REE separations is well documented in the scientific literature and the patent literature, but these extractants have always been used diluted in molecular diluents and not as pure ionic liquids [9–17]. Gaudernack et al. described the use of a quaternary ammonium nitrate dissolved in organic diluents to extract light rare earth elements (LREE) from an aqueous nitrate solution, followed by extraction of the heavy rare earth elements (HREE) using a quaternary ammonium thiocyanate dissolved in organic solvents, with the aim to separate yttrium from the other rare earths [16]. Bauer et al. found that the separation factors (SFs) for extraction of the LREE could be improved by the addition of a chelating agent such as diethylenetriaminepentaacetic acid (DTPA) to the aqueous solution [12]. This improvement of the SFs is based on the fact that the quaternary ammonium nitrate extracts the LREE stronger than the HREE, whereas a chelating agent forms more stable complexes with the HREE than with the LREE. The combination of these two effects makes that the LREE are even more efficiently extracted than in the absence of a chelating agent. Trimble et al. described the use of an amine extractant to extract LREE, followed by extraction with an acidic extractant to remove the HREE, with the aim of obtaining pure yttrium [17]. Morais et al. investigated the use of tricaprylmethylammonium nitrate dissolved in kerosene for Eu/Gd separation, and found that it was independent of the nitrate concentration [10]. Lu et al. studied the Pr/Nd separation by tricaprylmethylammonium nitrate in an aromatic diluent with DTPA as the chelating agent in the aqueous phase [11].

Recently, our team has reported on the use undiluted hydrophobic nitrate ionic liquids for the extraction of REE, both for the separation of REE mixtures and for the separation of REE from transition metal ions [18–23]. The fact that the concentration of the extractant is very high since the ionic liquid phase acts itself as the extractant also leads to high distribution ratios. These studies made use of two commercially available nonfluorinated hydrophobic ionic liquids as a starting material: tricaprylmethylammonium chloride (Aliquat<sup>®</sup> 336, [A336][Cl]) and trihexyl(tetradecyl)phosphonium chloride (Cyphos<sup>®</sup> IL 101, [C101][Cl]), both of which can have their chloride anions easily replaced by nitrate ions, resulting in the ionic liquids [A336][NO<sub>3</sub>] and [C101][NO<sub>3</sub>], respectively. The extraction occurs via an anion exchange mechanism.

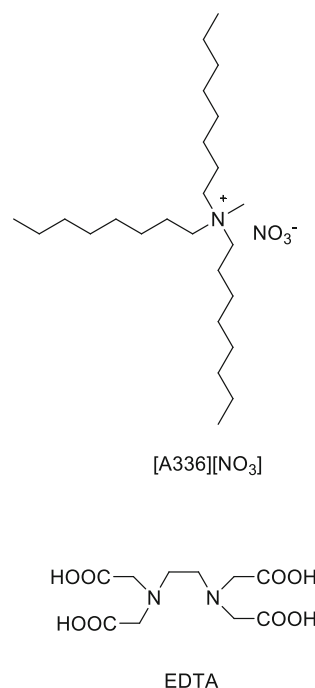
In this paper, we report on the separation of REE mixtures by extraction with the undiluted nitrate ionic liquid [A336][NO<sub>3</sub>] from an aqueous nitrate feed solution containing ethylenediaminetetraacetic acid (EDTA) as a

chelating agent (Fig. 1). This is an extension of work that has been reported earlier as a preliminary report [24]. The “rare-earth crisis” and the accompanying supply risk has led to the desire to exploit geographically more diverse REE resources, with a consequence that REE ores with a mineralogy different from that of the conventional REE ores bastnäsite, monazite, or xenotime have to be processed [25]. This study therefore uses a European REE source as a model for the distribution of the aqueous solutions used for extraction studies, the Norra Kärr deposit in southern Sweden [26, 27]. The main REE-bearing mineral in this deposit is the complex silicate eudialyte.

## Experimental

### Chemicals

Tricaprylmethylammonium chloride (Aliquat<sup>®</sup> 336, [A336][Cl]) is a mixed quaternary ammonium salt containing mainly trioctylmethylammonium and tridecylmethylammonium chloride (Sigma-Aldrich, 88.2–90.6 %). The following rare earth oxides were used: Y<sub>2</sub>O<sub>3</sub> (Rhodia, 99.99 %), La<sub>2</sub>O<sub>3</sub> (Sigma-Aldrich, 99.99 %), Pr<sub>6</sub>O<sub>11</sub> (Janssen Chemicals, 99.9 %), Nd<sub>2</sub>O<sub>3</sub> (Rhodia, 99.9 %), Sm<sub>2</sub>O<sub>3</sub> (Rare Earth Products Ltd., 99.9 %), Eu<sub>2</sub>O<sub>3</sub> (Inframatt Advanced Materials, 99.99 %), Gd<sub>2</sub>O<sub>3</sub> (Ventron LFA Products, 99.9 %), Tb<sub>4</sub>O<sub>7</sub> (Rare Earth Products Ltd.,



**Fig. 1** Chemical structure of the main component of the ionic liquid [A336][NO<sub>3</sub>] and EDTA

99.99 %), Dy<sub>2</sub>O<sub>3</sub> (Rare Earth Products Ltd., 99.99 %), Ho<sub>2</sub>O<sub>3</sub> (Rare Earth Products Ltd., 99.99 %), Er<sub>2</sub>O<sub>3</sub> (Acros Organics, 99.99 %), Tm<sub>2</sub>O<sub>3</sub> (GFS Chemicals, 99.9 %), Yb<sub>2</sub>O<sub>3</sub> (Rhodia, 99.9 %), and Lu<sub>2</sub>O<sub>3</sub> (Rare Earth Products Ltd., 99.9 %). EDTA (99 %) and DTPA (99 %) were purchased from Acros Organics, while KNO<sub>3</sub> (>99 %), NaNO<sub>3</sub> (>99 %), and NH<sub>4</sub>NO<sub>3</sub> (>99 %) were obtained from Chem-Lab. All dilutions were made using pure water (MilliQ, Millipore, >18 MΩ/cm).

To prepare the ionic liquid [A336][NO<sub>3</sub>], Aliquat<sup>®</sup> 336 was preequilibrated three times for at least 1 h with a 2.5 M KNO<sub>3</sub> solution to exchange the chloride ions by nitrate ions. Three contacts with KNO<sub>3</sub> gave chloride levels in the ionic liquid phase close the detection limit of the total reflection X-ray fluorescence (TXRF) spectrometer, which is estimated to be lower than 100 ppm [28]. Potassium nitrate was used due to the easy detection of potassium by TXRF so that the contamination of the ionic liquid phase by the counter cation of the nitrate salts could easily be checked. However, no traces of potassium could be measured in [A336][NO<sub>3</sub>].

### Feed Solution

The aqueous feed solution mimicked the composition of a cerium-free REE concentrate (oxide, hydroxide, or carbonate) derived from the processing of ores of the HREE-rich eudialyte deposit of Norra Kärr in southern Sweden. A cerium-free solution was considered because it is common practice in the REE industry to remove cerium in a step prior to separation of the other REE, for instance by selective oxidation of Ce(III) to Ce(IV), followed by precipitation of CeO<sub>2</sub> or another Ce(IV) compound. The feed solution was prepared by dissolving REE oxides in HNO<sub>3</sub>. The total REE concentration was 2.14 mol/L, and the pH was 1.5. The concentration of the individual REE can be found in Table 1. Feed solutions with different total REE concentrations were prepared by dilution of this stock solution. The nitrate concentration in the diluted solutions was kept constant by the addition of NH<sub>4</sub>NO<sub>3</sub>. Nitrate concentrations higher than 6 M were achieved by dissolution of extra NH<sub>4</sub>NO<sub>3</sub> salt. The pH was modified by the addition of a 6 M NH<sub>3</sub> solution.

### Extraction Experiments

Batch solvent extraction experiments were performed using approximately 1 mL of each phase, unless other phase ratios were tested, in which case the ionic liquid volume was increased. Samples were shaken at constant temperature (30 °C) using a TMS-200 turbo thermo shaker (Hangzhou Allsheng Instruments Co., Ltd.) for 30 min, unless otherwise specified. The pH measurements were

**Table 1** Composition of the aqueous feed solution

REE	Concentration (mol/L)
La	0.23
Pr	0.08
Nd	0.23
Sm	0.012
Eu	0.002
Gd	0.068
Tb	0.02
Dy	0.084
Ho	0.02
Er	0.029
Tm	0.011
Yb	0.045
Lu	0.011
Y	1.3
pH 1.5	

performed using a S220 SevenCompact pH/ion meter (Mettler-Toledo) and a slimtrode electrode (Hamilton) and calibrated using pH 1.0 (Merck), 4.0 (Chem-Lab), 7.0 (VWR), and 10 (Fischer) buffers. A Picofox S2 (Bruker) TXRF spectrometer was used to determine the metal concentrations in the solutions.

The distribution ratio ( $D$ ) of a single metal is defined as the ratio of the total concentration in the ionic liquid phase ( $[M]_{IL}$ ) by the total concentration in the aqueous phase ( $[M]_{aq}$ ) after extraction and phase separation:

$$D = \frac{[M]_{IL}}{[M]_{aq}} \quad (1)$$

The separation factors ( $SF_{A,B}$ ) are defined in Eq. (2), where the metals A and B are chosen so that  $SF > 1$  by convention:

$$SF_{A,B} = \frac{D_A}{D_B} \quad (2)$$

The phase ratio is defined as the volume of the organic phase O (i.e., the ionic liquid phase) divided by the volume aqueous phase A.

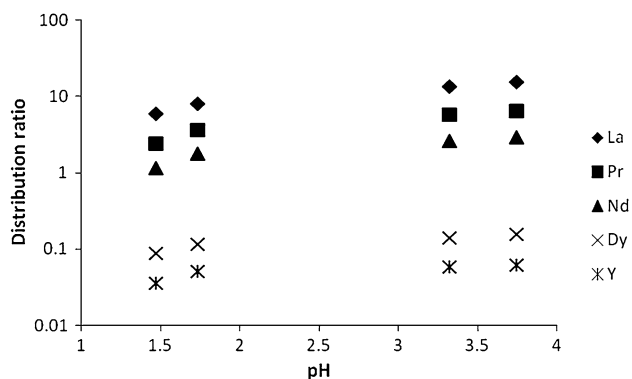
## Results and Discussion

EDTA was selected instead of DTPA as a chelating agent for forming complexes with the REE in the aqueous phase, because of the higher solubility of EDTA in water. A solution with an EDTA concentration of 0.8 M could easily be obtained. To evaluate the influence of pH on a system with the aqueous chelating agent EDTA, the pH was varied between 1.5 and 3.75 (Fig. 2). The distribution ratios for all REE increase with increasing pH values. However, the SFs

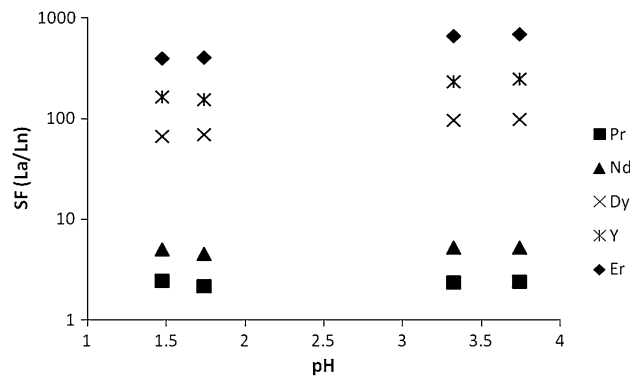
remain fairly constant as shown in Fig. 3. This means that for investigations into the effect of REE loading and EDTA concentration, the solutions should have the same pH value, but small variations in pH have only a small effect on SFs.

The EDTA concentration has a significant effect on both the distribution ratios and SFs. The influence of the EDTA concentration was investigated for solutions with two different total REE concentrations: 0.5 and 1 M. The nitrate concentration was kept constant at 6 M by the addition of  $\text{NH}_4\text{NO}_3$ . The initial pH of the solutions was 2.0. The ratios between the different REE was the same as in the 2 M feed solution (Table 1), because the 0.5 and 1 M solutions were prepared by dilution of the 2 M solution. The EDTA concentration was varied between 0.20 and 0.55 M. The effect of EDTA concentration on the 0.5 M REE system is shown in Figs. 4 and 5. The distribution ratios decrease strongly with an increase in EDTA concentration. All distribution ratios drop below 1 at a concentration of 0.55 M EDTA. Up to a concentration of 0.4 M EDTA, the distribution ratio of La remains fairly high and the SFs of La versus HREE+Y are around 10,000 or even higher. The effect of EDTA concentration on the solution with a total REE concentration of 1 M is shown in Figs. 6 and 7. The system is similar to the 0.5 M system with the differences that the distribution ratios are lower and the reduction of the  $D$  values with increasing EDTA concentration is less. The SFs are also somewhat lower, with the highest value observed for the highest tested EDTA concentration, with the SFs versus La for the HREE being about a factor of 10 lower.

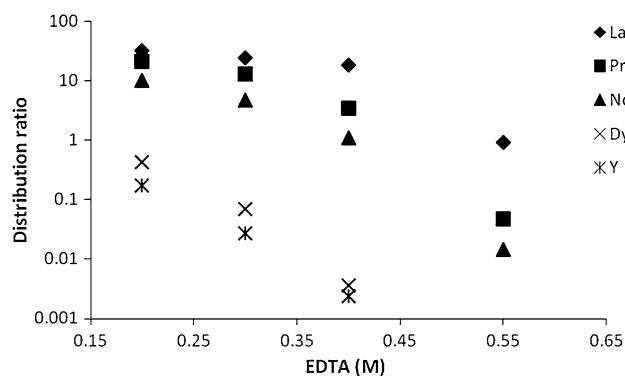
The SFs strongly depend on the total initial REE concentration in the feed solution (Fig. 8). For initial REE concentrations lower than 1 M at an EDTA concentration of 0.8 M, the SFs are very high, but the distribution ratios are low. For instance, the elements in the middle of the



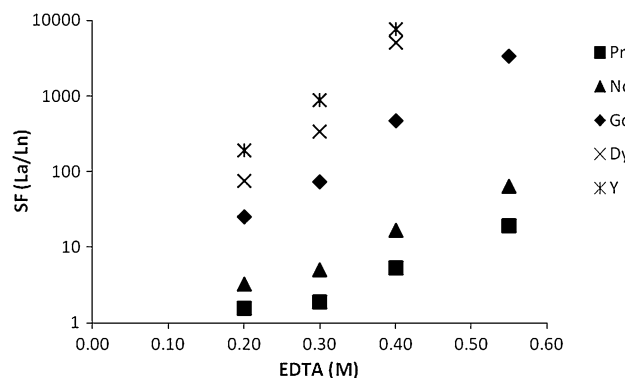
**Fig. 2** Distribution ratios for extraction of REE by  $[\text{A336}][\text{NO}_3]$  as a function of pH. The REE concentration in the initial feed solution was 2 M, the EDTA concentration was 0.8 M, and the initial pH was used. Only five elements are shown for the sake of clarity



**Fig. 3** Separation factors for extraction of REE by  $[\text{A336}][\text{NO}_3]$  as a function of pH. The REE concentration in the initial feed solution was 2 M, the EDTA concentration was 0.8 M, and the initial pH was used. Only five elements are shown for the sake of clarity

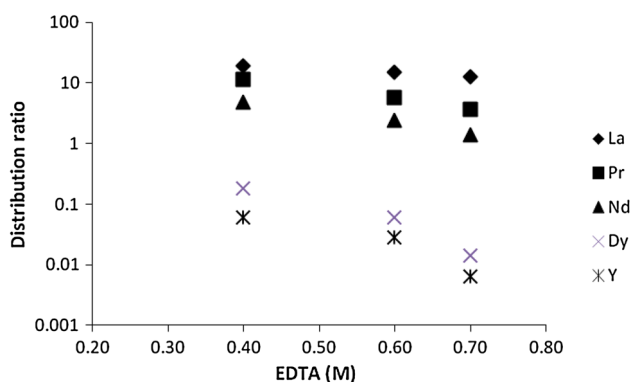


**Fig. 4** Influence of the EDTA concentration on the distribution ratios for extraction of REE by  $[\text{A336}][\text{NO}_3]$  from a feed solution with a total REE concentration of 0.5 M and a total nitrate concentration of 6 M. The initial pH was 2.0. Only five elements are shown for the sake of clarity

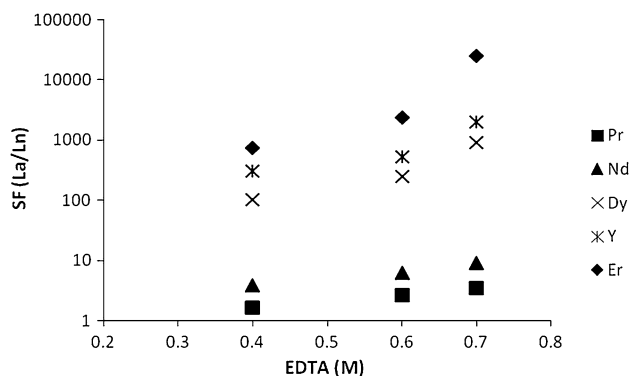


**Fig. 5** Influence of the EDTA concentration on the separation factors versus lanthanum for extraction of REE by  $[\text{A336}][\text{NO}_3]$  from a feed solution with a total REE concentration of 0.5 M and a total nitrate concentration of 6 M. The initial pH was 2.0. Only five elements are shown for the sake of clarity

lanthanide series have a SF versus lanthanum of approximately 10,000 at an initial REE concentration of 0.75 M. This means that the LREE can easily be extracted from the HREE. However, at a nitrate concentration of 6 M, the distribution ratios of the LREE are quite low ( $D < 10$ ). The distribution ratios can be increased by lowering the EDTA concentration and by increasing the nitrate concentration. By dissolving  $\text{NH}_4\text{NO}_3$  in the aqueous feed solution, solutions with a nitrate concentration of 11 M can be obtained at room temperature, without precipitation. In Table 2, the distribution ratios and the SFs versus La are given for extraction of REE by  $[\text{A336}][\text{NO}_3]$  from a nitrate solution with an initial total REE concentration of 1 M, a total nitrate concentration of 11 M, an EDTA concentration of 0.2 M, and a pH of 2.0. A longer mixing time (2 h) and a higher temperature (50 °C) were chosen than those for the other experiments to ensure that equilibrium was reached in these conditions so that the numerical data reflect the

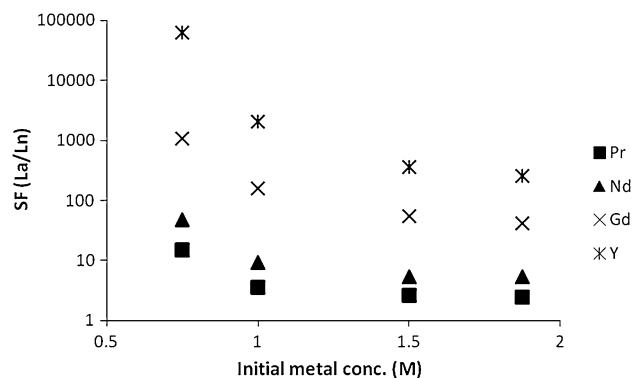


**Fig. 6** Influence of the EDTA concentration on the distribution ratios for extraction of REE by  $[\text{A336}][\text{NO}_3]$  from a feed solution with a total REE concentration of 1 M and a total nitrate concentration of 6 M. The initial pH was 2.0. Only five elements are shown for the sake of clarity



**Fig. 7** Influence of the EDTA concentration on the separation factors versus lanthanum for extraction of REE by  $[\text{A336}][\text{NO}_3]$  from a feed solution with a total REE concentration of 1 M and a total nitrate concentration of 6 M. The initial pH was 2.0. Only five elements are shown for the sake of clarity

equilibrium conditions. The concentrations of Pr in the aqueous phase and of Lu in the ionic liquid phase were below the detection limits of the TXRF spectrometer, so that no distribution ratios and SFs are given for these elements. The  $D$  values drop from 520 for La to 1.1 for Dy. The  $D$  values of the REE beyond Dy are well below 1. This extraction system works well for the separation of the LREE La–Nd from the REE in the middle of the lanthanide series and from the HREE. After extraction, the extracted REE can easily be stripped from the ionic liquid  $[\text{A336}][\text{NO}_3]$  by contacting the ionic liquid phase by water (slightly acidified to pH 2 to avoid hydrolysis of the REE). The possibility to strip the REE from the organic phase by water is a main advantage over extraction by acidic extractants, where extraction has to be done using a strong acid.



**Fig. 8** Separation factors as a function of total initial REE concentration for extraction of REE by  $[\text{A336}][\text{NO}_3]$ , at a constant EDTA concentration of 0.8 M and a nitrate concentration of 6 M. Only five elements are shown for the sake of clarity

**Table 2** Distribution ratios  $D$  and separation factors SF for extraction of REE by  $[\text{A336}][\text{NO}_3]$  from an aqueous nitrate solution (11 M nitrate and 0.2 M EDTA)

REE	$D$	SF (versus La)
La	520	1
Pr	–	–
Nd	42	13
Sm	6.3	83
Eu	4.7	110
Gd	4.1	130
Tb	2.7	190
Dy	1.1	480
Ho	0.5	1100
Er	0.2	3500
Tm	0.01	35,000
Yb	<0.01	82,000
Lu	–	–
Y	0.4	1200

Total initial REE concentration is 1 M. 2 h of mixing at 50 °C, phase ratio = 2 (O:A)



## Conclusions

A solvent extraction system based on the ionic liquid [A336][NO<sub>3</sub>] has been investigated for REE separations using high REE concentrations (up to 2 M total REE concentration) in nitrate aqueous feed solutions. EDTA was added to the aqueous phase as a chelating agent to increase the SFs. High distribution ratios and SFs could be achieved. The distribution ratios decrease with increasing atomic number across the lanthanide series. It is advantageous to work at high nitrate concentrations (11 M) because the distribution ratios for the LREE (La–Nd) are sufficiently high for efficient extraction by [A336][NO<sub>3</sub>]. An advantage of this extraction system is the easy stripping of the REE from the ionic liquid phase by water.

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