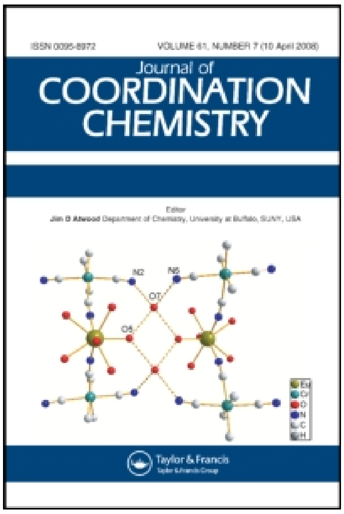

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Preparation, crystal structure and thermal stability of porous lanthanide complexes with 5-methyl isophthalic acid

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Preparation, crystal structure and thermal stability of porous lanthanide complexes with 5-methyl isophthalic acid

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Three lanthanide complexes **1**–**3** with 5-methyl isophthalic acid (5-CH_3-H_2bdc) were prepared under hydrothermal conditions, two have formula Eu_2(5-CH_3-bdc)$_3$(EtOH) (**2**) and [Er(5-CH_3-bdc)$_{1.5}$]·(H_2O) (**3**) and were characterized by X-ray single crystal diffraction. In **2**, there are two europium(III) ions in seven-coordinate pentagonal bipyramid and eight-coordinate bicapped trigonal prism geometries. Complex **2** is a 3-D porous structure with 1-D channels (potential solvent area = 453 Å^3, 15.7%). One crystallographic independent erbium(III) ion exists and lies in a pentagonal bipyramidal geometry in **3**, in which one-dimensional channels are rectangular (potential solvent area = 470 Å^3, 16.4%). TG-DTG experiments show that these complexes have stability to 750–880°C decomposing to corresponding Ln_2O_3.

Keywords: Porous structure; Hydrothermal; 5-Methyl isophthalic acid; Lanthanide

1. Introduction

Isophthalic acid and its derivatives are widely employed in construction of metal-organic frameworks (MOFs) as bridging ligands [1] because they are sterically rigid, chemically robust and, with multiple coordination modes of carboxylate groups thermally stable. Many transition metal coordination polymers with isophthalic acid and its derivatives [2] and many lanthanide complexes with these ligands were prepared [3].

Lanthanides have received attention for their special coordination properties to form isostructural complexes and interesting physical and chemical characteristics. The high coordination number and flexible coordination geometry of lanthanide metal ions, however, make it difficult to control the preparation of lanthanide complexes in formation of unusual molecular architectures.

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In comparison to lanthanide compounds from isophthalic acid, a few compounds from 5-amino-isophthalate [4], 5-hydroxy-isophthalate [5] and 5-sulfonic-isophthalate [6] lanthanide complexes have been isolated. In our previous work, we prepared high-dimensional lanthanide complexes from 5-nitroisophthalate [7]. As revealed in these complexes, the fifth site substituents of isophthalic acid affect the construction of lanthanide complexes. Extending this work, in this communication, we chose 5-methylisophthalic acid as ligand and lanthanide ions Sm^{3+}, Eu^{3+} and Er^{3+} to synthesize lanthanide complexes and study their thermal stability. Of particular interest is that, different with the documental complexes [4–7], the as-prepared lanthanide complexes possess channel structures containing guest solvent molecules.

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 analyzer. IR spectra were recorded with a BEQ VZNDX-550 FT-IR spectrometer using KBr pellets. The TG-DTG curves were obtained with a Perkin Elmer thermogravimetric analyzer in air with a heating rate of 10°C min^{-1}.

2.2. *Syntheses*

$Sm_2(5\text{-}CH_3\text{-bdc})_3(H_2O)$ (1): 5-CH_3-H_2bdc (0.0360 g, 0.2 mmol) in EtOH (4 mL) was mixed with an aqueous solution (8 mL) 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 mL Teflon-lined autoclave and heated at 160°C for 72 h. The autoclave was cooled at a rate of 5°C h^{-1} to 100°C, preserved at 100°C for 10 h, then cooled to room temperature naturally when the heater was shut off. Some colorless sheet-like crystals of **1** were collected by filtration, washed with water, and dried in air (Ca 62% yield, based on Sm). Anal. (%) Calcd for $C_{27}H_{20}O_{13}Sm_2$ (**1**): C, 38.01; H, 2.36. Found: C, 38.40; H, 1.97. IR (KBr, 400–4000 cm^{-1}): 3389.7(w), 1617(m), 1536(s), 1430(s), 1378(s), 927(w), 730(s).

$Eu_2(5\text{-}CH_3\text{-bdc})_3(EtOH)$ (2): Complex **2** was synthesized similar to **1**, except that $Eu(NO_3)_3 \cdot 6H_2O$ was used instead of $Sm(NO_3)_3 \cdot 6H_2O$. Some brown stick-like crystals of **2** were collected by filtration, washed with water, and dried in air (Ca 55% yield, based on Eu). Anal. (%) Calcd for $C_{29}H_{24}O_{13}Eu_2$ (**1**): C, 39.38; H, 2.74. Found: C, 39.59; H, 2.55. IR (KBr, 400–4000 cm^{-1}): 3391.3(w), 1621(m), 1532(s), 1431(s), 1381(s), 930(w), 726(s).

$Er(5\text{-}CH_3\text{-bdc})_{1.5}(H_2O)$ (3): Complex **3** could be obtained by following the same procedures as for **1** except using $Er(NO_3)_3 \cdot 6H_2O$ instead of $Sm(NO_3)_3 \cdot 6H_2O$. Some maple stick-like crystals of **3** were collected by filtration, washed with water, and dried in air (Ca 53% yield, based on Er). Anal. (%) Calcd for $C_{13.5}H_{11}O_7Er$ (**3**): C, 35.83; H, 2.45. Found: C, 35.61; H, 1.99. IR (KBr, 400–4000 cm^{-1}): 3389(w), 1624(m), 1530(s), 1428(s), 1380(s), 932(w), 728(s).

Table 1. Crystallographic data and experimental details for structural analyses of **2** and **3**.

Complex	**2**	**3**
Empirical formula	$C_{29}H_{24}O_{13}Eu_2$	$C_{13.50}H_{11}O_7Er$
Formula weight	884.40	452.48
T(K)	273(2)	273(2)
Crystal system	Monoclinic	
Space group	$P2(1)/c$	$Pnma$
Unit cell and dimensions (Å, °)		
a	19.399(3)	18.6879(1)
b	8.2948(12)	19.3478(1)
c	18.714(3)	7.9438(6)
β	106.280(2)	90
V (Å^3)	2890.6(7)	2872.2(4)
Z	4	8
F(000)	1712	1728
D_{Calcd} (Mg m^3)	2.032	2.093
Absorption coefficient (mm^{-1})	4.369	5.874
Crystal size (mm^3)	$0.50 \times 0.16 \times 0.14$	$0.38 \times 0.15 \times 0.08$
Range hkl collected	$-23 \le h \le 22$, $-9 \le k \le 9$, $-12 \le l \le 22$	$-24 \le h \le 24$, $-18 \le k \le 25$, $-10 \le l \le 10$
Reflections collected	12633	16143
Independent reflections	5071 [$R_{\text{int}} = 0.0343$]	3404 [$R_{\text{int}} = 0.0430$]
Completeness to θ	99.7%	98.6%
Data/restraints/parameters	5071/18/401	3404/2/205
Goodness-of-fit on F^2	1.041	1.163
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0261$, $wR_2 = 0.0541$	$R_1 = 0.0904$, $wR_2 = 0.2041$
R indices (all data)	$R_1 = 0.0353$, $wR_2 = 0.0584$	$R_1 = 0.01031$, $wR_2 = 0.2098$
Largest difference peak and hole (e Å^{-3})	0.893 and -0.739	11.975 and -7.405

2.3. *X-ray crystallography*

Suitable single crystals for **2** and **3** were selected and their structure determinations were performed 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. Their structures were solved by the direct method. The lanthanide and some oxygens were first located and the carbon, nitrogen and other oxygen atoms were found in the final difference Fourier map. All the H atoms attached to the carbon were placed geometrically. The structures were refined on F^2 by the full-matrix least-squares method using the SHELXL-97 (Sheldrick, 1997) program package [8]. All non-hydrogen atoms were refined anisotropically. The crystallographic data and experimental details for structural analyses of **2** and **3** are shown in table 1.

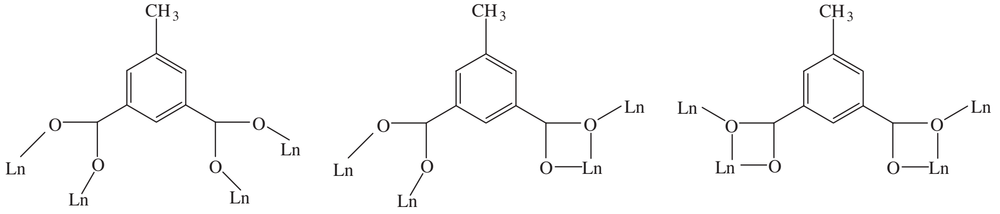
3. Results and discussion

Single crystal diffraction data of **1** could not be obtained because of poor crystal quality. Selected bond lengths and angles of **2** and **3** are listed in table 2. As shown in scheme 1, the 5-CH_3-bdc^{2-} adopts three coordination modes, *bis*(bidentate), *bis*(bidentate-μ_2-bridge) and *bis*(μ_2-bridge). There are two different Eu(III) ions of in **2**.

Table 2. Selected bond lengths (Å) and angles (°) for **2** and **3**.

$C_{29}H_{24}O_{13}Eu_2$ (**2**)				$C_{13.50}H_{11}O_7Er$ (**3**)	
Eu(1)–O(6)#1	2.304(3)	Eu(2)–O(9)	2.283(3)	Er(1)–O(6)#1	2.200(12)
Eu(1)–O(5)	2.306(3)	Eu(2)–O(12)#5	2.303(3)	Er(1)–O(4)	2.250(12)
Eu(1) –O(2)#1	2.322(3)	Eu(2)–O(11)#6	2.327(3)	Er(1)–O(3)#2	2.251(12)
Eu(1)–O(7)#2	2.401(3)	Eu(2)–O(10)#7	2.329(3)	Er(1)–O(5)#3	2.260(11)
Eu(1)–O(1)	2.402(3)	Eu(2)–O(3)	2.414(3)	Er(1)–O(2)#4	2.303(14)
Eu(1)–O(8)#3	2.464(3)	Eu(2)–O(4)#8	2.428(3)	Er(1)–O(1)	2.348(14)
Eu(1)–O(13)	2.556(4)	Eu(2)–O(4)	2.589(3)	Er(1)–O(2)	2.568(14)
Eu(1)–O(8)#2	2.633(3)	O(4)#8–Eu(2)–O(4)			
O(6)#1–Eu(1)–O(5)	155.60(1)	O(9)–Eu(2)–O(12)#5	84.20(1)	O(6)#1–Er(1)–O(4)	100.2(5)
O(6)#1–Eu(1)–O(2)#1	102.89(1)	O(9)–Eu(2)–O(11)#6	91.90(1)	O(6)#1–Er(1)–O(3)#2	83.8(5)
O(5)–Eu(1)–O(2)#1	82.43(1)	O(12)#5–Eu(2)–O(11)#6	153.89(1)	O(4)–Er(1)–O(3)#2	154.9(5)
O(6)#1–Eu(1)–O(7)#2	131.34(1)	O(9)–Eu(2)–O(10)#7	174.74(2)	O(6)#1–Er(1)–O(5)#3	172.0(5)
O(5)–Eu(1)–O(7)#2	72.83(1)	O(12)#5–Eu(2)–O(10)#7	97.30(2)	O(4)–Er(1)–O(5)#3	85.0(5)
O(2)#1–Eu(1)–O(7)#2	80.75(1)	O(11)#6–Eu(2)–O(10)#7	88.93(1)	O(3)#2–Er(1)–O(5)#3	94.1(5)
O(6)#1–Eu(1)–O(1)	79.62(1)	O(9)–Eu(2)–O(3)	84.07(2)	O(6)#1–Er(1)–O(2)#4	85.9(5)
O(5)–Eu(1)–O(1)	85.27(1)	O(12)#5–Eu(2)–O(3)	125.29(1)	O(4)–Er(1)–O(2)#4	77.0(5)
O(2)#1–Eu(1)–O(1)	153.33(1)	O(11)#6–Eu(2)–O(3)	79.68(1)	O(3)#2–Er(1)–O(2)#4	78.6(5)
O(7)#2–Eu(1)–O(1)	117.88(1)	O(10)#7–Eu(2)–O(3)	90.98(2)	O(5)#3–Er(1)–O(2)#4	101.2(5)
O(6)#1–Eu(1)–O(8)#3	77.11(1)	O(9)–Eu(2)–O(4)#8	82.53(1)	O(6)#1–Er(1)–O(1)	80.5(5)
O(5)–Eu(1)–O(8)#3	81.33(1)	O(12)#5–Eu(2)–O(4)#8	78.25(1)	O(4)–Er(1)–O(1)	78.5(5)
O(2)#1–Eu(1)–O(8)#3	75.60(1)	O(11)#6–Eu(2)–O(4)#8	75.64(1)	O(3)#2–Er(1)–O(1)	126.5(5)
O(7)#2–Eu(1)–O(8)#3	146.92(1)	O(10)#7–Eu(2)–O(4)#8	102.70(1)	O(5)#3–Er(1)–O(1)	94.7(5)
O(1)–Eu(1)–O(8)#3	79.21(1)	O(3)–Eu(2)–O(4)#8	151.40(1)	O(2)#4–Er(1)–O(1)	149.3(5)
O(6)#1–Eu(1)–O(13)	75.55(1)	O(9)–Eu(2)–O(4)	88.44(1)	O(6)#1–Er(1)–O(2)	83.0(5)
O(5)–Eu(1)–O(13)	114.61(2)	O(12)#5–Eu(2)–O(4)	74.65(1)	O(4)–Er(1)–O(2)	129.5(5)
O(2)#1–Eu(1)–O(13)	142.65(2)	O(11)#6–Eu(2)–O(4)	131.18(1)	O(3)#2–Er(1)–O(2)	75.5(4)
O(7)#2–Eu(1)–O(13)	73.93(3)	O(10)#7–Eu(2)–O(4)	87.11(1)	O(5)#3–Er(1)–O(2)	89.0(4)
O(1)–Eu(1)–O(13)	63.98(1)	O(3)–Eu(2)–O(4)	51.79(1)	O(2)#4–Er(1)–O(2)	152.80(3)
O(8)#3–Eu(1)–O(13)	137.21(1)	O(7)#2–Eu(1)–O(8)#2	51.80(1)	O(1)–Er(1)–O(2)	52.1(5)
O(6)#1–Eu(1)–O(8)#2	82.20(1)	O(1)–Eu(1)–O(8)#2	132.58(1		
O(5)–Eu(1)–O(8)#2	121.89(1)	O(8)#3–Eu(1)–O(8)#2	137.65(7)		
O(2)#1–Eu(1)–O(8)#2	73.60(1)	O(13)–Eu(1)–O(8)#2	69.19(1)		

Symmetry codes **1**: #1: $-x$, $y+1/2$, $-z+1/2$; #2: $-x$, $-y+1$, $-z+1$; #3: x, $-y+1/2$, $z-1/2$; #4: $-x$, $y-1/2$, $-z+1/2$; #5: $-x+1$, $-y+1$, $-z$; #6: x, $-y+3/2$, $z+1/2$; #7: $-x+1$, $y-1/2$, $-z+1/2$ #8: $-x+1$, $y+1/2$, $-z+1/2$.
Symmetry codes **2**: #1: $x+1/2$, y, $-z+3/2$; #2: $-x+3/2$, $-y+1$, $z-1/2$; #3: $-x+1$, $-y+1$, $-z+1$; #4: $-x+3/2$, $-y+1$, $z+1/2$.



(a) (b) (c)

Scheme 1. The coordination modes of 5-CH_3-bdc^{2-} ligand in **2** and **3**.

Eu1 is coordinated by five oxygens from bridging carboxylates, two oxygens from a chelating carboxylate and an oxygen from EtOH into an eight-coordinate bicapped trigonal prism, while Eu2 lies in a seven-coordinate pentagonal bipyramid by five oxygens from bridging carboxylates and two oxygens from a chelating carboxylate of 5-CH_3-bdc^{2-} (figure 1). Ligands in mode (a) connect all Eu2 ions (Eu2 ··· Eu2 = 4.311 Å) into a Eu2-2D sandwich network, in which the sandwich

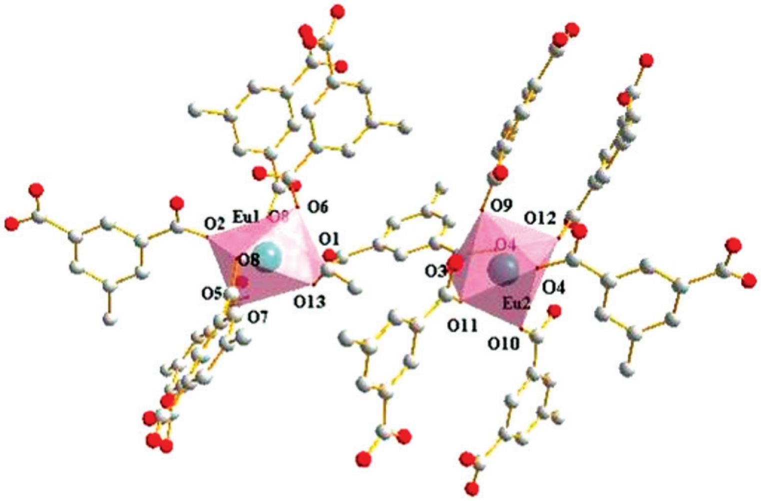


Figure 1. Coordination environments of two Eu^{2+} ions in **2**.

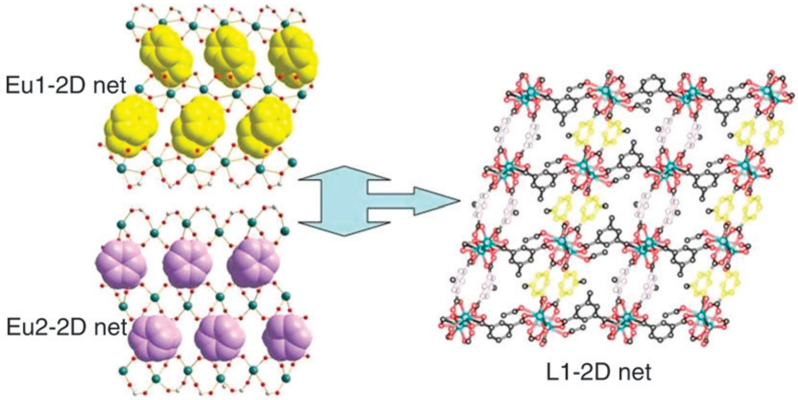


Figure 2. Eu1-2D network form and Eu2-2D nets in **2**.

consists of pairs of parallel 5-CH_3-bdc^{2-} ligands with π–π packing interactions (the distance face-to-face is 3.470 Å). The dihedral angle between adjacent ligands spaced by Eu1 metallic chains is 33°. Similarly, ligands in mode (b) combine all Eu1 ions (Eu1 $\cdots$ Eu1 = 4.426 Å) into a Eu1-2D sandwich network, in which pairs of parallel ligands also with π–π packing interactions, 3.277 Å face-to-face distance, shorter than the distance of 3.451 Å found in [$K_2Eu_2(ipa)_4(H_2O)_4 \cdot 8H_2O$] [3c]. The dihedral angle between adjacent ligands spaced by Eu2 metallic chains is 33°. Finally, ligands in mode (b) connect Eu1-2D and Eu2-2D sandwich networks into a 3-D structure with a 1-D channel parallel to *b* (figure 2). Dimensions of the channel are 4.2 × 8.3 Å, a little smaller than of 5.5 × 7 Å [3c]. The end methyl groups of ethanol molecules coordinating to Eu1 ions hang in 1-D rhombus channels. As all the ethanol molecules are excluded, the potential solvent area is 453 $Å^3$ (15.7%). There is a hydrogen bond from O13 of ethanol and O1 of carboxylate (O13–H13 $\cdots$ O1, 2.629 Å, 111.64°).

The structure of **3** differs from that of **2**; only one independent erbium(III) exists in the asymmetric unit. Er1 is in a distorted seven-coordinate pentagonal bipyramidal geometry (O5 and O6 are vertexs, O1, O2, O3, O4 and one symmetrical atom of O2 construct the pentagonal plane). Of the seven oxygens, O1 and O2 are from a chelating carboxylate; the other five oxygens are from bridging carboxylates (figure 3). The 5-CH_3-bdc^{2-} ligand adopts modes (a) and (c) in **3** connecting Er(III) ions into two kinds of 2-D networks. The ligands in mode (a) construct a sandwich network (scheme 2) as in **2**, and the face-to-face distance of π–π interactions between pairs

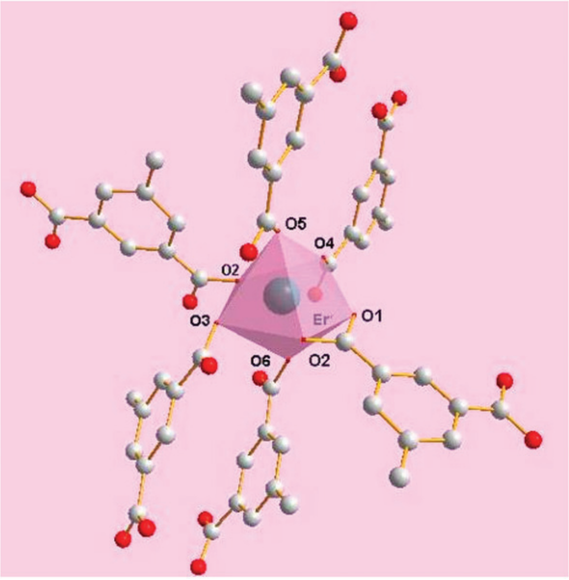
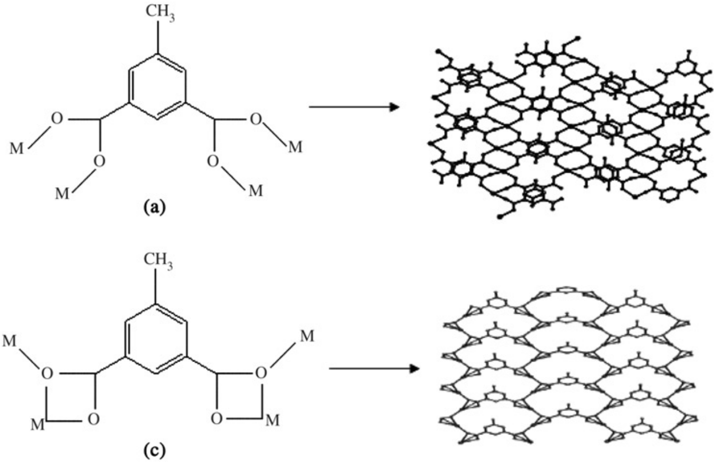


Figure 3. Coordination environment of Er^{3+} in **3**.



Scheme 2. 2-D networks from ligands in (a) and (c) modes in **3**.

of parallel ligands is 3.496 Å, similar to the distance of 3.451 Å found in $[K_2Eu_2(ipa)_4(H_2O)_4 \cdot 8H_2O]$ [3c]. The similar dihedral angle is 42.1°. Ligands in mode (*c*) link these 2-D sandwich networks into a 3-D structure with 1-D rectangle channels, shown in figure 4. The dimensions of the channel are approximately 5.1×8.0 Å, bigger than that of 5.5×7 Å [3c]. Lattice waters occupy these 1-D channels. As they are moved, the potential solvent area of **3** is 470 $Å^3$ (16.4%). The sandwich networks in **2** and **3** also exist in 5-nitroisophthalate lanthanide complexes [7a].

In order to investigate the thermal stabilities for **1**–**3**, their thermal decompositions were carried out in a temperature range from 30 to 1000°C. From the TG-DTG experiments, we found decomposition of these complexes divided into two stages: at the first stage the complexes lost solvent molecules, then converted into Ln_2O_3 at 750 ~ 880°C. The TG-DTG curves of **3** are shown in figure 5. At 200–500°C,

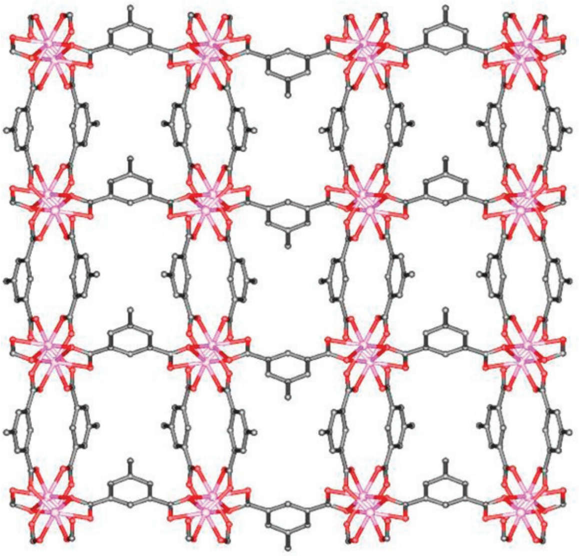


Figure 4. Cutaway view of 1D channel in **3**.

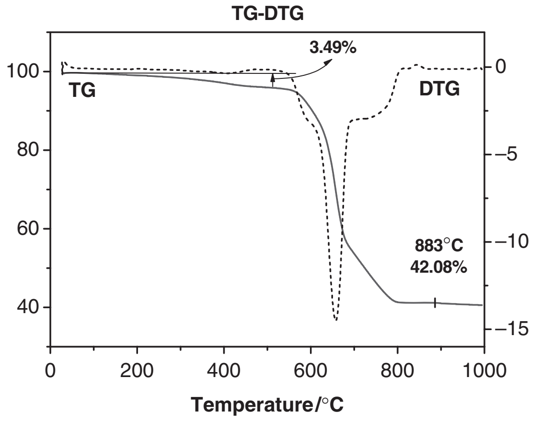


Figure 5. TG-DTG curves for **3**.

the skeleton of the complex loses water 3.49% (calculated value: 3.98%). In the second stage from 550 to 883°C, decomposition gives Er_2O_3 42.08% (calculated value: 42.27%). The decomposition temperature of unhydrated residue of **3** is higher than that of complexes reported by Li [9]. Complex **3** exhibits exceptional thermal stability.

Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 654278 and 654279. 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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References

[1] (a) P.S. Mukherjee, N. Das, Y.K. Kryschendo, A.M. Arif, P.J. Stang. *J. Am. Chem. Soc.*, **126**, 2464 (2004); (b) J.J. Nie, L.J. Liu, Y. Luo, D.J. Xu. *J. Coord. Chem.*, **53**, 365 (2001); (c) Y.F. Zhou. *Private Communication*. The Cambridge Crystallographic Data Centre, Cambridge, England (2003); (d) L.Y. Zhang, G.F. Liu, S.L. Zheng, B.H. Ye, X.M. Chen. *Eur. J. Inorg. Chem.*, 2965 (2003); (e) S.A. Boume, A. Mondal, M.J. Zaworotko. *Cryst. Eng.*, **4**, 25 (2001).

[2] (a) L. Gao, B.J. Zhao, G.H. Li, Z. Shi, S.H. Feng. *Inorg. Chem. Commun.*, **6**, 1249 (2003); (b) J. Tao, X.M. Chen, R.B. Huang, L.S. Zheng. *J. Solid State Chem.*, **170**, 130 (2003); (c) B. Moulton, H. Abourahma, M.W. Bradner, J.J. Lu, G.J. Mcmanus, M.J. Zaworotko. *Chem. Commun.*, 1342 (2003); (d) C.B. Ma, C.N. Chen, Q.T. Liu, D.Z. Liao, L.C. Li, L.C. Sun. *New J. Chem.*, **27**, 890 (2003); (e) S.A. Boume, J.J. Lu, A. Mondal, B. Moulton, M.J. Zaworotko. *Angew. Chem. Int. Ed.*, **40**, 2111 (2001).

[3] (a) Y.H. Wan, L.P. Zhang, L.P. Jin, S. Gao, S.Z. Lu. *Inorg. Chem.*, **42**, 4985 (2003); (b) Y.F. Zhou, F.L. Jiang, Y. Xu, R. Cao, M.C. Hong. *J. Mol. Struct.*, **691**, 191 (2004); (c) A. de Bettencourt-Dias. *Inorg. Chem.*, **44**, 2734 (2005); (d) Y.L. Qua, Y.X. Kea, S.M. Lua, R. Fan, G.Q. Pan, J.M. Li. *J. Mol. Struct.*, **734**, 7 (2005).

[4] (a) H.T. Xu, N.W. Zheng, X.L. Jin, R.Y. Yang, Z.Q. Li. *Chem. Lett.*, 1144 (2002); (b) R.Y. Yang, N.W. Zheng, H.T. Xu, Z.Q. Li, X.L. Jin. *Chin. Sci. Bull.*, **48**, 1111 (2003); (c) R.Y. Yang, N.W. Zheng, H.T. Xu, Z.Q. Li. *Chem. Bull.*, 492 (2003).

[5] (a) H.T. Xu, Y.D. Li. *J. Mol. Struct.*, **137**, 690 (2004); (b) X.J. Li, R. Cao, W.H. Bi, Y.Q. Wang, Y.L. Wang, X. Li, Z.G. Guo. *Cryst. Growth Des.*, **5**, 1651 (2005).

[6] (a) Q.Y. Liu, L. Xu. *Eur. J. Inorg. Chem.*, 3458 (2005); (b) Z. Wang, M. Strobele, K.L. Zhang, H.J. Meyer, X.Z. You, Z. Yu. *Inorg. Chem. Commun.*, **5**, 230 (2002); (c) J.L. Song, C. Lei, J.G. Mao. *Inorg. Chem.*, **43**, 5630 (2004).

[7] (a) Y.X. Ren, S.P. Chen, S.L. Gao. *J. Coord. Chem.*, **59**, 2135 (2006); (b) Y.X. Ren, S.P. Chen, S.L. Gao, Q.Z. Shi. *Inorg. Chem. Commun.*, **9**, 649 (2006).

[8] G.M. Sheldrick, *SHELXS-97, Program for Crystal Structure Solution*, University of Göttingen, Göttingen (1997).

[9] S.F. Si, C.H. Li, R.J. Wang, Y.D. Li. *J. Mol. Struct.*, **703**, 11 (2004).