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Hydrothermal synthesis and crystal structure of neodymium(III) coordination polymers with isophthalic acid and 1,10-phenanthroline

Li-Ping Zhang [a,b], Yong-Hong Wan [a], Lin-Pei Jin [a,*]

[a] *Department of Chemistry, Beijing Normal University, Beijing 100875, People's Republic of China*
[b] *Department of Chemistry, Anyang Teacher College, Anyang, Henan Province 455000, People's Republic of China*

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Abstract

Two polymeric complexes, $[Nd_2(bdc)_2(Hbdc)_2(H_2O)]_n \cdot nH_2O$ (**1**) and $[Nd_2(bdc)_3(phen)(H_2O)]_n \cdot nH_2O$ (**2**) (H_2bdc = isophthalic acid; phen = 1,10-phenanthroline) were synthesized and characterized by IR spectra, TGA, and X-ray crystallography. In complex **1**, the Nd(III) ions are bridged by bdc^{2-} and $Hbdc^{-}$ in three modes to form a two-dimensional layer. Adjacent layers are linked by hydrogen bonds composed of coordinated and lattice water molecules, together with carboxylate groups, forming a three-dimensional network. In complex **2**, the Nd(III) ions are connected by bdc^{2-} ligands in three modes into a two-dimensional layer. Weak C–H···O hydrogen bonding interactions between the chelated phen ligands and the carboxylate O atoms across adjacent layers result in a three-dimensional network.

Keywords: Neodymium complexes; Hydrothermal synthesis; X-ray crystallography; IR; TGA; Supramolecular structures

1. Introduction

Coordination polymers are of great interest due to their physical properties and potential application in functional materials [1–3]. Aromatic polycarboxylic acids such as 1,2,4,5-benzenetetracarboxylic acid [4], 1,3,5-benzenetricarboxylic acid [5], and 1,4-benzenedicarboxylic acid [6] are used extensively in the synthesis of coordination polymers. We selected isophthalic acid as a ligand based on the following considerations: (a) it has multiple coordination sites that may generate structures of higher dimensions; (b) it has two carboxyl groups which may be completely or only partly deprotonated and thus result in acidity-dependent coordination modes; (c) it can act not only as a hydrogen-bond acceptor but also as a hydrogen-bond donor because of the presence of the protonated carboxyl group; (d) it can form short bridges via one carboxylato end or long bridges via the benzene ring. We believe that coordination functionality of isophthalate could lead to interesting structures. A variety of coordination polymers containing the isophthalate ligand have been synthesized, but previous studies are focused mainly on those of transition or post-transition elements [7]. To the best of our knowledge, no lanthanide isophthalate coordination polymer has been reported. Herein we report two novel neodymium isophthalate complexes $[Nd_2(bdc)_2(Hbdc)_2(H_2O)]_n \cdot nH_2O$ (**1**) and $[Nd_2(bdc)_3(phen)(H_2O)]_n \cdot nH_2O$ (**2**), which were characterized by single crystal X-ray diffraction.

2. Experimental

$NdCl_3 \cdot 6H_2O$ was prepared by dissolving neodymium oxide in hydrochloric acid and then dried. All other materials were purchased and used as received without further purification. Infrared spectra were recorded with a Nicolet Avatar 360 FT-IR spectrometer. Elemental analyses were obtained on an Elementar Vario EL analyzer. TG-DTA curves were recorded on a ZRY-2P thermal analyzer.

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* Corresponding author. Tel.: +86-106-220-7838; fax: +86-106-220-0567.

E-mail address: lpjin@bnu.edu.cn (L.-P. Jin).

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*2.1. Synthesis of complexes **1** and **2***

A mixture of $NdCl_3 \cdot 6H_2O$ (0.109 g), H_2bdc (0.075 g), phen (0.059 g), and H_2O (10 ml) was placed in a Teflon-lined stainless steel autoclave (23 ml). The pH value was adjusted to about 4 with sodium hydroxide. After stirring, the mixture was sealed in the bomb and heated at 150 °C for 3 days (3 d). Then the bomb was cooled slowly at 20 °C per day to room temperature. After filtration, the product was washed with ethanol and then dried under ambient. Pale purple needle-like (46.6% yield) and purple block (5.3% yield) crystals were found. Elemental analysis indicates that the formula of the needle-like crystals is $[Nd_2(bdc)_2(Hbdc)_2(H_2O)] \cdot (H_2O)$ (**1**) [Calc.: C, 39.17; H, 2.06. Found: C, 38.79; H, 1.74%] and the formula of the block crystals is $[Nd_2(bdc)_3(phen)(H_2O)] \cdot (H_2O)$ (**2**) [Calc.: C, 43.36; H, 2.43; N, 2.81. Found: C, 43.30; H, 2.08; N, 2.75%]. IR (KBr pellet (cm^{-1})) for complex **1**: 3509(vs), 3197(vs), 1728(s), 1690(s), 1606(s), 1534(vs), 1484(s), 1435(s), 1369(vs), 1316(s), 1213(s), 1156(s), 1077(m), 798(m), 742(s), 728(s), 713(s), 681(s), 644(m). IR (KBr pellet (cm^{-1})) for complex **2**: 3419(vs), 3077(vs), 1615(vs), 1546(vs), 1477(s), 1447(s), 1387(vs), 1159(w), 1102(w), 1083(w), 844(m), 822(w), 749(s), 730(s), 712(s), 670(m), 655(w), 537(w), 421(m).

2.2. Single-crystal X-ray diffraction

Single-crystal X-ray data were collected on a Bruker SMART 1000 CCD diffractometer equipped with graphite monochromatized Mo Kα radiation ($\lambda = 0.71073$ Å). Semiempirical absorption corrections were applied using the SADABS program. All calculations were carried out with use of SHELXS-97 and SHELXL-97 programs [8]. The structures were solved by the direct method. All structures were refined on F^2 by full-matrix least-squares methods. The crystallographic data of complexes **1** and **2** are summarized in Table 1 and the selected bond distances in Table 2.

3. Results and discussion

3.1. Preparation

The complexes were prepared by the hydrothermal reaction of $NdCl_3 \cdot 6H_2O$ with H_2bdc in the presence of phen at 150 °C. When the pH value was adjusted to about 4, the mixture of complexes **1** and **2** was obtained. We sensed that a lower pH value may suppress the deprotonation of H_2bdc and make the N(phen) atoms protonated and thus weaken phen coordination. Adjusting the pH to about 5 yielded purple block crystals of ternary complex **2** in 96.4% yield.

Table 1
Crystallographic data for complexes **1** and **2**

	1	**2**
Formula	$C_{32}H_{22}Nd_2O_{18}$	$C_{36}H_{24}N_2Nd_2O_{14}$
Formula weight	982.98	997.05
Color, habit	pale purple, needle-like	purple, block
Temperature (K)	293(2)	293(2)
Crystal size (mm)	0.30 × 0.25 × 0.10	0.30 × 0.25 × 0.10
Crystal system	monoclinic	orthorhombic
Space group	$P2_1/c$	*Pbca*
Unit cell dimensions		
a (Å)	10.408(4)	14.495(7)
b (Å)	13.954(5)	20.586(10)
c (Å)	21.347(8)	23.443(12)
α (°)	90	90
β (°)	95.557(7)	90
γ (°)	90	90
V (Å^3)	3086(2)	6995(6)
Z	4	8
D_{calc} (Mg m^{-3})	2.116	1.893
$F(000)$	1912	3888
μ (mm^{-1})	3.418	3.010
Limiting indices	$-12 \leq h \leq 11$, $-16 \leq k \leq 12$, $-24 \leq l \leq 25$	$-17 \leq h \leq 16$, $-24 \leq k \leq 24$, $-27 \leq l \leq 19$
Reflections collected/unique	12 629/5446 ($R_{int} = 0.0728$)	27 841/6173 ($R_{int} = 0.0510$)
Completeness to $\theta = 25.03$ (%)	99.9	100.0
Max and min transmission	0.7262 and 0.4271	0.7528 and 0.4654
Data/restraints/parameters	5446/6/485	6173/3/495
Goodness-of-fit on F^2	0.903	1.099
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0385$, $wR_2 = 0.0576$	$R_1 = 0.0291$, $wR_2 = 0.0575$
R indices (all data)	$R_1 = 0.0765$, $wR_2 = 0.0646$	$R_1 = 0.0477$, $wR_2 = 0.0620$
Largest difference peak and hole (e Å^{-3})	0.817 and −0.826	0.961 and −0.569
Method of locating and refining H atoms	mixed	mixed

*3.2. Structural description of $[Nd_2(bdc)_2(Hbdc)_2(H_2O)]_n \cdot nH_2O$ (**1**)*

In the structure of complex **1**, there are two types of neodymium environment. The dinuclear species $[Nd_2(bdc)_2(Hbdc)_2(H_2O)]$ with Nd···Nd interatomic distance of 3.841(1) Å may be viewed as the building blocks shown in Fig. 1. Nd(1) atom is coordinated by nine oxygen atoms, six from bdc^{2-} and three from $Hbdc^{-}$ ligands. Nd(2) atom is surrounded by nine oxygen atoms, five from bdc^{2-}, three from $Hbdc^{-}$, and an aqua ligand (Fig. 1). Each Nd(III) ion exhibits the coordination geometry of a distorted tricapped trigonal prism. The bdc^{2-} ligands coordinate to Nd(III) ions in two modes: (i) two chelating-bridging tridentates (Scheme 1(a)); Nd(III) ions are connected into 1-D chain structure by this coordination mode along the b axis (Fig. 2(a)); (ii) chelating-bridging tridentate and

Table 2
Selected bond distances (Å) for complexes **1** and **2**

Bond distances in **1**					
Nd(1)–O(7)#1	2.301(5)	Nd(1)–O(13)	2.548(5)	Nd(2)–O(17)	2.500(6)
Nd(1)–O(2)#2	2.432(5)	Nd(1)–O(3)#3	2.647(4)	Nd(2)–O(5)	2.510(5)
Nd(1)–O(6)	2.439(5)	Nd(1)–O(5)	2.687(5)	Nd(2)–O(3)#2	2.524(5)
Nd(1)–O(10)	2.454(4)	Nd(2)–O(8)#4	2.397(5)	Nd(2)–O(10)	2.595(5)
Nd(1)–O(4)#3	2.475(5)	Nd(2)–O(1)	2.403(5)	Nd(2)–O(9)	2.605(5)
Nd(1)–O(14)	2.544(5)	Nd(2)–O(13)	2.411(4)	Nd(2)–O(2)	2.693(5)
Bond distances in **2**					
Nd(1)–O(12)#1	2.332(3)	Nd(1)–N(1)	2.742(4)	Nd(2)–O(11)#4	2.437(3)
Nd(1)–O(8)#2	2.351(3)	Nd(1)–O(1)	2.772(4)	Nd(2)–O(10)	2.441(3)
Nd(1)–O(4)#3	2.365(3)	Nd(2)–O(3)	2.353(3)	Nd(2)–O(6)	2.457(3)
Nd(1)–O(9)	2.386(3)	Nd(2)–O(7)#2	2.386(3)	Nd(2)–O(13)	2.567(4)
Nd(1)–O(5)	2.548(3)	Nd(2)–O(2)#2	2.411(3)	Nd(2)–O(1)#2	2.866(4)
Nd(1)–N(2)	2.740(4)				

Symmetry transformations used to generate equivalent atoms in complex **1**: #1 $-x+1,-y+1,-z+1$; #2 $-x+1,y+1/2,-z+3/2$; #3 $x,y+1,z$; #4 $x,-y+1/2,z+1/2$. Symmetry transformations used to generate equivalent atoms in complex **2**: #1 $-x+1/2,y+1/2,z$; #2 $x-1/2,-y+3/2,-z+1$; #3 $x+1/2,-y+3/2,-z+1$; #4 $-x,-y+1,-z+1$.

syn–syn bridging bidentate (Scheme 1(b)); 1-D chains are connected into a 2-D layer structure by this coordination mode along the *c* axis (see Fig. 2(a)). Ligand Hbdc$^-$ coordinates to two neodymium ions in chelating-bridging mode at the deprotonated carboxylate group (Scheme 1(c)). The protonated end of Hbdc$^-$ is free and the two Hbdc$^-$ groups of [$Nd_2(bdc)_2(Hbdc)_2H_2O$] stretch out in different directions (Fig. 2(b)). The carboxylic group O(15)=C(31)–O(16)–H(16B) of a Hbdc$^-$ runs roughly along the *c* axis (see Fig. 2(b)) and stays in the layer. Only one kind of hydrogen bond, O(16)–H(16B)···O(9) [x, $-y+1/2$, $z+1/2$] exists. The carboxylic group O(11)=C(23)–O(12)–H(12B) of another Hbdc$^-$ ligand runs roughly along the *b* axis and stretches out (see Fig. 2(b)). O(12)–H(12B) bonds to O(4) [$x+1$, $y+1$, z] in the neighboring layer. O(17)–H(1) from the coordinated water molecule bonds to O(11) [$x-1$, y, z]. O(18)–H(3) from the lattice water molecule bonds to O(11) [$-x+2$, $y-1/2$, $-z+3/2$]. So the carboxylic group O(11)=C(23)–O(12)–H(12B) can form three types of hydrogen bonds. The lattice water molecule lies between two layers and is

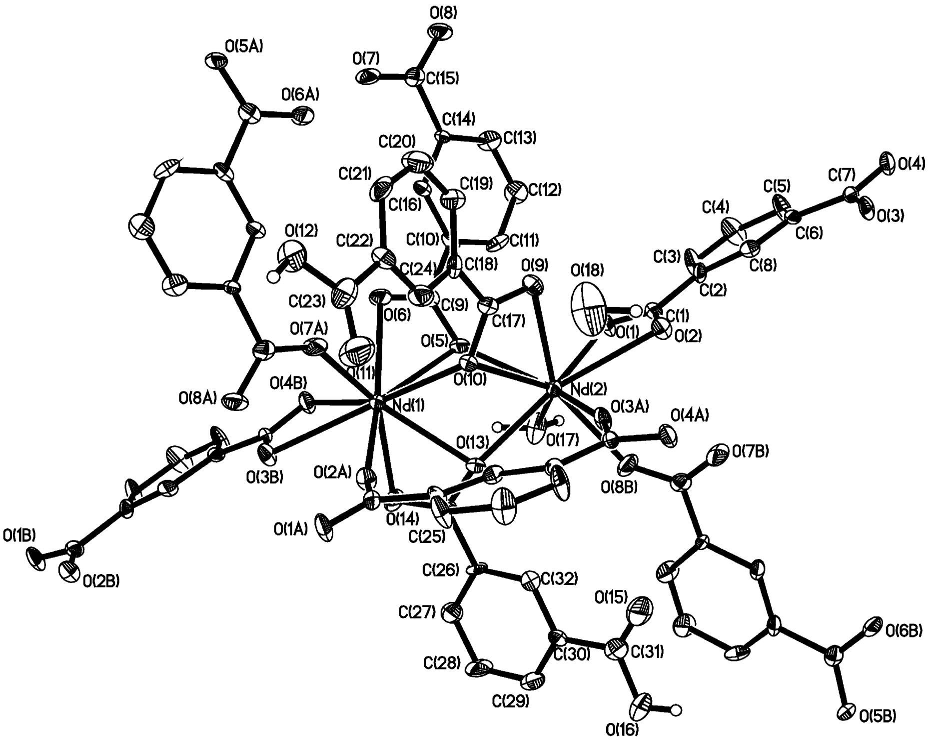
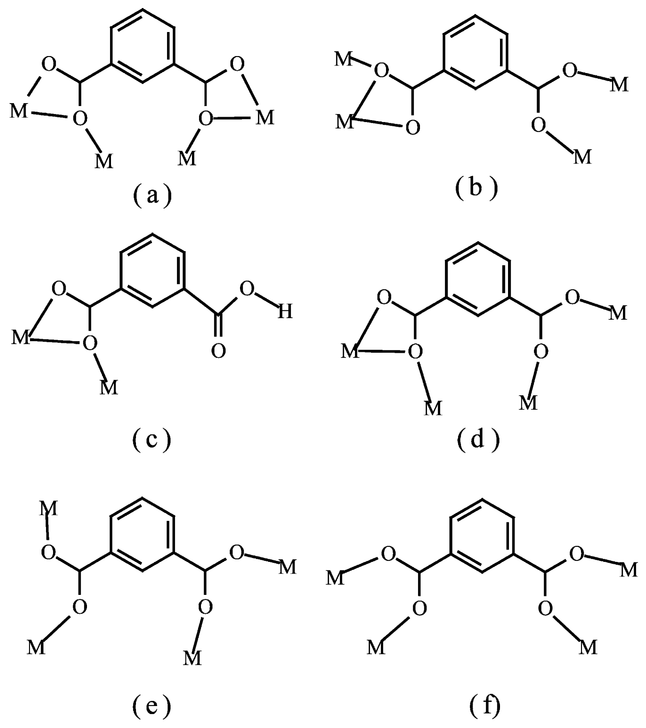
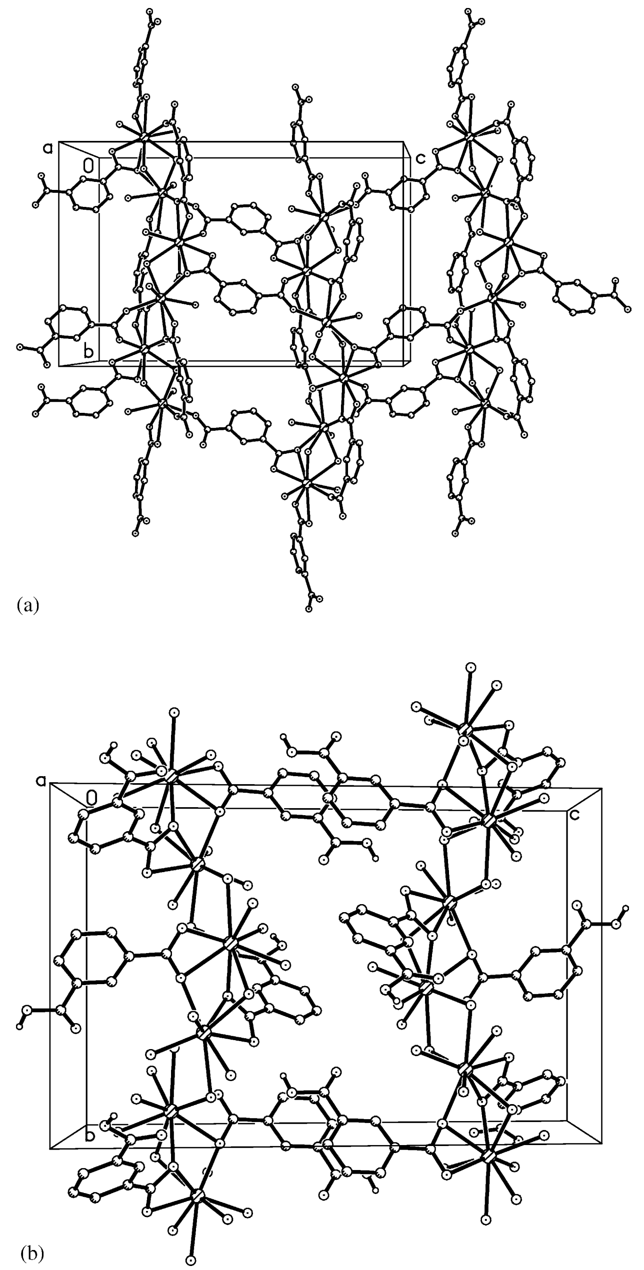


Fig. 1. Asymmetric unit of complex **1** showing 50% thermal ellipsoids. The hydrogen atoms attached to C atoms are omitted for clarity.



Scheme 1. Coordination modes of isophthalate groups in complexes **1** and **2**.

stabilized by three kinds of hydrogen bonds in the crystal. Besides O(18)–H(3)···O(11) [$-x+2$, $y-1/2$, $-z+3/2$] mentioned above, there are O(18)–H(4)···O(14) [$-x+1$, $y-1/2$, $-z+3/2$] and O(17)–H(2)···O(18) [$x-1$, y, z]. Therefore, there are six types of hydrogen bonds in **1** in total (Table 3). And thus complex **1** has a 3-D supramolecular structure with adjacent layers interlinked by five kinds of hydrogen bonds.



(a)

(b)

Fig. 2. View of an intralamellar structure down the a axis for complex **1**, showing the environments of Nd(III) ions: (a) the coordination modes for bdc^{2-}, (b) the coordination modes for Hbdc^{-}.

*3.3. Structural description of [Nd$_2$(bdc)$_3$(phen)(H$_2$O)]·(H$_2$O) (**2**)*

Complex **2** possesses an asymmetric unit containing two neodymium(III) ions (shown in Fig. 3). The nearest Nd···Nd distance is 4.966 Å, indicating the lack of direct metal–metal interaction. Nd(1) ion is eight-coordinated and possesses an N$_2$O$_6$ environment by virtue of six oxygen atoms from six bdc^{2-} ligands and two N atoms from a chelating phen ligand. The Nd–O bond distances range from 2.332 to 2.773 Å. The average distance is 2.459(4) Å, similar to other related Nd–O distances [3,9]. Both Nd(1)–N(phen) bond lengths are 2.741 Å. Nd(2) is coordinated by eight oxygen atoms from six bdc^{2-} and one aqua ligand. The Nd(2)–O(1) [$x-1/2$, $-y+3/2$, $-z+1$] distance (2.866 Å) is quite long while the other seven Nd(2)–O distances are typical. The reason is probably due to the stronger strain in the four-membered ring of chelating/bridging coordination. Each of two Nd(III) ions possesses a distorted triangular dodecahedron. Three types of coordination modes of bdc^{2-} ligands exist in the structure: (i) chelating/bridging tridentate and *syn–anti* bridging bidentate (Scheme 1(d)); (ii) two *syn–anti* bridging bidentates (Scheme 1(e)); Nd(III) ions are connected into a helical chain by coordination modes Scheme 1(d and e) along the *a* axis (Fig. 4(a)); (iii) two *syn–syn* bridging bidentates (Scheme 1(f)); the helical chains are connected into a layer in this coordination mode along the *b* axis (Fig. 4(b)). In **2**, there are three types of hydrogen bonds (Table 4). Lattice water molecules located between two layers are stabilized by one kind of hydrogen bond O(13)–

Table 3
Hydrogen bonds parameters (Å, °) for complex **1**

D–H	d(D–H)	d(H···A)	∠DHA	d(D···A)	Position of A
O(12)–H(12B)	0.820	2.055	166.19	2.858	O(4) $[x+1,y+1,z]$
O(16)–H(16B)	0.820	1.923	165.81	2.726	O(9) $[x,-y+1/2,z+1/2]$
O(17)–H(1)	0.926	2.126	159.33	3.010	O(11) $[x-1,y,z]$
O(17)–H(2)	0.928	1.848	164.31	2.753	O(18) $[x-1,y,z]$
O(18)–H(3)	0.931	2.000	175.06	2.928	O(11) $[-x+2,y-1/2,-z+3/2]$
O(18)–H(4)	0.929	2.344	123.74	2.962	O(14) $[-\mathrm{x}+1,y-1/2,-\mathrm{z}+3/2]$

H(2)···O(14)(lattice water), though the water oxygen has large ellipsoid due to partial occupancy. The aqua ligand forms two types of hydrogen bonds. Besides O(13)–H(2)···O(14) mentioned above, there is O(13)–H(1)···O(6). C(27)–H(27A) of the chelated phen ligand bonds to O(7) from the adjacent layer at C(27)···O(7) 3.420 Å, falling in the range from 3.00 to 4.00 Å [10]. Such intermolecular hydrogen bonding interactions between layers result in a 3-D network.

3.4. IR spectra

The IR spectrum of complex **1** shows characteristic bands of the carboxylate groups in the usual region [7b,7c,7e,11] at 1605(s) and 1534(vs) cm^{-1} for asymmetric stretching and at 1434(sh), 1368(vs) for symmetric stretching. For complex **2**, the characteristic bands of the dicarboxylate unit occur at 1614(s) and 1546(s) for asymmetric stretching and at 1446(s), 1386(vs) for symmetric stretching. The splitting of $v_{asym}(CO_2)$ and $v_{sym}(CO_2)$ in each complex indicates that carboxylate groups of bdc^{2-} and Hbdc^{-} function in two different coordination fashions [12] (chelating/bridging tridentate and bridging bidentate modes), consistent with the crystal structures of **1** and **2**. For complex **1**, two well-defined bands are observed at 1728 and 1689 cm^{-1} which are characteristic of the C=O double bond [4a,13] and there are two strong peaks at

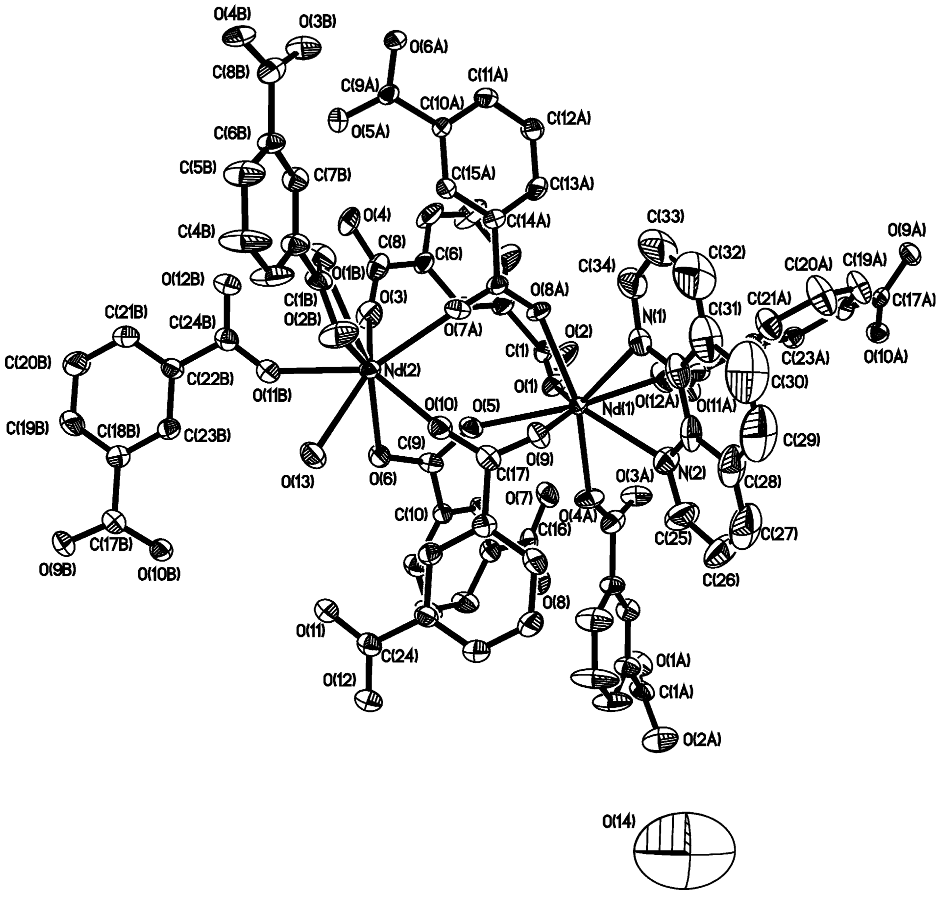


Fig. 3. Asymmetric unit of $[\mathrm{Nd_2(bdc)_3(phen)(H_2O)}]_n \cdot n(\mathrm{H_2O})$ (**2**) showing 50% thermal ellipsoids. The hydrogen atoms are omitted for clarity.

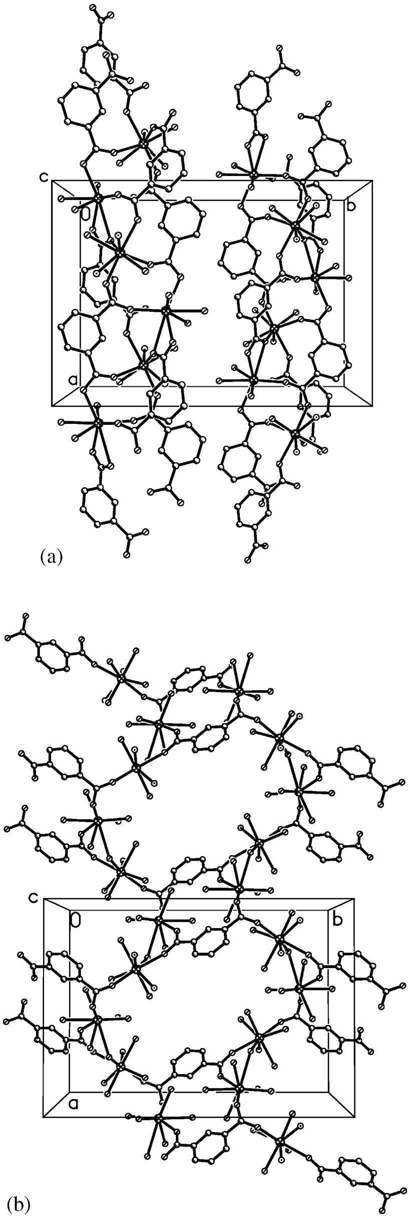


Fig. 4. Projection down the *c* axis for complex **2**, showing the environments of Nd(III) ions: (a) the coordination modes Scheme 1(d and e) for bdc^{2-}, (b) the coordination mode Scheme 1(f) for bdc^{2-}.

1213 and 1156 cm^{-1} which are characteristic bands of the C–O single bond [13], indicating the occurrence of two types of $Hbdc^{-}$ in different environments, which is in accordance with the results of single crystal X-ray analysis.

3.5. Thermogravimetric analyses (TGA)

DTA and TG results show that both complexes are stable in air and that the loss of lattice and coordinated water molecules (Found: 3.2%; Calc.: 3.7%) took place at 218–253 °C for complex **1**, and the loss of lattice water molecules (Found: 2.0%; Calc.: 1.8%) took place at 124–155 °C for complex **2**. The results indicate that the lattice water molecules in complex **1** are stabilized by stronger hydrogen bonds, which is supported by single crystal X-ray analysis. The temperatures of further decomposition are 417 and 548 °C for complexes **1** and **2**, respectively. The final residue could be considered as neodymium oxide Nd_2O_3 at 820 °C for **1** (Found: 36.3; Calc.: 34.2%) and 813 °C for **2** (Found: 37.9%; Calc.: 33.7%).

4. Conclusion

The first examples of lanthanide isophthalato complexes, $[Nd_2(bdc)_2(Hbdc)_2(H_2O)]_n \cdot nH_2O$ (**1**) and $[Nd_2(bdc)_3(phen)(H_2O)]_n \cdot n(H_2O)$ (**2**) have been obtained by hydrothermal synthesis and characterized by single crystal X-ray analysis. The results show that the carboxylate groups of H_2bdc ligands have two coordination modes, chelating-bridging tridentate and bridging bidentate. The Nd(III) ions in **1** are bridged by carboxylate groups of bdc^{2-} and $Hbdc^{-}$ forming a layer structure, while layers in **2** are formed by the coordination of bdc^{2-} to Nd(III) ions and are further connected via a variety of hydrogen bonds to give a 3-D network. Coordination polymers **1** and **2** were formed at pH 4, and **2** was also obtained at pH 5 indicating that pH control plays an important role in formation of lanthanide coordination polymers.

5. Supplementary material

Supplementary crystallographic data (CCDC Nos. 192083 and 192084) can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1 EZ, UK (fax: +44-1223-336033; e-mail:

Table 4
Hydrogen bonds parameters (Å, °) for complex **2**

D–H	*d*(D–H)	*d*(H···A)	∠DHA	*d*(D···A)	Position of A
O(13)–H(1)	0.929	1.867	162.83	2.768	O(6) $[-x,-y+1,-z+1]$
O(13)–H(2)	0.925	2.155	115.20	2.683	O(14) $[x-1,y,z]$
C(27)–H(27A)	0.930	2.558	154.2	3.420	O(7) $[x,-y+3/2,z+1/2]$

deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/conts/retrieving.html).

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