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# A 3-D lanthanide coordination polymer constructed from biphenyl-2,2′,6,6′-tetracarboxylic acid: synthesis of a trinodal (3,4,5)-connected topology and luminescence

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A 3-D lanthanide coordination polymer, $[La_2(bta)_{1.5}(H_2O)_4{\cdot}2H_2O]_n$ (**1**) ($H_4$bta = biphenyl-2,2′,6,6′-tetracarboxylic acid), has been hydrothermally synthesized and structurally characterized as an unprecedented trinodal (3,4,5)-connected $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ topology. The luminescence and thermal stability of **1** are also discussed.

*Keywords*: Biphenyl-2,2′,6,6′-tetracarboxylic acid; Lanthanide coordination polymer; Trinodal (3,4,5)-connected; $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ Topology; Luminescence

## 1. Introduction

Current interest in coordination polymers not only stems from intriguing structures and topological features, but from potential applications in magnetism, electric conductivity, molecular adsorption, heterogeneous catalysis, nonlinear optics, and luminescence [1–21]. Lanthanide ions have been employed in preparation of coordination polymers with interesting networks and properties because of higher coordination numbers and inherent flexibility [17, 22–29]. Lanthanide-based coordination polymers can possess interesting luminescent properties, which may offer applications in chemical sensing, medical imaging, radiation detection, biological, and electro-optical devices [30–39]. Multidentate organic polycarboxylates, such as 1,2-benzenedicarboxylate [40–43], 1,3,5-benzenetricarboxylate [44–47], and 1,2,4,5-benzenetetracarboxylate [48–50], have been used for construction of coordination polymers with multidimensional networks. We and others have used biphenyl-2,2′,6,6′-tetracarboxylic acid ($H_4$bta) to yield discrete dinuclear complexes, 1D polymers, and 2D and 3D frameworks [51–67]. The ligand has some unique features (Scheme 1): (a) it has a variety of coordination modes and completely/partially deprotonated forms with four carboxylic groups; (b) it is a flexible ligand and the two phenyl rings can rotate around the C–C single bond, which may help to construct chiral coordination polymers due to noncoplanarity of the phenyl rings; (c) it possesses $D_{2d}$ symmetry, which is easy for assembly of higher symmetrical topological networks. However, complexes based on $H_4$bta mainly focus on Ag(I), Zn(II), Cd(II), Mn(II), Co(II), and Ni (II) ions, and no lanthanide coordination polymer has been reported so far [51–67].

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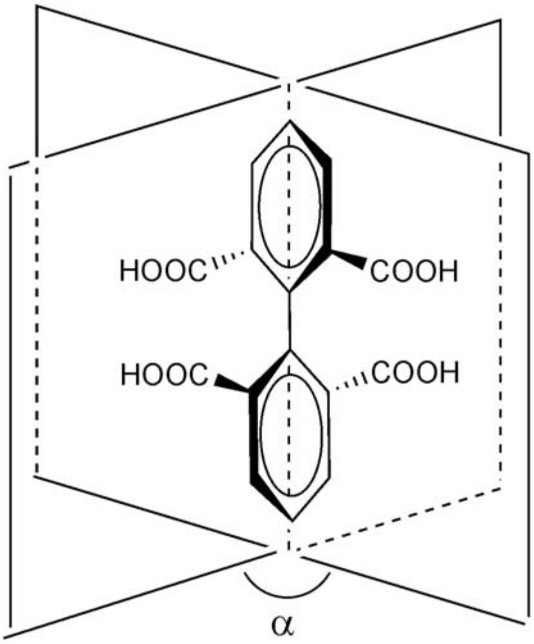

Scheme 1. Local coordination environments of La1 (a) and La2 (b) in **1**. All hydrogens are omitted for clarity. Bond lengths (Å): La1-O1 2.791(5), La1-O6d 2.765(5), La1-O10d 2.581(5), La1-O1w 2.572(4), La1-O2 2.613(6), La1-O12d 2.486(6), La1-O2 W 2.526(5), La1-O3 g 2.653(4), La1-O3 W 2.579(5), La1-O4 g 2.686(5), La2-O1 2.474(4), La2-O9d 2.583(6), La2-O4 W 2.533(5), La2-O10d 2.675(6), La2-O5 2.473(6), La2-O4 g 2.526(4), La2-O11 2.580(4), La2-O8 g 2.390(5). Symmetry codes, d: x, −y, 1/2 + z; g: 1/2−x, 1/2 + y, 3/2−z; j: −x, −y, 1−z.

As an extension of coordination polymers using $H_4$bta as a multidentate polycarboxylate [51–56], herein are reported the synthesis, crystal structure, and luminescence of a 3-D coordination polymer $[La_2(bta)_{1.5}(H_2O)_4 \cdot 2H_2O]_n$ (**1**) with an unprecedented trinodal (3,4,5)-connected $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ topology. This is the first lanthanide coordination polymer constructed from $H_4$bta.

## 2. Experimental

### 2.1. *Materials and instruments*

Infrared (IR) spectroscopic studies have been carried out in the mid-IR region as a KBr pellet (Nicolet FT-IR 200). C and H microanalyses were made with a Perkin-Elmer 1400C analyzer. Emission spectra were collected on a Perkin-Elmer LS55 spectrophotometer at room temperature under air. Thermal analysis (TG) was performed on a TA SDTQ600 thermal analyzer under nitrogen at a scan rate of 10 °C $min^{-1}$. Powder X-ray diffraction (XRD) intensities were measured on a Rigaku D/max-IIIA diffratometer (CuK, 1.54056 Å) with a scan rate of 1 $min^{-1}$ from 3 to 60°.

### 2.2. *Synthesis of 1*

A mixture of $La(NO_3)_2 \cdot 6H_2O$ (0.037 g, 0.1 mM), $H_4$bta (0.066 g, 0.2 mM), 0.1 $ML^{-1}$ NaOH solution (3 mL), and $H_2O$ (8 mL) was heated in a 15 mL Teflon-lined vessel at

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

| | |
|---|---|
| Empirical formula | $C_{24}H_{21}La_2O_{18}$ |
| Formula weight | 875.23 |
| Crystal system | Monoclinic |
| Space group | *C2*/*c* |
| $a$, Å | 26.745(3) |
| $b$, Å | 14.5124(14) |
| $c$, Å | 18.6197(18) |
| $\beta$, ° | 129.068(2) |
| $V$, Å$^3$ | 5611.0(10) |
| Z | 8 |
| $D_{\text{calcd}}$ (gm$^{-3}$) | 2.072 |
| $T$ / K | 291(2) |
| $F(0\,0\,0)$ | 3384 |
| $\mu$, mm$^{-1}$ | 3.090 |
| $h_{\min}/h_{\max}$ | −29/32 |
| $k_{\min}/k_{\max}$ | −17/17 |
| $l_{\min}/l_{\max}$ | −21/22 |
| Ref. collected/unique | 14,717/5476<br>($R_{\text{int}} = 0.025$) |
| $^{a}R_1$ ($I > 2\sigma$) | 0.0389 |
| $^{b}wR_2$ (all data) | 0.0908 |
| GOF | 1.054 |
| Max./min., $\Delta\rho$ (e·Å$^{-3}$) | 0.85/−1.06 |

$^{a}$ $R_1 = \sum \| F_o| - |F_c\| / \sum |F_o$; $^{b}$ $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$.

120 °C for 3 days, followed by slow cooling (5 °C h$^{-1}$) to room temperature. After filtration and washing with $H_2O$, colorless block crystals were collected and dried in air (0.012 g, yield 27.4% based on La). Anal. Calcd (%) for $C_{24}H_{21}La_2O_{18}$: C, 32.94; H, 2.42. Found: C, 33.01; H, 2.37. Main IR (KBr, cm$^{-1}$): 3328(b,s), 1651(m), 1541(s), 1459(m), 1391(vs), 709(m).

### 2.3. *Determination of crystal structure*

Diffraction intensities were collected on a Bruker SMART CCD diffractometer (Mo Kα, $\lambda = 0.71073$ Å). Absorption corrections were applied by using the multiscan program SADABS [68]. The structure was solved by direct methods and refined with full-matrix least-squares with the SHELXTL program package [69]. Anisotropic thermal parameters were applied to all the nonhydrogen atoms. Organic hydrogens were generated geometrically. Crystal data and details of data collection and refinement for **1** are summarized in table 1. Selected bond distances and angles are listed in table S1.

## 3. Results and discussion

### 3.1. *Synthesis and spectral characterization*

The synthesis exploits hydrothermal conditions, which are particularly effective for fabrication of coordination polymers. NaOH solution was added to the reaction mixture to promote deprotonation and coordination of $H_4$bta.

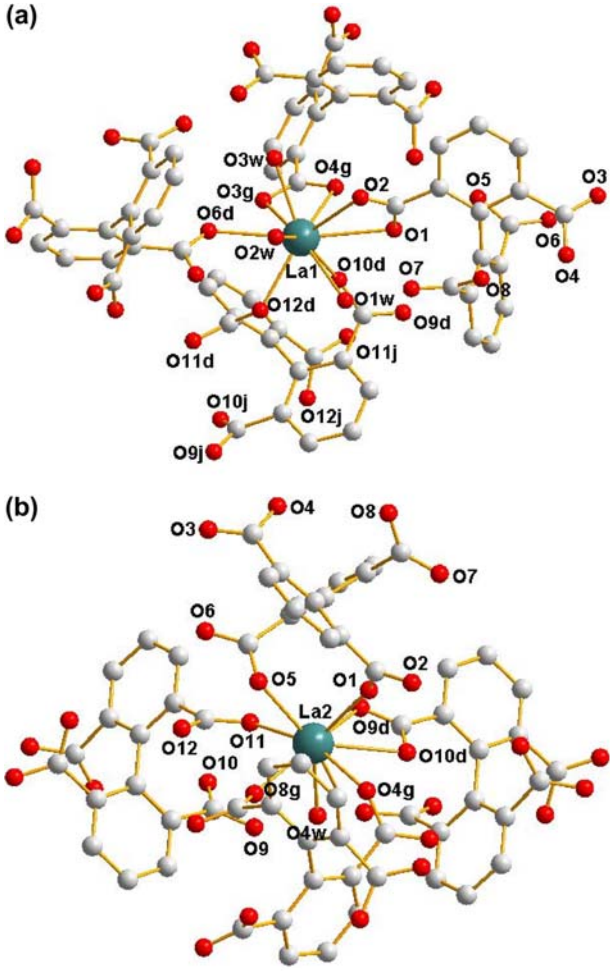

Figure 1. Structure of the dinuclear $[La_2(bta)_3(H_2O)_4]$ ($La_2$) unit (a), the 1-D chain (b) and its polyhedral form (c) along the crystallographic *c* axis, and the 3-D network (d) in **1**.

The infrared spectrum of **1** was consistent with its formulation (figure S1). Features corresponding to the skeletal vibrations of aromatic rings for **1** were observed at 1459–1391 $cm^{-1}$. At 1540 $cm^{-1}$, strong absorption was in accord with the C=O stretch of carboxylates.

### 3.2. *Crystal structure of 1*

Single-crystal XRD reveals that **1** crystallizes in the monoclinic *C*2/*c* space group, with the asymmetric unit of two crystallographically independent La(III) ions, 1.5 bta, as well as four coordinated and two free waters. As shown in figure 1, La1 is surrounded by 10 oxygens, in which seven are from four individual organic ligands and three from three coordinated waters, forming a distorted $LaO_{10}$ bicapped dodecahedron; La2 is eight-coordinate and exhibits a distorted dodecahedral geometry, with seven coordination sites occupied by seven oxygens of four bta, completed by one water.

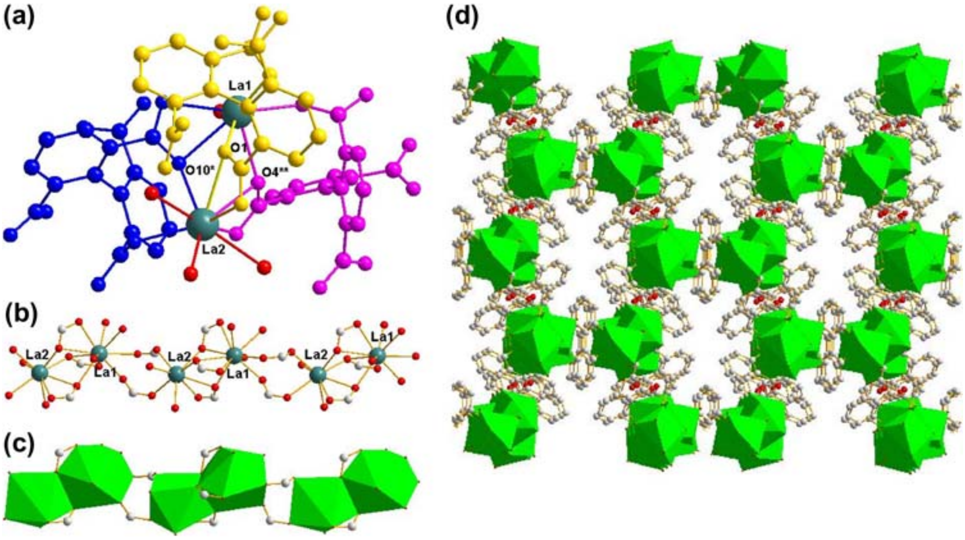

Figure 2. The $\eta^8,\mu_5$-octadentate (a) and $\eta^9,\mu_6$-nonadentate (b) coordination modes of bta (a, b), and the trinodal (3,4,5)-connected $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ topology (c) in **1**. In c, green, purple and orange-red balls represent three- and four-coordinate bta, as well as five-coordinate binuclear $La_2$, respectively.

La1 and La2 are linked by three $\mu_2$-O of three carboxylates from three bta, resulting in a dinuclear $[La_2(bta)_3(H_2O)_4]$ ($La_2$) with La1 … La2 separation of 4.133 Å [figure 2(a)]. The dinuclear $La_2$ is further connected with two adjacent dinuclear units, *via* two *syn-anti* carboxylates of two bta from two adjacent $La_2$ units, to form a 1-D chain running along the crystallographic *c* axis, as shown in figure 2(b) and (c). The shortest intrachain La1 … La2 distance separated by *syn-anti* carboxylates is 5.637 Å. These infinite chains are joined into a 3-D network by bta [figure 2(d)].

Bta ligands in **1** have two coordination modes, $\eta^8,\mu_5$-octadentate coordinating to three La1 and two La2 with the angle of two benzene rings of 73.95° [figure 3(a)] and $\eta^9,\mu_6$-nonadentate to two La1 and four La2 with the angle of two benzene rings of 71.79° [figure 3(b)]. The three La1 and two La2 coordinated to $\eta^8,\mu_5$-octadentate bta come from three $La_2$ units, while the two La1 and four La2, linked to $\eta^9,\mu_6$-nonadentate ligands, originate from four $La_2$ units. Each $La_2$ unit is connected to three $\eta^8,\mu_5$-octadentate and two $\eta^9,\mu_6$-nonadentate bta ligands. From the topological view, each binuclear $La_2$ is a five-connecting node, while each $\eta^8,\mu_5$-octadentate and $\eta^9,\mu_6$-nonadentate bta are a three-connecting and a four-connecting linker, respectively. Consequently, the 3-D network can be regarded as a trinodal (3,4,5)-connected $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ topology [figure 3(c)] by topologic analysis with the OLEX program [70]. Though some complicated examples of trinodal (3,4,5)-connected network topologies have been reported [71–91], this (3,4,5)-connected $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ network is unprecedented.

The 3D structure is sustained by O–H … O hydrogen bonds, which originate from coordinated water and carboxylate oxygen, as well as those between coordinated waters. There are spaces in the 3-D network, which are filled with free waters. These crystal waters are stabilized in the apertures of this 3-D construction by O–H … O hydrogen bonds, which involve free waters and carboxylate oxygens, as well as free water and coordinated water.

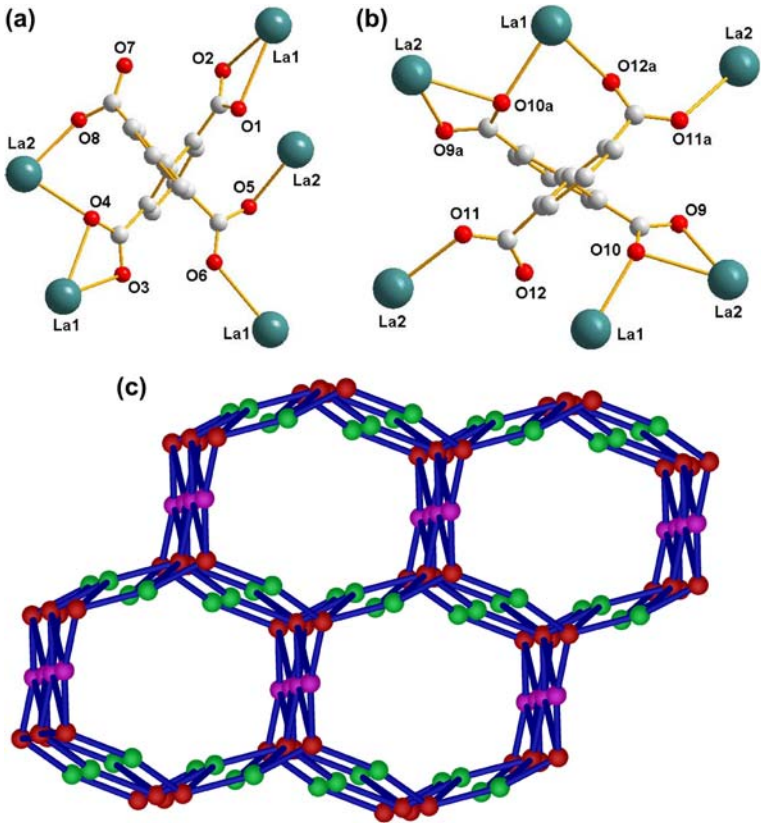

Figure 3. Luminescence spectra of **1** and $H_4$bta in the solid state at room temperature.

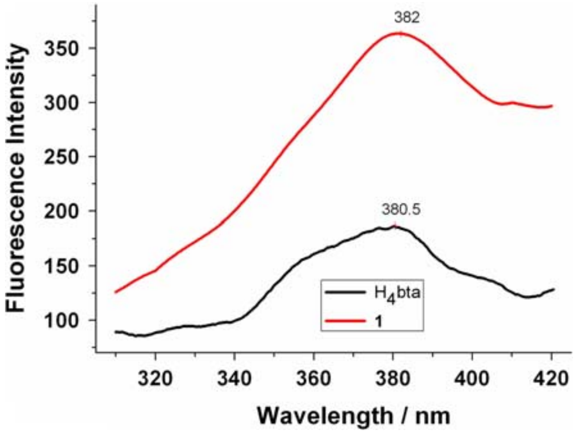

Figure 4. Luminescent spectra of **1** and $H_4$bta in the solid state at room temperature

### 3.3. *Thermogravimetric analysis*

Thermogravimetric analysis of powder sample of **1** was carried out from 24 to 637 °C under nitrogen at the heating rate of 10 °C $min^{-1}$, as shown in figure S2. The TGA curve for the compound shows that there is no weight loss between 24 and 62 °C. Decomposition of the compound begins at 62 °C with three obvious weight losses. From 62 to 115 °C, the first weight loss of 3.8% of the total weight occurs, which can be assigned to loss of crystal waters (calc. 4.1%). The second slow weight loss of 10.5% between 130 and 209 °C was in agreement with removal of coordinated waters (calc. 8.2%). The last weight loss from 248 to 402 °C corresponds to decomposition of bta (obs. 63.9%; calc. 55.9%) and collapse of the whole framework.

### 3.4. *Luminescence*

Solid-state luminescence spectra of **1** and free $H_4$bta were investigated at room temperature, as shown in figure 4. In the solid state, the maximum emission band of **1** is located at 382 nm upon excitation at 234 nm, which is similar to $\{[Cd_5(bta)_4]\cdot 6H_2NMe_2\}_n$ [56]. Since the free ligand exhibits a weak and similar UV radiation at 380.5 nm ($\lambda_{ex} = 238$ nm), the emission band at 382 nm of **1** may be attributed to intraligand luminescent emissions ($\pi$–$\pi*$). Compared with $H_4$bta, the enhancement in **1** is probably attributable to the strong interaction between the ligand and metal, or increasing rigidity due to the coordination, effectively reducing the loss of energy by nonradiative decay [56].

## 4. Conclusions

We have synthesized and characterized the first 3-D lanthanide coordination polymer constructed from $H_4$bta, with an unprecedented trinodal (3,4,5)-connected $(4^2.6).(4^4.6^2).(4^4.6^3.8^3)$ topology. The polymer displays luminescence, indicating that it may have application as an optical material.

## Supplementary material

IR spectrum, TG plot, and XRD patterns of **1**. CCDC reference numbers 879090 contain the supplementary crystallographic data for this paper. The data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

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