Formation and stability of Sc(III), La (III), Dy(III), Pr(III) and Y(III) complexes with hydroxypyridine-carboxylic acids in aqueous solution

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ABSTRACT

The complexation of three hydroxy-substituted pyridine carboxylic acids: 2-hydroxypicolinic acid (L^1), 3-hydroxynicotinic acid (L^2), and 2-hydroxy-6-methylnicotinic acid (L^3) - with Sc^{3+}, Y^{3+}, La^{3+}, Pr^{3+}, and Dy^{3+} was systematically studied using spectrophotometric and potentiometric titrations. Stepwise ligand deprotonation allowed determination of complex stoichiometries and formation constants ($\log \beta = 4.63 - 11.51$), revealing a strong dependence on metal ionic radius, charge density, and donor atom type. Oxygen-donor ligands L^1 and L^3 preferentially stabilize small, charge-dense cations (Sc^{3+}, Dy^{3+}), whereas the nitrogen-donor ligand L^2 favors larger ions (Y^{3+}, La^{3+}). The 6-methyl substituent in L^3 introduces steric hindrance that reduces complex stability for larger cations. These results demonstrate the combined influence of donor atom identity, steric effects, and metal-ion properties on rare-earth complex stability, providing a quantitative basis for the rational design of selective ligands and functional coordination materials.

Keywords: Rare-earth metals; Hydroxypyridine carboxylic acids; Metal complexes; Spectrophotometric titration; Potentiometric titration.

1. INTRODUCTION

Rare-earth elements (REEs), including scandium (Sc), yttrium (Y), and the fifteen lanthanides, are recognised as strategically critical metals due to their unique optical, magnetic, catalytic, and coordination-chemical properties. These properties are essential for defence and security technologies such as high-performance permanent magnets, advanced sensing systems, laser materials and specialised alloys. Beyond these sectors, REEs are also essential for a wide range of civilian applications, including clean energy technologies, electronics, automation, catalysis and the biomedical sciences. The rapid growth of these sectors has consequently led to a sustained increase in global demand for REEs [1, 2].

In nature, REEs occur at low concentrations and typically coexist within the same mineral matrices, rendering their extraction and primary enrichment intrinsically challenging. An additional difficulty arises from the pronounced chemical similarity of REE^{3+} ions, which differ mainly in terms of their ionic radius due to lanthanide contraction. Consequently, separating and purifying individual REEs requires highly selective, often energy-intensive processes [3, 4]. Despite the fact that conventional chelating agents like EDTA and simple pyridinecarboxylic acids have been used, they often have insufficient selectivity for effective separation of lanthanoids. Therefore, systematic investigations into the complexation of REE with tailored O,N-donor ligands are of considerable importance. Parameters including complex stability, coordination number and metal-ligand interaction modes provide key insights into ligand design and the development of functional materials [5].

Among potential ligand systems, hydroxypyridine-carboxylic acids are a particularly promising class. Compared with their unsubstituted analogues, these ligands exhibit enhanced electronic tunability arising from hydroxyl-induced resonance and inductive effects. These effects significantly influence the ligands' acid-base behavior and coordination strength. Structurally, they are hydroxy-substituted derivatives of picolinic, nicotinic and isonicotinic acids, and are found in biologically relevant compounds. Nicotinic acid, for example, is a precursor of nicotinamide adenine dinucleotide (NAD^+) [6, 7]. These ligands, which include a variety of oxygen and nitrogen donor locations, and, in certain instances, enol-keto tautomerism, demonstrate a range of coordination behavior and easily form stable metal complexes [8, 9].

Previous studies on REE complexation have largely focused on classical chelators, typically aminopolycarboxylates or simple pyridine-carboxylic acids, which often provide strong but unselective binding across the lanthanide series. By contrast, hydroxypyridine-carboxylic acids provide a platform with tunable structure, allowing the identity of the donor atom, the chelation mode and the steric environment to be varied systematically [5, 8]. Available comparative data indicates that non-hydroxylated derivatives of picolinic and nicotinic acids generally exhibit lower stability constants, highlighting the beneficial role of hydroxyl substitution in strengthening metal-ligand interactions [10]. Nevertheless, quantitative studies addressing the combined effects of ionic radius, donor atom type, and steric factors across multiple REE^{3+} ions remain limited.

In this context, 2-hydroxynicotinic acid (2Hnic), 3-hydroxypicolinic acid (3Hpic), and 2-hydroxy-6-methylnicotinic acid (2H6Mnic) constitute a representative ligand set with closely related frameworks but distinct functional-group positions and steric characteristics. This selection enables systematic evaluation of ortho effects and methyl-induced steric hindrance, which directly influence acidity, chelate-ring formation, and coordination flexibility. Combined with REE^{3+} ions, these ligands provide an appropriate model system for elucidating correlations between ionic radius, ligand structure, and complex stability.

From technological and environmental perspectives, such insights are highly relevant. A better understanding of REE-ligand complexation could lead to more selective and efficient separation processes, reducing chemical consumption and the generation of secondary waste. Furthermore, stability data facilitates the safe handling and recycling of REEs from electronic wastem[1][3]. The present study aims to advance the rational design of selective ligands for REE separation, and to support the development of REE-based materials for use in optics, electronics, catalysis and analytical chemistry.

2. EXPERIMENTS

2.1. Chemicals

2-Hydroxynicotinic acid (L^1); 3-Hydroxypicolinic acid (L^2); 2-hydroxy-6-methylnicotinic acid (L^3), inorganic salts: $LaCl_3$, $ScCl_3$, $YCl_3.6H2O$; $PrCl_3$, $DyCl_3.6H2O$; NaOH, HCl were purchased from Merck and Sigma-Aldrich. All reagents of 99% purity are used without further purification.

2.2. UV-Vis titration

UV-Vis absorption spectra of the organic ligands in aqueous-ethanol media were recorded over the pH range 2 - 9 and during titration with metal salts using a Varian Cary 50 Scan spectrophotometer (200 - 750 nm, 1 cm quartz cuvette). Solution concentrations were individually adjusted for each ligand. Ionization constants and metal-ligand formation constants were determined by the saturation method [11] using the following equations:

$$pK = -lgK = pH \pm lg\frac{A_{ion} - A}{A - A_{mol}} \tag{1}$$

$$\beta = \frac{\left({}^{A}/_{A_{max}}\right) \times C_M}{(C_M - ({}^{A}/_{A_{max}}) \times C_M) \times (C_L - ({}^{A}/_{A_{max}}) \times C_M)^n} \tag{2}$$

2.3. Potentiometric titration

Potentiometric titrations were performed by gradually adding standardised acid or base solutions to ligand samples, while continuously monitoring the pH using a calibrated glass electrode at room temperature. These titration profiles provided quantitative information on the protonation behavior of the ligands. pH measurements were taken during the potentiometric titration of ligand solutions (10^{-2} mol/L) and equimolar metal-ligand mixtures with a 10^{-1} mol/L NaOH solution on an EV-74 potentiometer at 21-23 °C. Prior to each experiment, the instrument was calibrated using freshly prepared buffer solutions to ensure the accuracy and stability of the pH readings.

3. RESULTS AND DISCUSSION

3.1. Study of complexation processes by spectrophotometric method

To examine ligand ionization and complex formation in solution, spectrophotometric titrations of ethanol solutions of ligands L^1-L^3 were carried out using NaOH, HCl, and trivalent metal salts. This method enables direct monitoring of deprotonation processes and metal-ligand interactions through changes in electronic absorption spectra (figure 1).

The UV-Vis spectra of L^1 and L^3 exhibit three characteristic absorption bands in the UV region, with the main maximum occurring at 326 and 330 nm, whereas L^2 shows a dominant band at 304 nm accompanied by a shoulder near 227 nm. These distinct spectral features provide a reliable basis for tracking ligand deprotonation and complex formation. Upon titration with NaOH, pronounced hypsochromic shifts of the main absorption band are observed for L^1 and L^3 ($\Delta\lambda$ = 34-35 nm), accompanied by a decrease in absorbance intensity. In contrast, L^2 displays only a minor shift ($\Delta\lambda$ = 9 nm). This behavior reflects the ability of L^1 and L^3 to undergo hydroxypyridine/oxopyridone tautomerism and form intramolecular hydrogen bonds, which strongly influence their electronic structures. Deprotonation disrupts these interactions, leading to substantial electron redistribution and marked spectral changes. In contrast, L^2 lacks comparable tautomeric equilibria and intramolecular hydrogen bonding, and its alkaline titration mainly involves carboxylate deprotonation, resulting in limited spectral variation.

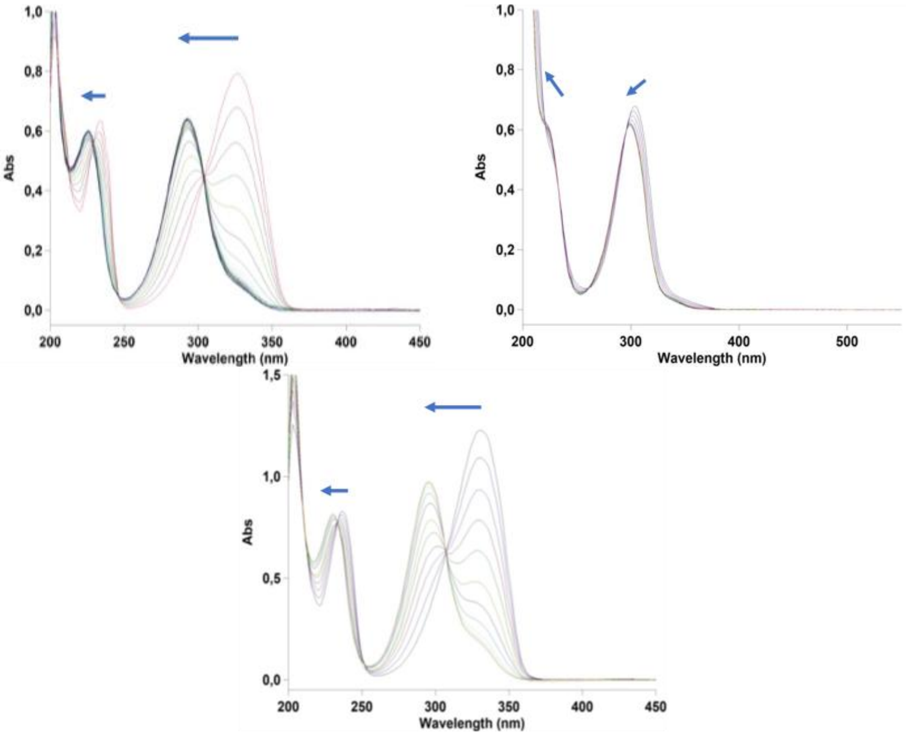


Figure 1. *Changes in the absorption spectra of ethanol solutions of* L^1 *(a),* L^2 *(b), and* L^3 *(c) upon addition of NaOH.*

Addition of HCl to water-ethanol solutions of L^1-L^3 down to pH 2 produces no significant spectral changes, consistent with stabilization of zwitterionic forms, in which the protonation of the pyridine nitrogen limits the uptake of further protons [12]. Saturation curves derived from the titration data were used to determine complex stoichiometries (ML_n) and formation constants (log β), as shown in figure 2, with the corresponding values summarized in table 1.

The stability constants of complexes of L^3 are systematically lower than those of L^1 and L^2, primarily due to the steric hindrance introduced by the methyl substituent at the 6-position of the pyridine ring. This substituent increases congestion near the coordinating site, distorts the chelate geometry, and reduces effective metal-ligand orbital overlap [13]. The destabilizing effect is more pronounced for larger REE^{3+} ions, such as La^{3+}, highlighting the strong sensitivity of lanthanide complex stability to subtle steric modifications in ligand structure.

Comparison with reported data for non-hydroxylated picolinic acid complexes of the same REE^{3+} ions suggests that 3-hydroxypicolinic acid (L^2) generally forms more stable complexes. The presence of the hydroxyl substituent can modify the electronic properties of the ligand through inductive and resonance effects, thereby enhancing the donor strength of the pyridine nitrogen and carboxylate group. This electronic influence may strengthen N,O-chelation and overall metal-ligand interactions, consistent with the trend discussed in the introduction.

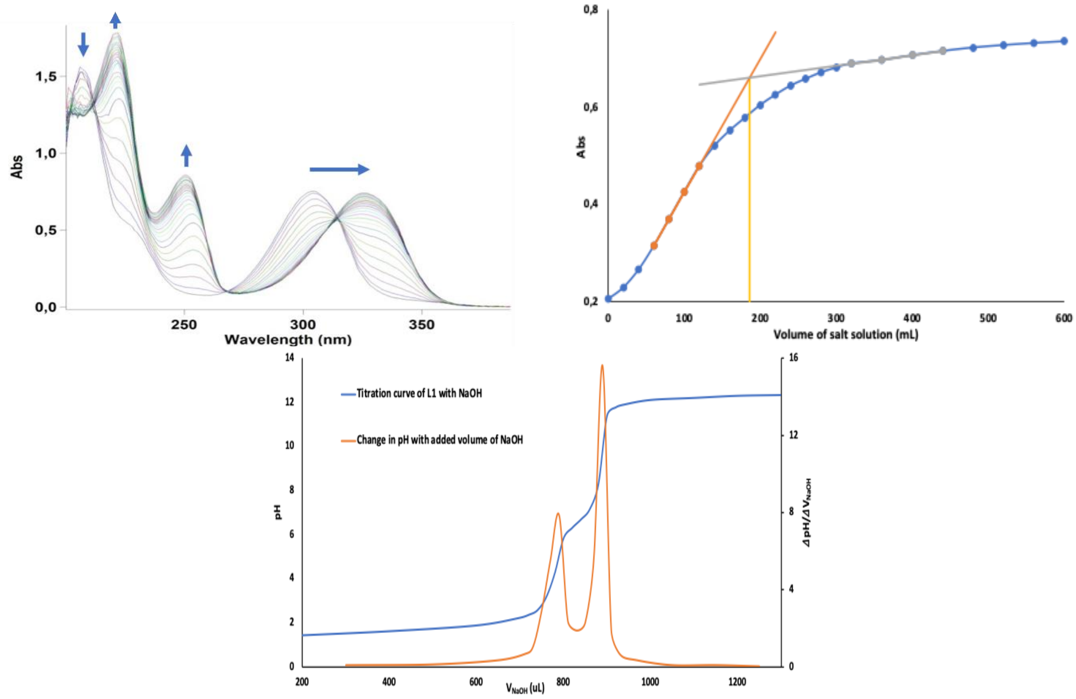


Figure 2. *Example of electronic absorption spectra (a), saturation curve (b) obtained from the titration of 3-hydroxypicolinic acid (L^2) with Sc^{3+} and potentiometric titration curves of L^1 (c).*

Table 1. *Formation constants of rare-earth metal complexes (Sc, Y, La, Pr, Dy) with ligands.*

STT	**M^{n+}**	**L^1**		**L^3**		**L^2**		**Picolinic acid** [14]	
		n (MLn)	lg*β*	n (MLn)	lg*β*	n (MLn)	lg*β*	n (MLn)	lg*β*
1	Sc^{3+}	1.5	8.33	1.5	7.49	1.5	8.97	2	-
2	Y^{3+}	1.5	8.65	1	5.84	1	7.95	2	7.36
3	La^{3+}	1.5	9.00	1	4.63	1.5	11.51	2	6.28
4	Pr^{3+}	1.5	8.41	1	6.17	1.5	10.48	2	6.96
5	Dy^{3+}	2	11.25	1	7.23	1.5	10.20	2	7.76

3.2. Potentiometric titration

To identify the pH ranges relevant to complex formation, potentiometric titrations were performed for the free ligands and their mixtures with metal ions using standardized NaOH solutions. The titration curves of L^1-L^3 exhibit two well-defined inflection points, corresponding to the stepwise dissociation of two acidic protons (figure 2c). Analysis of the potentiometric data yielded the acidity constants $pK_{a1} = 2.52$ and $pK_{a2} = 7.25$ for L1, $pK_{a1} = 3.22$ and $pK_{a2} = 8.04$ for L^2, and $pK_{a1} = 2.50$ and $pK_{a2} = 6.23$ for L^3. These differences reflect variations in electronic structure, substituent effects, and donor-atom positioning within the heterocyclic ring, which in turn govern ligand deprotonation behavior, coordination preferences, and complex stability in solution.

It should be noted that reported pK_a values for hydroxypyridine carboxylic acids vary considerably in the literature. Such discrepancies mainly arise from the strong involvement of intra- and intermolecular hydrogen bonding and tautomeric equilibria, which are highly sensitive to solvent composition, temperature, concentration, and the experimental method used for pK_a determination [15, 16]. In addition, solvation effects in mixed aqueous-organic media are known to induce systematic shifts in dissociation constants [15], and differences in experimental protocols often lead to inconsistent pK_a datasets for closely related ligands [17]. For comparison, representative literature pK_a values for L^1 and L^3 are summarized alongside the present results in table 2.

Table 2. *Some of known values of dissociation constants L^1 and L^3 obtained by different experimental methods. (*: our study).*

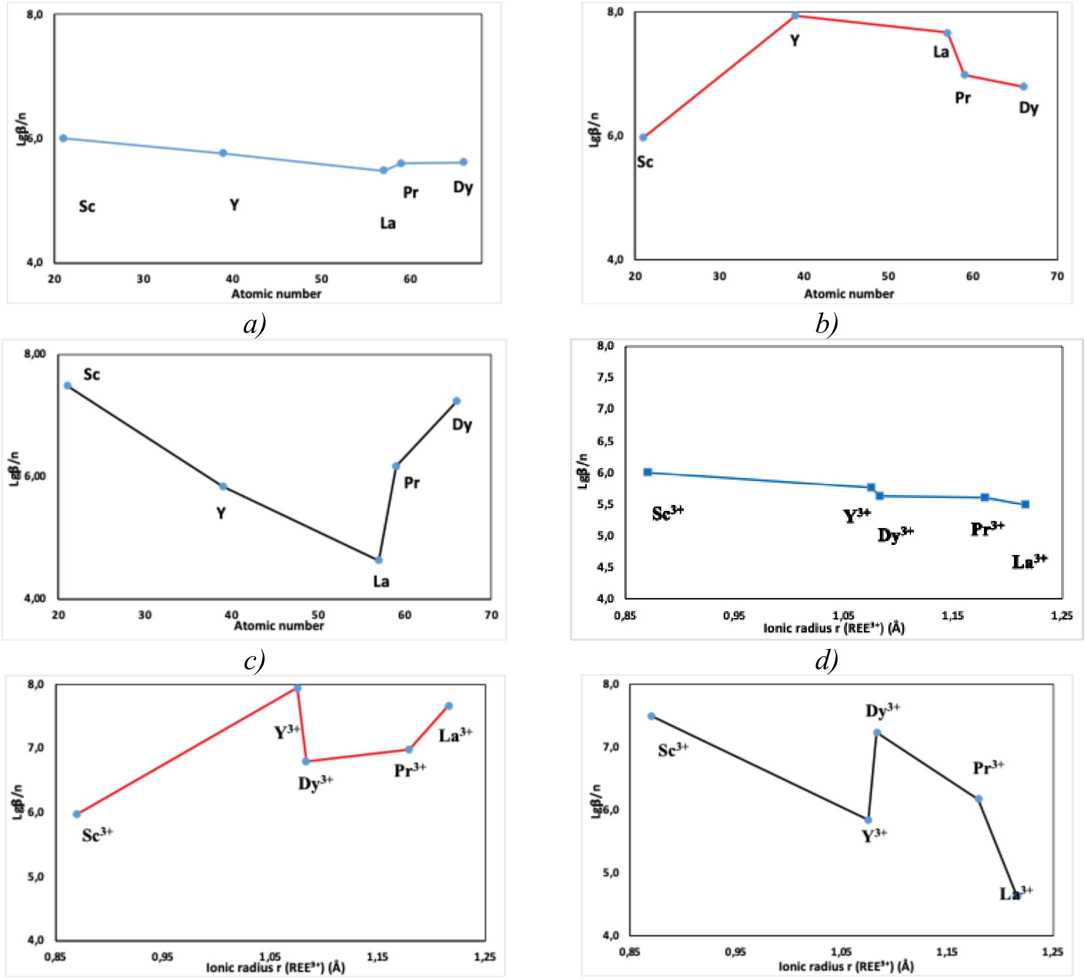
Ligand	Study method	Solvent	Ionic strength of the solution, mol/L	T, °C	pK_a	Ref.
L^1	Spectrophotometry	50% H_2O/EtOH	0,10 M $NaClO_4$	20	6,20 ± 0,02 9,65 ± 0,01	[10]
	Potentiometry	H_2O	0,2 M KCl	25	4,905 ± 0,006 11,94 ± 0,08	[18]
	Potentiometry	50% H_2O-EtOH	-	22	2,52 7,25	*
L^2	Potentiometry	H_2O	0,6 M NaCl	25	4,997 ± 0,001 10,87 ± 0,01	[18]
	Spectrophotometry	40% EtOH/H_2O	0,10 M $NaClO_4$	25	3,91 ± 0,02 9,22 ± 0,04	[19]
	Potentiometry	50% H_2O-EtOH	-	22	2,50 6,23	*

3.3. Correlation of metal compound formation constant and properties of the central atom and ligands

The variation in formation constants (log β) for complexes of Sc^{3+}, Y^{3+}, La^{3+}, Pr^{3+}, and Dy^{3+} with ligands L^1-L^3 can be explained by a combination of ionic radius, charge density (Z/r), donor atom type, and steric factors. The correlations presented in Fig.3a-f clearly illustrate these structure-stability relationships.

For ligands L^1 and L^3, a systematic increase in logβ/n with increasing nuclear charge is observed (Fig.3a, c), reflecting the strong affinity of hard oxygen donors (–OH and –COO-) toward metal ions with high charge density. This behavior is consistent with previous studies showing that 2Hnic and 2H6Mnic coordinate predominantly through hydroxyl and carboxylate oxygen atoms to form six-membered chelate rings with trivalent metal ions [7, 20]. Accordingly, L^1 and L^3 form their most stable complexes with the smallest and most charge-dense ions in the series, particularly Sc^{3+} and Dy^{3+}. An inverse correlation between ionic radius and complex stability (Fig.3d, f) further confirms that smaller REE^{3+} ions fit more favorably within the O-donor chelation environment.

Despite their similar coordination modes, L^3 exhibits larger variations in log β/n across the metal series than L^1. This effect arises from steric hindrance introduced by the methyl substituent at the 6-position, which restricts optimal chelate-ring geometry. The pronounced decrease in stability for the largest ion, La^{3+}, highlights the sensitivity of lanthanide complexation to subtle steric mismatches, even among closely related ligands.



a) *b)*

c) *d)*

e) *f)*

Figure 3. *Dependence of the formation constants (log β/n) of rare-earth metal complexes on atomic number and ionic radius, according to Shannon [21].*

In contrast, ligand L^2 (3Hpic) displays a distinct stability trend (Fig. 3b, e), governed primarily by coordination through the pyridine nitrogen donor. As a relatively softer donor compared with oxygen, the nitrogen atom preferentially stabilizes larger and less charge-dense cations. Consequently, L^2 forms more stable complexes with Y^{3+} and La^{3+} (log β/n = 7.95 and 7.67), while complexes with the smaller Sc^{3+} ion are significantly weaker (log β/n = 5.98). The weak dependence of L^2 stability on nuclear charge and its positive correlation with ionic radius are consistent with hard-soft acid-base (HSAB) principles [22] and with previous reports on nitrogen-donor ligands [23, 24].

Overall, these results highlight the decisive roles of donor atom identity and steric architecture in modulating rare-earth complex stability. The established correlations between ionic radius, ligand structure, and formation constants provide valuable guidelines for the rational design of selective ligands for rare-earth separation, as well as for applications in electronic materials and biomedical systems where controlled metal coordination is essential.

4. CONCLUSIONS

Spectrophotometric and potentiometric titrations were employed to elucidate the acid-base behavior and complexation properties of ligands L^1-L^3 with selected trivalent rare-earth ions. The determined dissociation constants (pK_a), complex stoichiometries (n), and formation constants (log β = 4.63 - 11.51) reveal a strong dependence of complex stability on metal ionic radius, charge density, donor atom type, and steric effects. Oxygen-donor ligands L^1 and L^3 preferentially stabilize small, charge-dense cations such as Sc^{3+} and Dy^{3+}, whereas the nitrogen-donor ligand L^2 exhibits higher affinity toward larger ions, including Y^{3+} and La^{3+}. The presence of a 6-methyl substituent in L^3 introduces steric constraints that reduce complex stability for larger ions, highlighting the sensitivity of lanthanide coordination to subtle ligand modifications. These quantitative structure-stability correlations provide a rational basis for designing selective ligands for rare-earth separation and functional coordination systems in electronic and biomedical applications. The study is limited to equilibrium measurements in aqueous–ethanol media and a restricted set of rare-earth ions; future work will extend to the full lanthanide series, fully aqueous systems, and kinetic investigations to evaluate practical applicability.

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TÓM TẮT

Nghiên cứu sự hình thành và độ bền của các phức Sc(III), La(III), Dy(III), Pr(III) và Y(III) với các axit hydroxypyridine-carboxylic trong dung dịch nước

Khả năng tạo phức của ba axit pyridinecarboxylic được thế hydroxyl gồm 2-hydroxypicolinic acid (L^1), 3-hydroxynicotinic acid (L^2) và 2-hydroxy-6-methylnicotinic acid (L^3) với các ion Sc^{3+}, Y^{3+}, La^{3+}, Pr^{3+} và Dy^{3+} đã được nghiên cứu một cách có hệ thống bằng các phương pháp chuẩn độ UV-vis và chuẩn độ thế. Quá trình khử proton từng bước của phối tử cho phép xác định hệ số phối trí và hằng số tạo phức ($\log \beta = 4,63 - 11,51$), qua đó cho thấy sự phụ thuộc rõ rệt của độ bền phức vào bán kính ion kim loại, mật độ điện tích và bản chất nguyên tử cho điện tử của phối tử. Các phối tử cho oxy L^1 và L^3 ưu tiên ổn định các cation nhỏ có mật độ điện tích cao (Sc^{3+}, Dy^{3+}), trong khi phối tử cho nitơ L^2 thể hiện ái lực cao hơn đối với các ion có bán kính lớn như Y^{3+} và La^{3+}. Nhóm metyl ở vị trí 6 trong L^3 gây cản trở không gian, làm giảm độ bền phức đối với các cation có kích thước lớn. Các kết quả này làm rõ vai trò tổng hợp của bản chất nguyên tử cho điện tử, hiệu ứng cản trở không gian và đặc tính của ion kim loại trong việc quyết định độ bền phức của các nguyên tố đất hiếm, đồng thời cung cấp cơ sở định lượng cho việc thiết kế hợp lý các phối tử chọn lọc và các hệ phối trí chức năng.

Từ khoá: Nguyên tố đất hiếm; Axit hydroxypyridine-carboxylic; Phức kim loại; Chuẩn độ UV-vis; Chuẩn độ điện thế.