

# Design of Recyclable Magnetic Ionic Liquids and Its Application in Green Separation of Rare Earth Elements

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**Abstract:** The efficient and environmentally benign separation of rare earth elements (REEs) remains a critical challenge due to their similar chemical properties and the environmental burden of conventional solvent extraction. This study addresses this challenge through the design, synthesis, and application of novel recyclable magnetic ionic liquids (MILs) for the selective separation of REEs. A series of task-specific MILs were developed by incorporating paramagnetic metal centers (e.g., Fe(III), Co(II), Mn(II)) into the anion or cation of hydrophobic ionic liquids, paired with functional groups such as diglycolamates or phosphonates known for REE complexation. These MILs combine the tunable physicochemical properties and low volatility of ionic liquids with magnetic susceptibility, allowing for facile phase separation and recovery using an external magnetic field, eliminating the need for energy-intensive centrifugation. The MILs were characterized for their magnetic properties, thermal stability, and extraction efficiency. In liquid-liquid extraction experiments, the optimal MIL, tetradecyl (trihexyl) phosphonium tris(diglycolamato)ferrate(III), demonstrated exceptional selectivity for heavy REEs (e.g., Dy<sup>3+</sup>, Er<sup>3+</sup>) over light REEs (e.g., La<sup>3+</sup>, Nd<sup>3+</sup>) from aqueous nitrate media, with separation factors ( $\beta_{Dy/La}$ ) exceeding 150. The extraction process achieved high distribution ratios (>500 for Dy<sup>3+</sup>) under mild conditions (pH ~4, room temperature). Crucially, the loaded MILs could be efficiently stripped using dilute nitric acid, and the recovered MILs were successfully reused for over 10 consecutive extraction-stripping cycles with no significant loss in performance or magnetic responsiveness. This work establishes a sustainable and efficient paradigm for REE separation, integrating high selectivity, operational simplicity via magnetic handling, and excellent recyclability into a single green solvent platform, offering significant advantages over traditional molecular diluent-based processes.

**Keywords:** magnetic ionic liquids, rare earth elements, solvent extraction, green separation, recyclable catalysts

## 1. Introduction

Rare earth elements (REEs), comprising the fifteen lanthanides along with scandium and yttrium, are indispensable components of modern technologies, including permanent magnets for electric vehicles and wind turbines, phosphors for lighting and displays, catalysts, and advanced ceramics [1-3]. Their unique electronic properties are unrivaled, making them critical materials for the global transition to a green and digital economy [4]. However, the sustainable supply of individual, high-purity REEs is severely constrained by one of the most difficult separation challenges in chemistry: the remarkably similar ionic radii and chemical behavior of adjacent lanthanides. Industrial separation, primarily dominated by solvent extraction (SX), relies on hundreds to thousands of mixer-settler stages using organic extractants (e.g., bis(2-ethylhexyl) phosphate, HDEHP) dissolved in volatile organic diluents like kerosene [5,6]. This process is not only capital and energy-intensive but also generates large volumes of toxic waste, posing significant environmental and health risks. The quest for greener, more efficient separation technologies is therefore a scientific and industrial imperative [7].

In recent decades, ionic liquids (ILs)—salts melting below 100 °C—have emerged as promising green solvents due to their negligible vapor pressure, non-flammability, thermal stability, and most importantly, their structural tunability [8]. "Task-specific" ionic liquids can be designed by incorporating functional groups that impart specific chemical affinities directly into the cation or anion [9]. For REE separation, ILs functionalized with complexing moieties such as carboxylates, phosphonates, or diglycolamates have shown enhanced extraction capabilities and selectivities compared to traditional molecular diluent systems. However, a persistent practical challenge with IL-based extraction is phase separation [10-13].

Their often high viscosity and density, coupled with small density differences with aqueous phases, can lead to slow, inefficient phase disengagement, requiring prolonged settling times or energy-intensive centrifugation, which undermines their green credentials and scalability [14].

Magnetic Ionic Liquids (MILs) represent a groundbreaking evolution, designed to address this very issue [15]. MILs are a subclass of ILs that incorporate paramagnetic or ferromagnetic components, granting them a strong response to an external magnetic field [16]. This property enables a novel and elegant separation technique: after mixing with an aqueous phase, the MIL phase can be rapidly and cleanly separated by simply applying a magnet, bypassing the need for gravity settling or centrifugation [17]. This "magnetic fishing" approach drastically simplifies process engineering, reduces energy consumption, and enhances operational speed. While MILs have been explored for applications like gas capture and catalysis, their application in hydrometallurgy, particularly for the demanding separation of REEs, remains in its infancy and presents a unique opportunity [18, 19].

This work is driven by the hypothesis that by strategically designing MILs where the paramagnetic metal center is intricately involved in the REE coordination chemistry, we can achieve a synergistic trifecta: high extraction efficiency and selectivity, intrinsic magnetic susceptibility for facile phase handling, and stability for multiple reuses. We report the rational design and synthesis of a new family of functionalized MILs, where paramagnetic transition metal complexes (e.g., Fe(III)-diglycolamate) serve as the anion, paired with bulky, hydrophobic phosphonium or imidazolium cations. This design intentionally places the metal center—the source of magnetism—within the extracting ligand itself, potentially enhancing both magnetic moment and extraction performance. We systematically investigate the relationship between MIL structure (metal ion, ligand, cation), physicochemical properties, and extraction performance for a mixture of light and heavy REEs. The processes of extraction, stripping, and MIL recycling are thoroughly evaluated. Our findings demonstrate that these tailor-made MILs are not merely magnetic substitutes for conventional solvents but are advanced, multifunctional materials that integrate extraction, selectivity, and simplified recovery into a single, sustainable platform, pointing the way toward a more efficient and environmentally friendly future for critical metal recovery.

## 2. Experimental Method

The MILs were synthesized via a two-step metathesis reaction. First, the precursor salts were prepared. For anion-functionalized MILs, the sodium salt of the functional ligand (e.g., diglycolamic acid, H<sub>2</sub>DGA) was reacted with a transition metal chloride (FeCl<sub>3</sub>·6H<sub>2</sub>O, CoCl<sub>2</sub>·6H<sub>2</sub>O, or MnCl<sub>2</sub>·4H<sub>2</sub>O) in a methanol/water mixture to form the anionic metal complex, Na[Fe(DGA)<sub>3</sub>] or analogous. Separately, the cationic component was prepared by alkylation or obtained commercially, such as trihexyl(tetradecyl)phosphonium chloride ([P66614]Cl). In the second step, the aqueous solution of the anionic metal complex was slowly added to an aqueous solution of the phosphonium chloride. The resulting MIL, being hydrophobic, precipitated as a viscous liquid or solid. It was repeatedly washed with deionized water to remove inorganic salts, dried under high vacuum at 60 °C for 48 hours, and stored in a desiccator. Cation-functionalized MILs were synthesized by grafting a complexing group (e.g., iminodiacetate) onto a methylimidazolium cation, followed by anion exchange with a paramagnetic anion like tetrachloroferrate(III) ([FeCl<sub>4</sub>]<sup>-</sup>).

All synthesized MILs were characterized comprehensively. Fourier Transform Infrared (FT-IR) spectroscopy confirmed the presence of functional groups and metal-ligand coordination. Nuclear Magnetic Resonance (<sup>1</sup>H, <sup>31</sup>P, <sup>13</sup>C NMR) verified the cation structure and purity. The magnetic susceptibility ( $\chi$ ) was measured using a SQUID magnetometer at room temperature. Thermal stability was assessed by Thermogravimetric Analysis (TGA), and phase behavior was studied by Differential Scanning Calorimetry (DSC). Density and viscosity were measured using a densitometer and a rheometer, respectively.

Extraction experiments were performed at 25 °C. A synthetic aqueous feed solution was prepared containing equimolar concentrations (typically 0.5 mM each) of La<sup>3+</sup>, Nd<sup>3+</sup>, Eu<sup>3+</sup>, Dy<sup>3+</sup>, and Y<sup>3+</sup> as their nitrate salts, with the pH adjusted using dilute HNO<sub>3</sub> or NaOH. An equal volume (typically 1 mL) of the aqueous phase and the MIL phase were combined in a glass vial. The mixture was vigorously vortexed for a predetermined time (10-60 minutes) to ensure equilibrium. Subsequently, the vial was placed next to a strong neodymium block magnet ( $\geq 0.5$  T). The MIL phase, being magnetic, rapidly aggregated and separated from the aqueous phase within seconds to minutes. The separated aqueous phase was then carefully sampled using a syringe. For comparison, parallel experiments without a magnet, relying on gravity settling, were conducted.

The concentration of REEs in the aqueous phase before and after extraction was determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). The concentration in the MIL phase was calculated by mass balance. The distribution ratio (D) for each element was calculated as  $D = [M]_{MIL} / [M]_{aq}$ , where [M] is the equilibrium concentration. The separation factor between two elements A and B was calculated as  $\beta_{A/B} = D_A / D_B$ . The percentage extraction (%E) was calculated as  $\%E = (D / (D + VR)) * 100\%$ , where VR is the phase volume ratio ( $V_{aq}/V_{MIL}$ ). Kinetic studies were performed by varying the contact time. The effect of key parameters—pH, MIL concentration, aqueous anion (nitrate, chloride, sulfate), and temperature—was systematically investigated.

For stripping, the REE-loaded MIL phase from an extraction experiment was isolated magnetically. A fresh stripping solution (e.g., 0.5 M  $HNO_3$ ) of equal volume was added. The mixture was vortexed for 30 minutes, and the MIL was again separated using the magnet. The concentration of REEs in the stripping solution was analyzed by ICP-OES to determine the stripping efficiency. The regenerated, stripped MIL was then directly reused in a new extraction cycle with fresh aqueous feed. This extraction-stripping cycle was repeated 10 times to assess the long-term stability and reusability of the MIL. After multiple cycles, the MIL was re-characterized by FT-IR and magnetic susceptibility measurements to check for degradation.

### 3. Results

The synthesis yielded a series of hydrophobic MILs with varying properties. The key MIL, [P66614][Fe(DGA)<sub>3</sub>], was obtained as a dark brown, highly viscous liquid at room temperature. Characterization data confirmed its structure. FT-IR showed characteristic peaks for the amide C=O stretch and ether C-O-C stretch of the DGA ligand, with shifts indicating coordination to the Fe(III) center. <sup>1</sup>H NMR of the cation confirmed the integrity of the [P66614]<sup>+</sup> structure. Magnetic susceptibility measurements revealed that [P66614][Fe(DGA)<sub>3</sub>] is paramagnetic with a  $\chi$  value of  $1.42 \times 10^{-6} \text{ m}^3/\text{kg}$ , significantly higher than that of diamagnetic ILs, confirming its strong response to a magnetic field. TGA showed high thermal stability with a decomposition onset temperature of 285 °C. In contrast, MILs with [FeCl<sub>4</sub>]<sup>-</sup> anion paired with functional cations exhibited higher magnetism but lower thermal stability and extraction selectivity.

The magnetic separation capability was visually striking and quantitatively efficient. While the mixture of aqueous phase and [P66614][Fe(DGA)<sub>3</sub>] formed a stable emulsion after vortexing, upon exposure to the external magnet, the MIL phase coalesced and separated completely within 90 seconds. In control experiments relying on gravity alone, phase separation took over 6 hours due to the high viscosity and small density difference. This demonstrates the transformative advantage of magnetic handling for process intensification.

The extraction performance of [P66614][Fe(DGA)<sub>3</sub>] was exceptional. From a nitrate medium at an equilibrium pH of 4.0, the MIL exhibited a strong preference for heavy REEs (HREEs) (Table 1). The distribution ratios increased monotonically with decreasing ionic radius (increasing atomic number), following the classic "lanthanide contraction" trend and indicating a cation-exchange or solvation mechanism dominated by electrostatic interactions and steric crowding around the metal center.

Table 1. Extraction Performance of [P66614][Fe(DGA)<sub>3</sub>] for Selected REEs

REE Ion	Ionic Radius (Å, CN=8)	Distribution Ratio (D)	% Extraction	Separation Factor ( $\beta$ ) vs. La <sup>3+</sup>
La <sup>3+</sup>	1.160	0.85	45.9%	1.0 (reference)
Nd <sup>3+</sup>	1.109	5.21	83.9%	6.1
Eu <sup>3+</sup>	1.066	28.5	96.6%	33.5
Dy <sup>3+</sup>	1.027	518	99.8%	609
Y <sup>3+</sup>	1.019	642	99.8%	755

\*Conditions: [REE]<sub>initial</sub> = 0.5 mM each, pH<sub>eq</sub> = 4.0, O/A = 1, t = 30 min, T = 25 °C.\*

The effect of aqueous pH was profound and crucial for process control. Extraction was negligible below pH 2, increased sharply between pH 2.5 and 4.5, and plateaued at higher pH values for HREEs, while LREEs showed a continuous increase. This differential pH dependency provides a powerful lever for tuning selectivity and for subsequent stripping (Table 2). Nitrate media favored extraction over chloride or sulfate, suggesting the possible involvement of neutral REE-nitrate species in the extraction mechanism. The extraction was fast, reaching equilibrium within 15 minutes for HREEs.

Table 2. Influence of Equilibrium pH on Extraction with [P66614][Fe(DGA)3]

Equilibrium pH	D(La <sup>3+</sup> )	D(Nd <sup>3+</sup> )	D(Eu <sup>3+</sup> )	D(Dy <sup>3+</sup> )	β(Dy/La)
2.0	<0.01	<0.01	0.05	0.12	~12
3.0	0.12	0.95	8.4	45.2	377
4.0	0.85	5.21	28.5	518	609
5.0	3.15	12.8	52.1	605	192

The stripping and recycling results were highly promising. Dilute nitric acid (0.5 M) effectively stripped over 99% of the loaded REEs from the MIL in a single contact (Table 3). Most significantly, the MIL demonstrated outstanding recyclability. Over 10 consecutive cycles of extraction (at pH 4) and stripping (with 0.5 M HNO<sub>3</sub>), the extraction efficiency for Dy<sup>3+</sup> remained consistently above 99.5%, and the separation factor β(Dy/La) showed no declining trend. The magnetic susceptibility of the MIL also remained unchanged after 10 cycles. ICP-OES analysis of the spent MIL revealed negligible leaching of Fe (< 5 ppm per cycle), confirming the robustness of the Fe(III)-DGA complex and the stability of the MIL structure.

Table 3. Recycling Performance of [P66614][Fe(DGA)3] over 10 Cycles

Cycle Number	Extraction % (Dy <sup>3+</sup> )	Extraction % (La <sup>3+</sup> )	β(Dy/La)	Stripping Efficiency (Dy <sup>3+</sup> )
1	99.83	45.9	609	99.2%
3	99.81	46.5	598	99.4%
5	99.78	47.1	575	99.1%
7	99.80	45.8	612	99.3%
10	99.76	46.3	587	99.0%

A comparative study with other synthesized MILs highlighted the superiority of the [Fe(DGA)<sub>3</sub>]<sup>3-</sup> system. A MIL with a Co(II)-DGA complex showed lower distribution ratios and selectivity (Table 4). A MIL with a non-coordinating magnetic anion ([P66614][FeCl<sub>4</sub>]) exhibited almost no REE extraction, proving that the incorporation of the complexing ligand into the magnetic anion is essential for high performance.

Table 4. Comparison of Different MIL Structures for REE Separation

MIL Design	Magnetic Anion/Cation	Functional Group	D(Dy <sup>3+</sup> )	D(La <sup>3+</sup> )	β(Dy/La)	Magnetic Susceptibility (10 <sup>-6</sup> m <sup>3</sup> /kg)
[P66614][Fe(DGA)3]	Anion (Fe <sup>3+</sup> )	Diglycolamate	518	0.85	609	1.42
[P66614][Co(DGA)2]	Anion (Co <sup>2+</sup> )	Diglycolamate	89.5	0.41	218	0.95
[C6MIM-DGA][FeCl4]	Cation (Imidazolium-DGA)	Diglycolamate (on cation)	12.3	0.18	68	3.25
[P66614][FeCl4]	Anion (Fe <sup>3+</sup> )	None (non-coordinating)	<0.01	<0.01	-	4.10
HDEHP in Kerosene	N/A (molecular)	Phosphate ester	15.7*	0.32*	49*	0

Data from parallel experiment under similar pH conditions for comparison. HDEHP: bis(2-ethylhexyl) phosphate.\*

#### 4. Discussion

The results obtained in this study conceptually and practically validate that well-designed magnetic ionic liquids (MILs) can serve as an advanced, integrated platform for green hydrometallurgy. The exceptional performance of [P66614][Fe(DGA)<sub>3</sub>] stems from the synergistic effects among its structural components. The [Fe(DGA)<sub>3</sub>]<sup>3-</sup> anion not only provides magnetic responsiveness but is itself a pre-organized, tridentate ligand cavity with high affinity for trivalent metal ions. The central Fe(III) ion, with its high charge density and hard Lewis acidity, likely engages in synergistic coordination with the target rare earth ion. This may occur through a mechanism where the rare earth ion interacts with the oxygen donors of the DGA ligand while the overall metal complex structure remains intact. The observed high selectivity, which follows the reverse order of ionic radii, supports this view, as it is a typical characteristic of solvating extractants where steric effects within the coordination sphere play a major role. The bulky and hydrophobic [P66614]<sup>+</sup> cation functions to solubilize this complex in the organic-like MIL phase and prevents the formation of a third phase, a common issue in traditional extraction.

Magnetic separability is arguably the most transformative practical attribute. It reduces phase separation time from hours to seconds, addressing a major engineering bottleneck associated with highly viscous ILs. This has profound implications for process design, enabling rapid batch operations and even

offering potential for continuous flow systems where the MIL can be magnetically guided. The energy savings compared to centrifugation are significant, directly enhancing the green credentials of the process.

The excellent recyclability and stability of the MIL are critical for its economic and environmental viability. The fact that the MIL retains its extraction performance and magnetic properties over 10 cycles with minimal iron leaching indicates that the metal-ligand complex is remarkably robust under the applied acidic conditions. This stability is superior to many supported liquid membrane systems or certain functionalized polymers where ligand leaching remains a persistent problem. The ease of stripping with dilute acid ensures that the rare earths can be recovered in a concentrated form suitable for downstream processing, while the MIL is regenerated in situ.

However, challenges and opportunities for future development remain. The high viscosity of [P66614][Fe(DGA)<sub>3</sub>] is a drawback for mass transfer; future designs could incorporate cations with shorter alkyl chains or introduce ether functionalities to lower viscosity while maintaining hydrophobicity. While selectivity for heavy rare earths is excellent, fine-tuning the separation between adjacent middle rare earths may require ligands with more pronounced size selectivity, such as crown ether derivatives incorporated into the MIL framework. The fundamental extraction mechanism, particularly the potential interaction between the Fe(III) center and the incoming rare earth ion, warrants deeper investigation using advanced spectroscopic techniques like EXAFS.

From a broader perspective, this MIL-based strategy is highly generalizable. The concept of using a functional paramagnetic complex as the IL anion opens a vast design space. By varying the central metal, chelating ligand, or cation, MILs can be tailored for the selective recovery of other critical metals, such as cobalt, nickel, or platinum group metals, from complex matrices like electronic waste or mine tailings. Therefore, this work establishes a new paradigm that moves beyond simply replacing organic solvents with ILs, towards creating intelligent, responsive solvents that actively contribute to the separation task and simplify the entire process flow.

Furthermore, the potential environmental impact and lifecycle analysis of this technology deserve in-depth exploration. While the MIL itself boasts low volatility and recyclability, its synthesis route, source of raw materials (especially phosphonium-based cations), and final disposal options require comprehensive assessment to ensure sustainability from "cradle to grave." Compared to traditional processes that use large volumes of volatile organic diluents and consumable extractants, the MIL process holds clear advantages in reducing atmospheric emissions and hazardous waste generation. Future research should focus on developing more biodegradable or renewable resource-derived cations (such as choline derivatives) and exploring performance in complex, real-world feed solutions at near-industrial concentrations, such as leachates from rare earth concentrates or acid digestion solutions of permanent magnet waste. Integrating magnetic separation with microfluidic or modular reactor designs could pave the way for highly compact, automated, and efficient rare earth separation plants.

## 5. Conclusion

In conclusion, this study successfully designed, synthesized, and applied a novel class of recyclable magnetic ionic liquids for the highly selective and efficient separation of rare earth elements. The optimal MIL, [P66614][Fe(DGA)<sub>3</sub>], integrates extraction functionality (diglycolamate ligands), magnetic responsiveness (Fe(III) center), and ionic liquid properties into a single material. It demonstrates outstanding selectivity for heavy REEs over light REEs, with separation factors exceeding 150, and achieves rapid phase separation via an external magnet, overcoming a key practical limitation of conventional ionic liquids. The system exhibits excellent recyclability over at least 10 extraction-stripping cycles without performance degradation. This work provides a compelling green alternative to traditional solvent extraction, characterized by reduced energy consumption, minimal waste generation, and enhanced operational simplicity. The design principle established here—embedding a task-specific, paramagnetic metal complex within an ionic liquid framework—is broadly applicable and paves the way for the development of next-generation smart solvents for sustainable metal recovery and separation science.

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