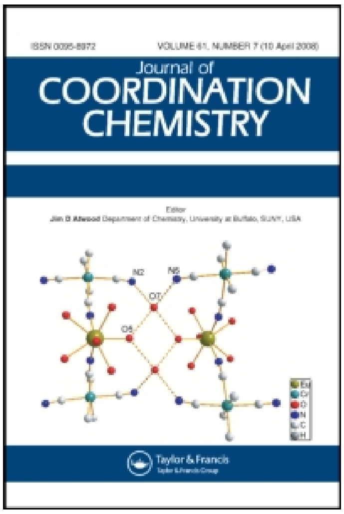

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Novel lanthanide complexes of 5-nitroisophthalic acid containing two kinds of π–π stacking interactions

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The novel two-dimensional coordination polymers $[Ln(5\text{-}nip)(5\text{-}Hnip)(H_2O)_2](H_2O)_2$ (Ln = Sm (**1**), Gd (**2**), Dy (**3**); $5\text{-}nipH_2$ = 5-nitroisophthalic acid) were prepared using hydrothermal methods. Their isomrphous structures were characterized by single-crystal X-ray diffraction analyses. Two-dimensional layer-like structures are constructed by lanthanide ions bridged by $5\text{-}nip^{2-}$ ligands with the layers packing in 3D motifs through two kinds of π–π stacking interactions and hydrogen bonds. The complexes are thermally stable, aside from loss of water, to ca 350°C.

Keywords: 5-Nitroisophthalic acid; Lanthanide(III); Crystal structure

1. Introduction

The rational design and synthesis of new discrete or polymeric metal-organic coordination architectures for their potential use as functional materials is an active area of research [1–3]. Coordination architectures depend mainly on the coordination geometry of the metal ions and the nature of the ligands [4]. For simple carboxylic acid ligands, many different geometries are known, even for the lanthanides. For example, for $Ln(Gly)_3Cl_3 \cdot H_2O$ (Ln = La, Pr, Gd, Yb; Gly = glycine) [5], the coordination number of lanthanide(III) ions decreases from ten (La) to nine (Pr, Gd) to eight (Yb) owing to the lanthanide contraction effect. Similar to isophthalic acid and its other 5-substituted derivatives, 5-nitroisophathalic acid is a simple rigid ligand with two carboxyl groups, which may engage in numerous coordination modes. The nitro group acts as an electron-withdrawing fragment, enhancing the π stacking that stabilizes the complexes [6]. From a survey of reported transitional metal complexes of 5-nitroisophathalic acid [7], ten coordination modes are discerned (scheme 1). Since few complexes are isostructural, we were led to consider the nature of its lanthanide complexes. Herein, we report structures of Sm(III), Gd(III) and Dy(III) complexes of 5-nitroisophthalic acid.

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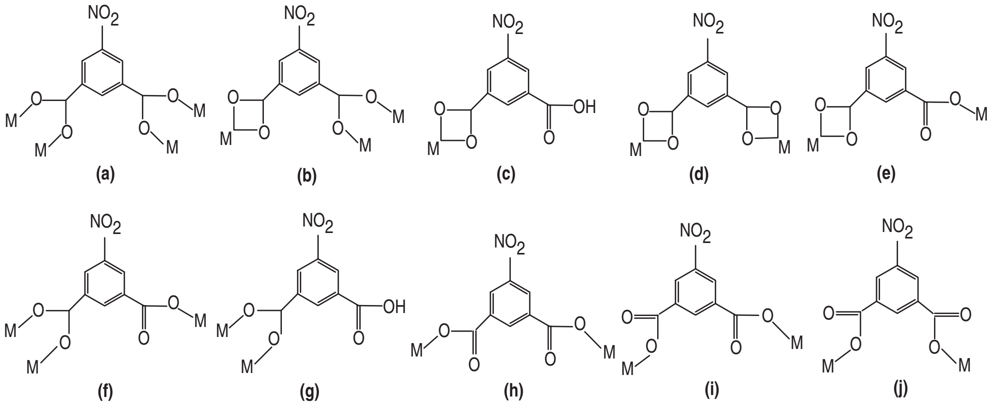


Scheme 1. Reported coordination modes of 5-nitroisophathalic acid.

2. Experimental

2.1. *Materials and methods*

All chemicals were obtained from commercial sources and used without further purification. Elemental analyses were performed on an Elemental Vario EL III CHNOS system. IR spectra (KBr pellets) were recorded with a BEQ VZNDX-550 FT-IR spectrophotometer. TG-DSC curves were obtained with a Perkin Elmer thermogravimetric analyzer in an air atmosphere with a heating rate of 10°C min^{-1}.

2.2. *Syntheses*

[Sm(5-nip)(5-Hnip)(H_2O)$_2$](H_2O)$_2$ (**1**): In a typical experiment, 5-H_2nip (0.0422 g, 0.2 mmol) in an aqueous solution (6 cm^3) of NaOH (0.016 g, 0.4 mmol) was mixed with an aqueous solution (2 cm^3) of $Sm(NO_3)_3 \cdot 6H_2O$ (0.0888 g, 0.2 mmol). After stirring for 30 min at room temperature the mixture was transferred to a 15 cm^3 Teflon-lined autoclave and heated at 155°C for 168 h. The autoclave was cooled over a period of 12 h at a rate of 5°C h^{-1}. Yellow prismatic crystals of the product were collected by filtration, washed with water, and dried in air (ca 36% yield, based on Sm). Anal. Calcd for $C_{16}H_{15}N_2O_{16}Sm$ (%): C, 29.95; H, 2.36; N, 4.37. Found: C, 30.10; H, 1.91; N, 4.28. IR (cm^{-1}): 3567(w), 3376(w), 3081(m), 1629(m), 1549(s), 1462(s), 1392(s), 1350(s), 931(w), 734(s).

[Gd(5-nip)(5-Hnip)(H_2O)$_2$](H_2O)$_2$ (**2**): The procedure was the same except that $Gd(NO_3)_3 \cdot 6H_2O$ (0.0891 g, 0.2 mmol) was substituted for $Sm(NO_3)_3 \cdot 6H_2O$. Colourless prismatic crystals were collected by filtration, washed with water, and dried in air (ca 40% yield, based on Gd). Anal. Calcd for $C_{16}H_{15}N_2O_{16}Gd$ (%): C, 29.63; H, 2.33; N, 4.32. Found: C, 29.95; H, 1.87; N, 4.18. IR (cm^{-1}): 3565 (w), 3373 (w), 3082 (m), 1629 (m), 1552 (s), 1462 (s), 1395 (s), 1349 (s), 932(w), 734 (s).

[Dy(5-nip)(5-Hnip)(H_2O)$_2$](H_2O)$_2$ (**3**): The procedure was the same except that $Dy(NO_3)_3 \cdot 6H_2O$ (0.0913 g, 0.2 mmol) substituted for $Sm(NO_3)_3 \cdot 6H_2O$. Colourless prismatic crystals were collected by filtration, washed with water, and

Table 1. Crystallographic data and experimental details for structural analyses of the complexes.

Complex	**1**	**2**	**3**
Empirical formula	$C_{16}H_{15}N_2O_{16}Sm$	$C_{16}H_{15}N_2O_{16}Gd$	$C_{16}H_{15}N_2O_{16}Dy$
Formula weight	641.65	648.55	653.80
T (K)	273(2)	273(2)	273(2)
Crystal system	Triclinic	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$
a (Å)	9.6894 (9)	9.6834 (2)	9.637 (3)
b (Å)	10.5183 (1)	10.4879 (2)	10.431 (3)
c (Å)	13.5770 (1)	13.6043 (2)	13.585 (4)
α (°)	69.9480 (1)	67.5660 (1)	112.537 (5)
β (°)	70.1000 (1)	69.7990 (1)	91.310 (5)
γ (°)	65.1870 (1)	65.0960 (1)	114.953 (4)
V (Å^3)	1136.27 (2)	1129.89 (4)	1115.6 (6)
Z	2	2	2
F(000)	630	634	638
D_{Calcd} (Mg m^{-3})	1.875	1.906	1.946
Absorption coefficient (mm^{-1})	2.667	3.018	3.434
Crystal size (mm^3)	$0.56 \times 0.33 \times 0.28$	$0.15 \times 0.12 \times 0.10$	$0.41 \times 0.34 \times 0.20$
Range hkl collected	$-11 \leq h \leq 11$, $-12 \leq k \leq 8$, $-16 \leq l \leq 16$	$-12 \leq h \leq 8$, $-13 \leq k \leq 13$, $-17 \leq l \leq 17$	$-11 \leq h \leq 11$, $-8 \leq k \leq 12$, $-16 \leq l \leq 13$
Reflections collected	6063	14471	5852
Independent reflections	4340 [$R_{\text{int}} = 0.0116$]	5176 [$R_{\text{int}} = 0.0182$]	4208 [$R_{\text{int}} = 0.0416$]
Completeness to θ	97.5%	97.7%	95.9%
Data/restraints/parameters	4340/0/317	5176/0/317	4208/0/317
Goodness-of-fit on F^2	1.038	1.097	1.054
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0210$, $wR_2 = 0.0556$	$R_1 = 0.0177$, $wR_2 = 0.0472$	$R_1 = 0.0789$, $wR_2 = 0.2114$
R indices (all data)	$R_1 = 0.0226$, $wR_2 = 0.0573$	$R_1 = 0.0189$, $wR_2 = 0.0476$	$R_1 = 0.0815$, $wR_2 = 0.2135$
Largest different peak and hole (e Å^{-3})	0.990 and −1.081	0.544 and −0.793	6.287 and −4.767

dried in air (ca 33% yield, based on Dy). Anal. Calcd for $C_{16}H_{15}N_2O_{16}Dy$ (%): C, 29.39; H, 2.31; N, 4.28. Found: C, 29.60; H, 1.83; N, 4.20. IR (cm^{-1}): 3556(w), 3386(w), 3083(m), 1604(m), 1554(s), 1462(s), 1396(s), 1351(s), 932(w), 735(s).

2.3. *X-ray crystallography*

Data were collected on an Apple II Smart-CCD diffractometer with graphite-monochromated Mo-Kα ($\lambda = 0.71073$ Å) radiation in the phi and omega scanning mode at 273 K. The structures were solved by direct methods. The lanthanide and some oxygen atoms were first located and remaining carbon, nitrogen and oxygen atoms were found in a different Fourier map. H atoms attached to carbon were positioned geometrically. Structures were refined on F^2 by full-matrix least-squares methods using the SHELXL-97 package [8]. All non-hydrogen atoms were refined anisotropically. Crystallographic data and other experimental details are given in table 1.

3. Results and discussion

Selected bond lengths and angles of the three isomorphous complexes are listed in table 2. The structure of **1** is described here representatively. A Sm(III) ion,

Table 2. Selected bond lengths (Å) and angles (°) for the complexes.

$C_{16}H_{15}N_2O_{16}Sm$ (**1**)		$C_{16}H_{15}N_2O_{16}Gd$ (**2**)		$C_{16}H_{15}N_2O_{16}Dy$ (**3**)	
Sm(1)–O(7)	2.3119 (2)	Gd(1)–O(4)	2.2896 (2)	Dy(1)–O(8)	2.260 (6)
Sm(1)–O(10)	2.378 (2)	Gd(1)–O(2)	2.3421 (2)	Dy(1)–O(10)	2.316 (6)
Sm(1)–O(8)	2.3919 (2)	Gd(1)–O(3)	2.3654 (2)	Dy(1)–O(7)	2.338 (7)
Sm(1)–O(9)	2.4224 (2)	Gd(1)–O(1)	2.3979 (2)	Dy(1)–O(9)	2.365 (6)
Sm(1)–O(1)	2.450 (2)	Gd(1)–O(1)	2.4239 (2)	Dy(1)–O(14)	2.390 (7)
Sm(1)–O(1)	2.452 (2)	Gd(1)–O(1)	2.4298 (2)	Dy(1)–O(13)	2.397 (7)
Sm(1)–O(1)	2.498 (2)	Gd(1)–O(8)	2.4790 (2)	Dy(1)–O(1)	2.452 (7)
Sm(1)–O(2)	2.530 (2)	Gd(1)–O(7)	2.5001 (2)	Dy(1)–O(2)	2.474 (7)
O(7)–Sm(1)–O(10)	89.40 (8)	O(4)–Gd(1)–O(2)	89.42 (6)	O(8)–Dy(1)–O(10)	89.2 (3)
O(7)–Sm(1)–O(8)	102.24 (7)	O(4)–Gd(1)–O(3)	102.76 (6)	O(8)–Dy(1)–O(7)	103.5 (2)
O(10)–Sm(1)–O(8)	147.15 (8)	O(2)–Gd(1)–O(3)	147.14 (7)	O(10)–Dy(1)–O(7)	146.8 (3)
O(7)–Sm(1)–O(9)	153.79 (8)	O(4)–Gd(1)–O(1)	154.30 (7)	O(8)–Dy(1)–O(9)	155.2 (3)
O(10)–Sm(1)–O(9)	107.48 (7)	O(2)–Gd(1)–O(1)	106.15 (6)	O(10)–Dy(1)–O(9)	105.0 (2)
O(8)–Sm(1)–O(9)	74.52 (7)	O(3)–Gd(1)–O(1)	75.25 (6)	O(7)–Dy(1)–O(9)	75.8 (2)
O(7)–Sm(1)–O(13)	73.88 (8)	O(4)–Gd(1)–O(13)	73.99 (7)	O(8)–Dy(1)–O(14)	74.8 (3)
O(10)–Sm(1)–O(13)	139.69 (8)	O(2)–Gd(1)–O(13)	139.66 (7)	O(10)–Dy(1)–O(14)	140.1 (3)
O(8)–Sm(1)–O(13)	73.11 (9)	O(3)–Gd(1)–O(13)	73.18 (7)	O(7)–Dy(1)–O(14)	73.1 (3)
O(9)–Sm(1)–O(13)	80.42 (8)	O(1)–Gd(1)–O(13)	81.07 (6)	O(9)–Dy(1)–O(14)	81.5 (3)
O(7)–Sm(1)–O(14)	86.40 (8)	O(4)–Gd(1)–O(14)	86.83 (7)	O(8)–Dy(1)–O(13)	87.0 (3)
O(10)–Sm(1)–O(14)	71.65 (7)	O(2)–Gd(1)–O(14)	71.45 (6)	O(10)–Dy(1)–O(13)	71.7 (3)
O(8)–Sm(1)–O(14)	138.86 (8)	O(3)–Gd(1)–O(14)	138.67 (7)	O(7)–Dy(1)–O(13)	138.5 (3)
O(9)–Sm(1)–O(14)	80.39 (8)	O(1)–Gd(1)–O(14)	79.24 (6)	O(9)–Dy(1)–O(13)	78.6 (3)
O(13)–Sm(1)–O(14)	70.93 (8)	O(13)–Gd(1)–O(14)	71.14 (7)	O(14)–Dy(1)–O(13)	71.2 (3)
O(7)–Sm(1)–O(1)	125.88 (7)	O(4)–Gd(1)–O(8)	126.50 (6)	O(8)–Dy(1)–O(1)	126.8 (3)
O(10)–Sm(1)–O(1)	74.37 (7)	O(2)–Gd(1)–O(8)	74.19 (6)	O(10)–Dy(1)–O(1)	73.9 (2)
O(8)–Sm(1)–O(1)	73.94 (8)	O(3)–Gd(1)–O(8)	74.07 (6)	O(7)–Dy(1)–O(1)	74.0 (2)
O(9)–Sm(1)–O(1)	78.90 (7)	O(1)–Gd(1)–O(8)	78.19 (6)	O(9)–Dy(1)–O(1)	77.4 (2)
O(13)–Sm(1)–O(1)	144.68 (8)	O(13)–Gd(1)–O(8)	144.68 (7)	O(14)–Dy(1)–O(1)	144.3 (3)
O(14)–Sm(1)–O(1)	132.21 (7)	O(14)–Gd(1)–O(8)	131.16 (6)	O(13)–Dy(1)–O(1)	130.6 (2)
O(7)–Sm(1)–O(2)	74.85 (7)	O(4)–Gd(1)–O(7)	75.11 (6)	O(8)–Dy(1)–O(2)	75.1 (3)
O(10)–Sm(1)–O(2)	80.96 (7)	O(2)–Gd(1)–O(7)	80.71 (6)	O(10)–Dy(1)–O(2)	80.8 (3)
O(8)–Sm(1)–O(2)	72.87 (8)	O(3)–Gd(1)–O(7)	73.37 (6)	O(7)–Dy(1)–O(2)	73.3 (3)
O(9)–Sm(1)–O(2)	126.58 (7)	O(1)–Gd(1)–O(7)	126.76 (6)	O(9)–Dy(1)–O(2)	126.6 (2)
O(13)–Sm(1)–O(2)	126.77 (8)	O(13)–Gd(1)–O(7)	127.25 (6)	O(14)–Dy(1)–O(2)	127.3 (3)
O(14)–Sm(1)–O(2)	146.89 (7)	O(14)–Gd(1)–O(7)	146.91 (6)	O(13)–Dy(1)–O(2)	147.3 (2)
O(1)–Sm(1)–O(2)	52.03 (6)	O(8)–Gd(1)–O(7)	52.40 (6)	O(1)–Dy(1)–O(2)	52.9 (2)

a bis(bidentate) 5-nip^{2-} ligand (mode (a)), a mono(chelate) protonated 5-Hnip^{-} ligand (mode (c)), two coordinated and two lattice water molecules are present in the asymmetric unit (figure 1). Each samarium ion is coordinated by eight oxygen atoms; four of them (O7A, O8, O9A and O10A) are from four bis(bidentate) 5-nip^{2-} ligands, two (O1 and O2) from a chelating carboxyl group of a mono(chelate) protonated 5-Hnip^{-} ligand and two from coordinated water molecules (O13 and O14) give rise to a distorted Archimedean antiprismatic geometry (one face comprises O1, O9A, O14 and O10A, and the other O2, O8, O13 and O7A; figure 2). Sm-O bond lengths range from 2.3119(2) to 2.530(2) Å, with a mean of 2.429 Å (table 3). Each pair of adjacent Sm(III) ions (Sm$\cdots$Sm is about 5.0 Å) are bridged by two μ_2 carboxyl groups of two bis(bidentate) 5-nip^{2-} ligands (scheme 1a) to form eight-membered rings that link into 1D chains. Adjacent chains are joined by bis(bidentate) 5-nip^{2-} ligands alternately, alternately through different carboxyl groups (Sm$\cdots$Sm is about 8.9 Å), into a 2D layer parallel to the *ab* plane (figure 3). A π–π stacking interaction between the alternating

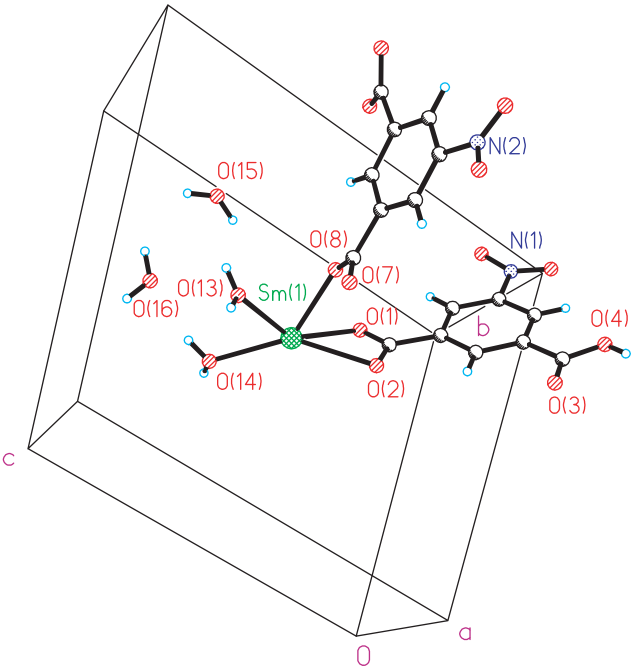


Figure 1. The independent crystallographic unit for complex **1**.

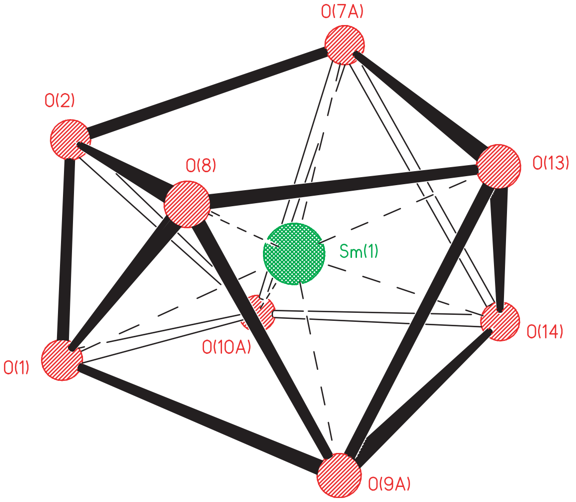


Figure 2. The coordination polyhedron of the Sm(III) ion.

Table 3. Ionic radii and mean Ln–O lengths of the complexes.

	1	**2**	**3**
Ionic radii (pm)	107.9	105.3	102.7
Mean Ln–O lengths (Å)	2.429	2.403	2.374

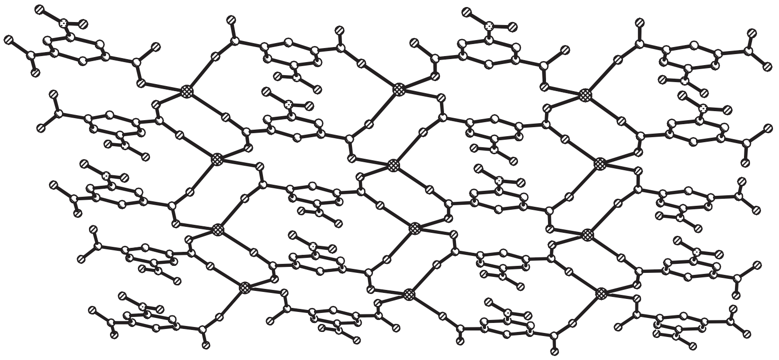


Figure 3. Arrangement of the 2D net in complex **1** parallel to the *ab* plane.

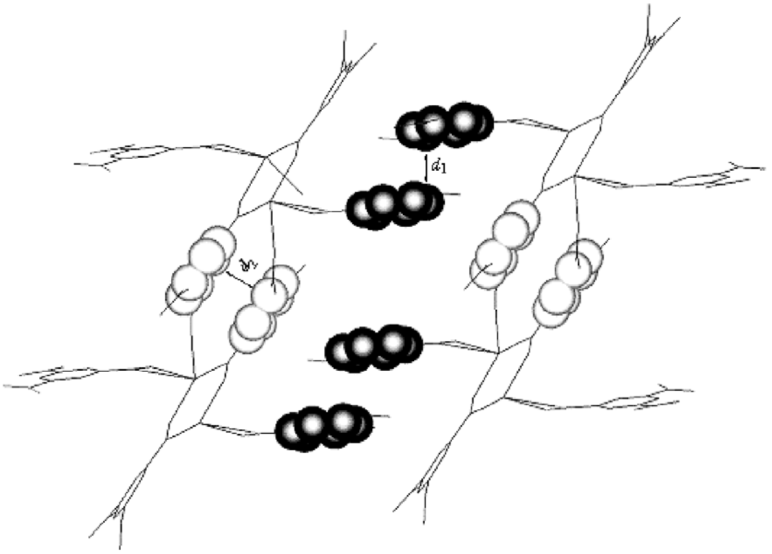
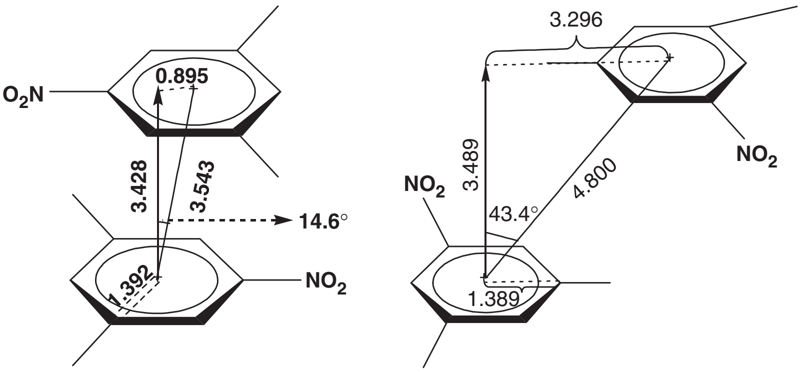


Figure 4. The two kinds of two kinds of π–π stacking interactions in the lattice (d_1 is the stack between layers and d_2 is the intra-layer stack).

5-nip^{2-} ligands has a face-to-face distance of 3.428 Å, and mono(chelate) 5-Hnip$^-$ ligands (scheme 1c) and coordinated water molecules form hydrogen bonds on both sides of the layer, further enhancing the stability of the structure. For 5-Hnip$^-$ ligands, two kinds of hydrogen bond exist in neighboring layers and an oxygen atom on the nitro group takes part in another hydrogen bond to give a 3D structure. Packing is further stabilized by other H-bonds and another kind of π–π stacking interaction along the *c* axis (face-to-face distance 3.489 Å) between 5-Hnip$^-$ ligands of adjacent layers.

The two kinds of π–π stacking interactions (figure 4) and their parameters in **1** are illustrated in scheme 2. The nitro group enhances the π–π interactions [6]. For case (1), the displacement angle is 14.6° and the offset displacement is 0.895 Å; for (2) the angle is 43.4° and the displacement is 3.296 Å. The bond in (1) should be stronger than that in (2). Distances involving π–π stacking for **1–3** are listed in table 4.



Scheme 2. Schematic illustration of π–π stacking interactions in **1**.

Table 4. Distances involved in π–π stacking in the complexes.

1		**2**		**3**	
In a layer	Between layers	In a layer	Between layers	In a layer	Between layers
3.428	3.489	3.421	3.494	3.390	3.478

Table 5. Thermal decomposition data for the complexes.

	Dehydration weight loss (%)		Decomposition weight loss (%)	
	Stage one	Stage two	Stage three	Stage four
Samples	Exp.(Calcd) T(°C)	Exp.(Calcd) T(°C)	Exp.(Calcd) T(°C)	Exp.(Calcd) T(°C)
1	8.40(8.42) 38–76–152	2.94(2.81) 152–200–244	14.03(13.77) 244–367–408	48.10(47.82) 408–447–563
2	8.06(8.33) 39–86–140	2.78(2.56) 140–207–246	11.95(12.68) 244–369–399	52.35(52.18) 399–445–607
3	8.74(8.27) 32–94–122	2.76(2.75) 122–219–277	8.99 (8.53) 277–331–400	51.27(51.92) 400–450–466

The three complexes are isomorphous and this implies that their formation is little affected by the radii of the metal ions or the lanthanide contraction effect. Ln–O bond lengths give a linear relationship against ionic radii and from this we may predict mean REE–O bond lengths for Eu and Tb analogues of 2.413 and 2.385 Å, respectively. Distances involving π–π stacking interactions are related also to ionic radii by smooth curves. However, in this case the 4f electronic cloud enlargement effect of lanthanide ions results in non-linear changes in π–π stacking distances.

The thermal stabilities of the complexes were measured from 30 to 800°C. Decomposition proceeds in four stages (table 5). In the first three water molecules are lost and in the second, the final one water molecule. Products checked by IR at this stage spectrum showed that the OH stretches at 3567 (**1**), 3545 (**2**) and 3556 $\mathrm{cm^{-1}}$ (**3**) had disappeared. Subsequent decomposition of the remaining ligands proceeds in two steps to give REE_2O_3 at 450–610°C, as confirmed by EDTA titration. It is noteworthy that the complexes are thermally stable, aside from loss of water, to ca 350°C.

Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos 294552, 294553 and 294556. Copies of this information can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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