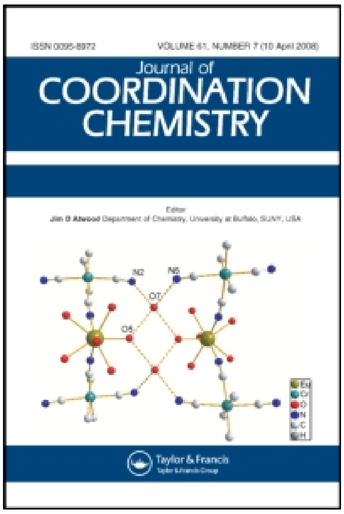

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La(III) and Eu(III) 2-D coordination polymers of 5-nitroisophthalic acid (H_2Nip) and 1,10-phenanthroline (phen), [M(phen)(HNip)(Nip)]*_n*

Hong-Ping Xiao[a]; Rahmatollah Rahimi[b]; Ya-Qian Cheng[a]; Ali Morsali[c]; Wei-Bing Zhang[a]; Jia-Guo Wang[a]
[a] School of Chemistry and Materials Science Engineering, Wenzhou University, Wenzhou, China [b] Faculty of Chemistry, Elmosanat University, Tehran, Iran [c] Faculty of Science, Department of Chemistry, Tarbiat Modares University, Tehran, Iran

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La(III) and Eu(III) 2-D coordination polymers of 5-nitroisophthalic acid (H_2Nip) and 1,10-phenanthroline (phen), [M(phen)(HNip)(Nip)]$_n$

HONG-PING XIAO*†, RAHMATOLLAH RAHIMI‡, YA-QIAN CHENG†, ALI MORSALI*§, WEI-BING ZHANG† and JIA-GUO WANG†

†School of Chemistry and Materials Science Engineering,
Wenzhou University, Wenzhou, China
‡Faculty of Chemistry, Elmosanat University, Tehran, Iran
§Faculty of Science, Department of Chemistry, Tarbiat Modares University,
P.O. Box 14155-4838, Tehran, Iran

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Lanthanum(III) and europium(III) complexes of 1,10-phenanthroline (phen) with 5-nitroisophthalate, [La(phen)(HNip)(Nip)]$_n$ (**1**) and [Eu(phen)(HNip)(Nip)]$_n$ (**2**), have been synthesized and characterized by elemental analysis and IR spectroscopy and studied by X-ray crystallography. The single crystal X-ray analyses show that both lanthanum(III) and europium(III) are coordinated by two nitrogens of phen and six oxygens from "Nip^{2-}" and "HNip^{-}", resulting in a distorted square antiprism.

Keywords: Lanthanum; Europium; 5-Nitroisophthalate; 1,10-Phenanthroline

1. Introduction

The interaction of metal ions with nucleic bases is of great interest because of their relevance to the bioactivity of metal centers [1]. Many benzenedicarboxylate (bdc^{2-}) coordination polymers with metal-organic frameworks have been synthesized and these coordination polymers have potential use in gas adsorption, chemical sensoring, and catalysis [2–12]. The "bdc^{2-}" ditopic ligand has two potentially equivalent coordinating carboxylates, thus the topologies in assembled networks constructed by this ligand are limited. The 5-nitroisophthalic acid (H_2Nip) ligand attracted our attention because of the potential structural chemistry of its multifunctional coordination modes, able to bind metal ions in different fashions via carboxylate and nitro oxygens. Several metal carboxylate coordination polymers with isophthalic acid and its derivative, 5-nitroisophthalic acid, have been reported [13, 14]. The nitro ($-NO_2$) as an electron-withdrawing group not only acts as a hydrogen bond acceptor, but can also take on spatial effects in the formation of polymeric networks [15]. Lanthanide ions are well

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*Corresponding authors. Email: hp_xiao@yahoo.com.cn; morsali_a@yahoo.com

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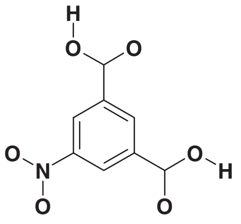


H_2Nip

Scheme 1. The ligand used in the construction of lanthanum(III) and europium(III) coordination polymer frameworks.

Table 1. Crystal data and structure refinement for **1** and **2**.

Identification code	**1**	**2**
Empirical formula	$C_{28}H_{15}LaN_4O_{12}$	$C_{28}H_{15}EuN_4O_{12}$
Formula weight	738.35	751.40
Temperature (K)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	10.4741(8)	10.2697(7)
b	11.1745(9)	12.0249(9)
c	13.1789(11)	12.1218(9)
α	102.034(1)	70.566(1)
β	111.613(1)	85.966(1)
γ	96.301(1)	76.636(1)
Volume (Å^3), Z	1372.70(19), 2	1373.40(17), 2
Calculated density (g cm^{-3})	1.786	1.817
Absorption coefficient (mm^{-1})	1.632	2.359
$F(000)$	728	740
Crystal size (mm^3)	0.24 × 0.23 × 0.15	0.33 × 0.17 × 0.13
θ range for data collection (°)	1.73–25.30	1.78–25.70
Limiting indices	$-12 \le h \le 12$; $-13 \le k \le 13$; $-15 \le l \le 15$	$-12 \le h \le 6$; $-14 \le k \le 14$; $-14 \le l \le 13$
Reflections collected	10,183	7632
Independent reflections	4963	5125
Absorption correction	Multi-scan	Multi-scan
Max. and min. transmission	0.7919 and 0.6955	0.732 and 0.619
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	4963/0/406	5125/0/407
Goodness-of-fit on F^2	1.075	1.066
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0276$, $wR_2 = 0.0774$	$R_1 = 0.0284$, $wR_2 = 0.0669$
R indices (all data)	$R_1 = 0.0278$, $wR_2 = 0.0776$	$R_1 = 0.0303$, $wR_2 = 0.0680$
Largest difference peak and hole (e Å^{-3})	0.598 and −0.494	0.986 and −0.551

known for their large radii and variable coordination number from 3 to 12, which make them excellent spacers in assembling metal-organic frameworks [13–23]. Some coordination polymers of transition metals and 5-nitroisophthalic acid have been prepared [24, 25]. In this work, we describe the syntheses and characterization of new lanthanum(III) and europium(III) complexes with 1,10-phenanthroline (phen),

Table 2. Bond lengths (Å) and angles (°) for **1** and **2**.

Compound **1**[a]		Compound **2**[b]	
La(1)–O(10)#1	2.394(3)	Eu(1)–O(2)#1	2.316(2)
La(1)–O(4)#2	2.445(2)	Eu(1)–O(7)	2.335(3)
La(1)–O(1)	2.482(2)	Eu(1)–O(1)	2.345(2)
La(1)–O(3)#3	2.488(2)	Eu(1)–O(8)#1	2.387(3)
La(1)–O(9)#4	2.519(2)	Eu(1)–O(3)#2	2.430(2)
La(1)–O(7)	2.687(2)	Eu(1)–O(4)#2	2.480(2)
La(1)–N(1)	2.703(3)	Eu(1)–N(2)	2.566(1)
La(1)–N(2)	2.725(3)	Eu(1)–N(1)	2.612(3)
O(10)#1–La(1)–O(4)#2	83.05(10)	O(2)#1–Eu(1)–O(1)	124.72(9)
O(10)#1–La(1)–O(1)	95.17(10)	O(2)#1–Eu(1)–O(7)	76.37(11)
O(4)#2–La(1)–O(1)	143.77(8)	O(7)–Eu(1)–O(1)	75.93(10)
O(10)#1–La(1)–O(3)#3	71.52(9)	O(2)#1–Eu(1)–O(8)#1	75.60(9)
O(4)#2–La(1)–O(3)#3	124.61(9)	O(7)–Eu(1)–O(8)#1	126.05(10)
O(1)–La(1)–O(3)#3	88.15(9)	O(1)–Eu(1)–O(8)#1	83.72(10)
O(10)#1–La(1)–O(9)#4	121.74(10)	O(2)#1–Eu(1)–O(3)#2	80.67(9)
O(4)#2–La(1)–O(9)#4	73.65(9)	O(7)–Eu(1)–O(3)#2	75.51(10)
O(1)–La(1)–O(9)#4	133.62(8)	O(1)–Eu(1)–O(3)#2	134.82(9)
O(3)#3–La(1)–O(9)#4	79.39(10)	O(8)#1–Eu(1)–O(3)#2	141.43(9)
O(10)#1–La(1)–O(7)	67.72(9)	O(2)#1–Eu(1)–O(4)#2	132.62(8)
O(4)#2–La(1)–O(7)	67.88(8)	O(7)–Eu(1)–O(4)#2	81.83(9)
O(1)–La(1)–O(7)	77.94(8)	O(1)–Eu(1)–O(4)#2	88.69(8)
O(3)#3–La(1)–O(7)	135.20(9)	O(8)#1–Eu(1)–O(4)#2	147.61(9)
O(9)#4–La(1)–O(7)	138.91(9)	O(3)#2–Eu(1)–O(4)#2	53.13(8)
O(10)#1–La(1)–N(1)	144.29(9)	O(2)#1–Eu(1)–N(2)	84.58(10)
O(4)#2–La(1)–N(1)	85.37(9)	O(7)–Eu(1)–N(2)	145.73(11)
O(1)–La(1)–N(1)	75.12(9)	O(1)–Eu(1)–N(2)	137.67(10)
O(3)#3–La(1)–N(1)	140.38(9)	O(8)#1–Eu(1)–N(2)	74.42(10)
O(9)#4–La(1)–N(1)	86.62(9)	O(3)#2–Eu(1)–N(2)	73.44(11)
O(7)–La(1)–N(1)	76.62(9)	O(4)#2–Eu(1)–N(2)	90.92(10)
O(10)#1–La(1)–N(2)	148.52(10)	O(2)#1–Eu(1)–N(1)	138.13(10)
O(4)#2–La(1)–N(2)	125.45(9)	O(7)–Eu(1)–N(1)	144.46(11)
O(1)–La(1)–N(2)	70.06(8)	O(1)–Eu(1)–N(1)	75.20(10)
O(3)#3–La(1)–N(2)	80.12(9)	O(8)#1–Eu(1)–N(1)	70.38(10)
O(9)#4–La(1)–N(2)	63.86(9)	O(3)#2–Eu(1)–N(1)	112.25(10)
O(7)–La(1)–N(2)	131.31(9)	O(4)#2–Eu(1)–N(1)	77.24(9)
N(1)–La(1)–N(2)	60.51(9)	N(2)–Eu(1)–N(1)	63.50(11)

[a] For compound **1**: #1 $-x, -y, -z$; #2 $x+1, y, z$; #3 $-x+1, -y+2, -z+2$; #4 $x, y-1, z$.
[b] For compound **2**: #1 $-x, -y, -z$; #2 $x-1, y, z$.

5-nitroisophthalate ligand, [La(phen)(HNip)(Nip)]$_n$ (**1**), and [Eu(phen)(HNip)(Nip)]$_n$ (**2**) (scheme 1).

2. Experimental

2.1. *Physical property measurements*

All reagents and solvents used for the synthesis and analysis were commercially available in high purity and used as received. IR spectra were recorded using Perkin–Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN–O– Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected.

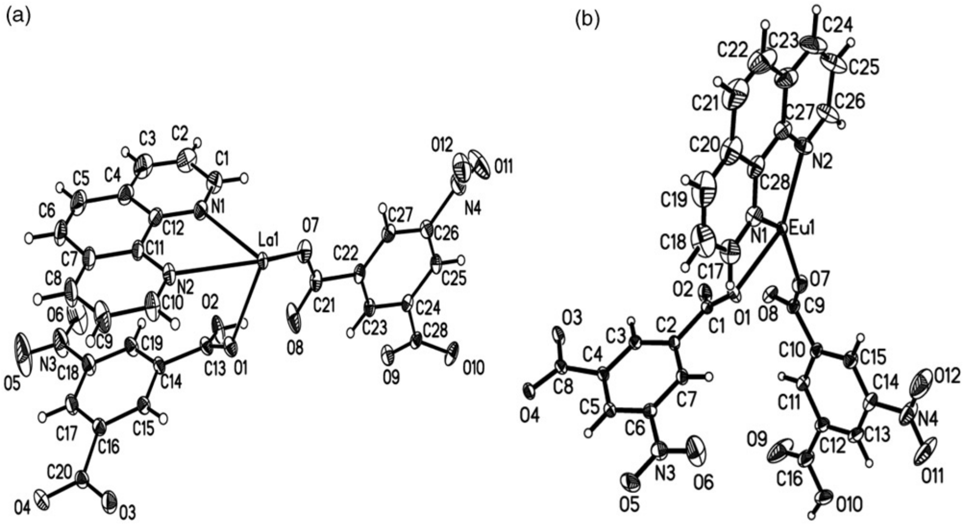


Figure 1. The ORTEP diagrams of: (a) $[La(phen)(HNip)(Nip)]_n$ (**1**) and (b) $[Eu(phen)(HNip)(Nip)]_n$ (**2**).

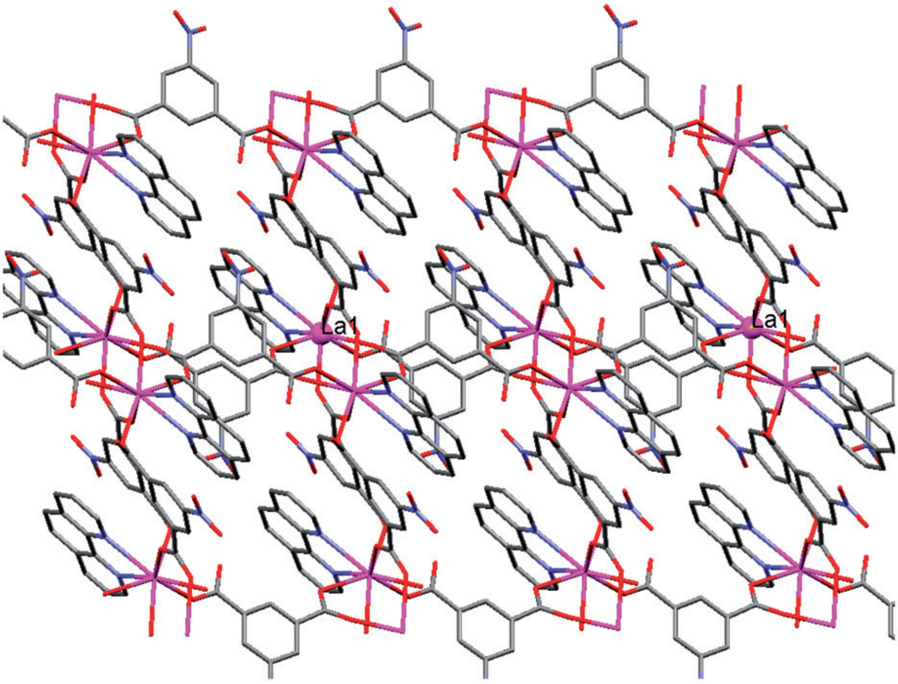


Figure 2. A perspective view of the 2-D coordination polymer in **1**, showing the coordination mode of Nip^{2-} and $HNip^{-}$.

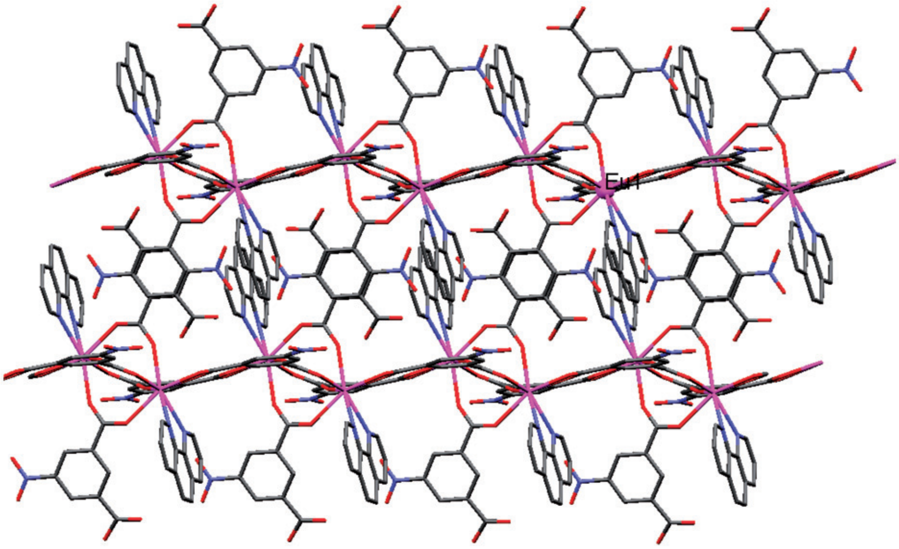


Figure 3. A perspective view of the 2-D coordination polymer in **2**, showing the coordination mode of Nip^{2-} and $HNip^{-}$.

2.2. *Syntheses of [La(phen)(HNip)(Nip)]$_n$ (1)*

$LaCl_3 \cdot 6H_2O$ (0.5 mmol), 5-nitroisophthalic acid (0.5 mmol), and 1,10-phenanthroline (1.0 mmol) were placed in a 30 mL Teflon-lined, stainless-steel Parr bomb with water (18 mL). The bomb was heated at 403 K for 3 days and cooled slowly to room temperature to furnish colorless crystals. Anal. Calcd for $C_{28}H_{15}N_4O_{12}La$ (%): C, 45.55; H, 2.05; N, 7.59. Found (%): C, 45.47; H, 1.97; N, 7.52. IR (cm^{-1}) selected bands: $\nu = 3095$ w, 1685 m, 1627 s, 1538 s, 1453 s, 1376 vs, 1348 s, 1204 w, 1136 m, 1097 m, 924 w, 841 s, 789 m, 726 m, and 536 m.

2.3. *Syntheses of [Eu(phen)(HNip)(Nip)]$_n$ (2)*

$Eu(NO_3)_3 \cdot 6H_2O$ (0.5 mmol), 5-nitroisophthalic acid (0.5 mmol), and 1,10-phenanthroline (1.0 mmol) were placed in a 30 mL Teflon-lined, stainless-steel Parr bomb with water (18 mL). The bomb was heated at 423 K for 5 days and cooled slowly to room temperature to furnish colorless crystals. Anal. Calcd for $C_{28}H_{15}N_4O_{12}Eu$ (%): C, 44.71; H, 1.92; N, 7.38. Found (%): C, 45.10; H, 1.52; N, 7.89. IR (cm^{-1}) selected bands: $\nu = 3097$ w, 1687 m, 1627 s, 1539 s, 1456 s, 1378 vs, 1349 s, 1205 w, 1137 m, 1099 m, 925 w, 841 s, 789 m, 726 m, and 537 m.

2.4. *Crystallography*

X-ray measurements were made at 298(2) using a Siemens R3m/V diffractometer. The intensity data were collected within the range of 1.73°–25.30° and 1.78°–25.70° using graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) for **1** and **2**. Accurate unit cell parameters and an orientation matrix for data collection were obtained from the

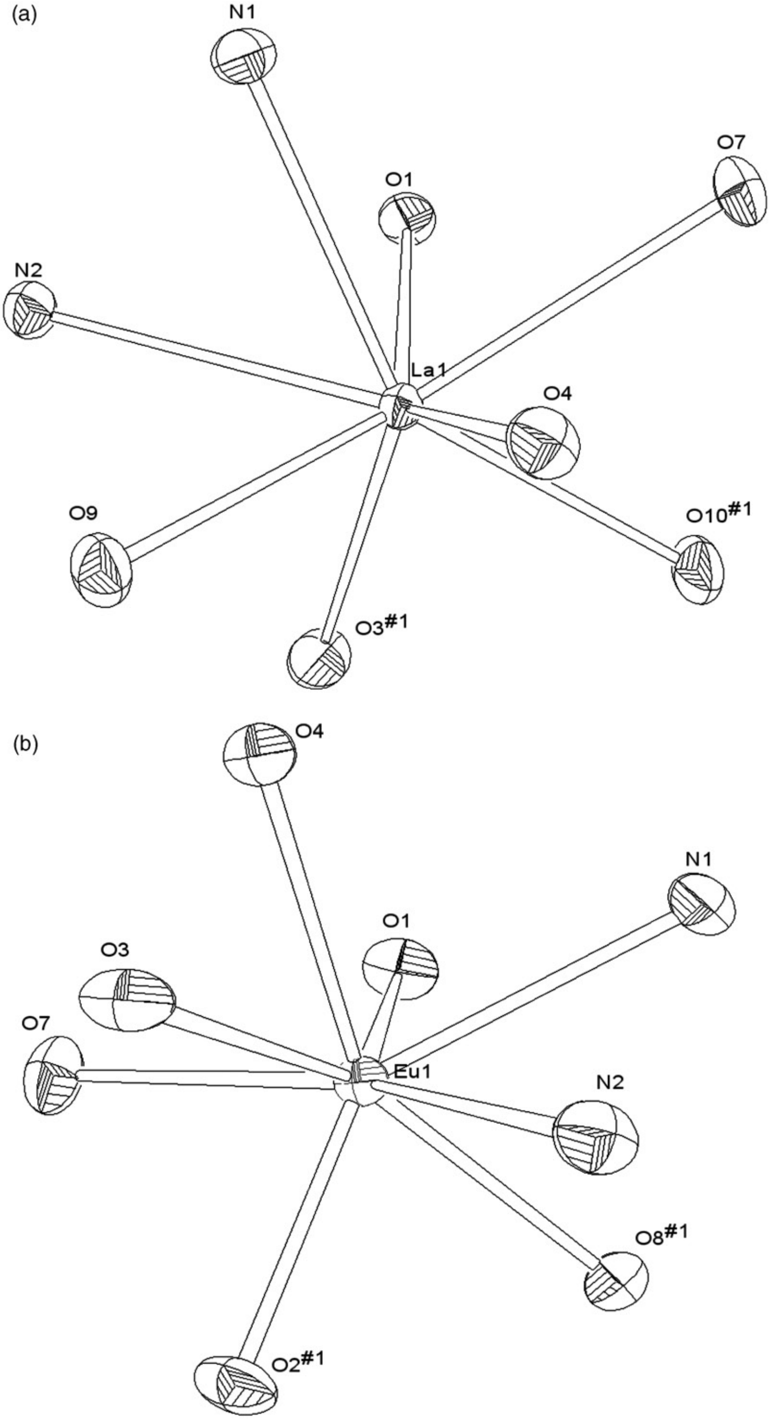


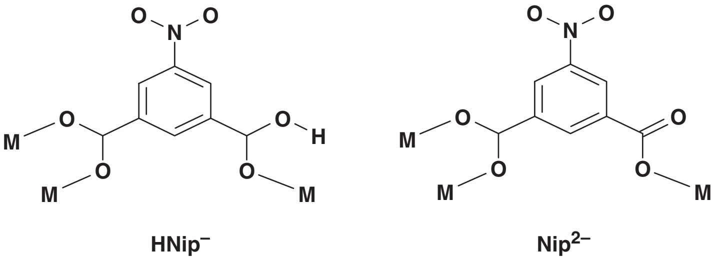
Figure 4. Environment of La- and Eu-atoms in: (a) (**1**) and (b) (**2**). #1: $-x$, $-y$, $-z$.

least-squares refinement. The structures were solved by direct methods and refined by full-matrix least-squares on F^2. Crystal data and refinement parameters are given in table 1 and selected bond lengths and angles in table 2. ORTEP diagrams and perspective views of the packing are shown in figures 1–3.

3. Results and discussion

Reaction between 1,10-phenanthroline and mixtures of lanthanum(III) chloride hexahydrate and europium(III) nitrate hexahydrate with 5-nitroisophthalic acid in water provided crystalline materials [La(phen)(HNip)(Nip)]$_n$ (**1**) and [Eu(phen)(HNip)(Nip)]$_n$ (**2**). The IR spectra and CHN analyses showed that 1,10-phenanthroline, and "Nip^{2-}" and "HNip$^-$", are coordinated. IR spectrum displays absorption bands for "H_2Nip" and 1,10-phenanthroline molecules in **1** and **2**. The relatively weak absorptions around 3097 cm^{-1} are due to C–H modes involving hydrogens in the aromatic ring. Absorption bands with variable intensity at 1400–1650 cm^{-1} correspond to ring vibrations of Ph and py of "Nip^{2-}", "HNip$^-$", and 1,10-phenanthroline. The characteristic bands of carboxylate groups appear at 1586 $\nu_{as(C–O)}$ and 1376 $\nu_{sym(C–O)}$ cm^{-1} for **1** and at 1538 $\nu_{as(C–O)}$ and 1378 $\nu_{sym(C–O)}$ cm^{-1} for **2**. Single X-ray crystal analysis reveals that **1** and **2** crystallize in the triclinic system with space group P$\bar{1}$. The structures of these complexes may be considered as 2-D polymers of lanthanum(III) and europium(III), respectively, bridged by both "HNip$^-$" and "Nip^{2-}", forming 2-D infinite frameworks (figures 1–3). In **1** and **2** each metal is coordinated by two nitrogens from "phen" and six carboxylate oxygens from "HNip$^-$" and "Nip^{2-}", resulting in a distorted square antiprism (figure 4). The coordination modes of "HNip$^-$" and "Nip^{2-}" in **1** and **2** are the same, with one carboxylate of "HNip$^-$" and "Nip2$^-$" bidentate bridging where two oxygens of carboxylate coordinate to two metals. In the second mode, the carboxylates of "HNip$^-$" and "Nip^{2-}" are unidentate (scheme 2).

In **1** and **2**, individual 2-D units interact via O $\cdots$ HO hydrogen bonds (tables 3 and 4) and π–π stacking between the aromatic rings of "HNip$^-$". The phenyl groups of "HNip$^-$" in **1** and **2** are almost parallel for face-to-face "π–π stacking" [26, 27] (figure 4b). The centroid to centroid distances of two planes are 3.662(8) and 3.727(7) Å, respectively.



HNip$^-$ **Nip^{2-}**

M = La and Eu

Scheme 2. Coordination mode of HNip$^-$ and Nip^{2-} in **1** and **2**.

Table 3. Hydrogen bonds for **1** (Å and °).

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
O(2)–H(2A) ···O(8)	0.82	1.61	2.428(4)	174.1

Table 4. Hydrogen bonds for **2** (Å and °).

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
O(10)–H(10)···O(4)#1	0.82	2.04	2.731(4)	142.2

Symmetry transformations used to generate equivalent atoms: #1: $-x+3$, $-y$, $-z+1$.

4. Conclusions

Two new lanthanum(III) and europium(III) complexes of 1,10-phenanthroline (phen) with 5-nitroisophthalate, $[La(phen)(HNip)(Nip)]_n$, and $[Eu(phen)(HNip)(Nip)]_n$, are reported. The compounds are structurally similar showing 2-D polymer of lanthanum(III) and europium(III) bridged by the "HNip^{-}" and "Nip^{2-}" ions. There are some reports discussing the preparation of lanthanide coordination polymers with 2,4-difluorobenzoate and 2-bromobenzoate [28], 1,3-benzenedicarboxylate [29], and oxydiacetic acid [30]. It seems that in contrast to other previously reported lanthanide coordination polymers of carboxylates [28–30], **1** and **2** contain both "HNip^{-}" and "Nip^{2-}" resulting in the formation of 3-D supramolecular compounds, and "HNip^{-}" and "Nip^{2-}" act both as acceptors and donors for hydrogen bonds. This research shows the significant influences of the coordination modes of "HNip^{-}" and "Nip^{2-}" on the structural topology and little influence on nature of metal, here La(III) and Eu(III).

Supplementary material

Crystallographic data for the structures reported in the article have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no, CCDC-659596 for **1** and CCDC-659595 for **2**. Copies of the data can be obtained on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: +44-1223/336033; E-mail: deposit@ccdc.cam.ac.uk].

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